### BEHAVIOUR OF SHRINKAGE REDUCING ADMIXTURES BASED ON POLYETHER STRUCTURE IN VARIOUS ALKALINE SOLUTIONS

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Key Words: Shrinkage reducing admixture, alkaline solution, alkali activated materials, Raman spectroscopy, FTIR, rheology, surface tension

The several types of alkali activated materials (AAM) are in global research interest for number of previous decades. One of them - alkali activated slag mortars/concretes become attractive due to reduction of the worldwide limestone reserves and rapidly growing carbon taxes<sup>1, 2, 3</sup>. So the development of these materials in large-scale is nowadays substantial.

Granulated blast furnace slag is commonly chosen as a suitable source of latent hydraulicity specie, that can be activated by high alkaline solutions (i.e. NaOH or sodium silicate glass). Although these mortars and concretes possess very durable products with quick strength development and good chemical resistance, the high shrinkage phenomena, drying, autogenous (2 – 4 more times higher than when the ordinary Portland cement is applied), is typically observed<sup>4, 5</sup>. Various type of polymer admixtures are applied to suppress this phenomenon. One of the possible explanation is attributed to capillary stresses resulting from fine pore size of formed hydration products<sup>6</sup>. The usage of admixtures, which can lower the surface tension and influenced the pore structure of formed CASH gels, is offered. The addition of shrinkage reducing admixture (SRA) based on polyether type is intensively studied and showed the interesting results in the shrinkage development suppression. The main research is done on the interaction between the SRA and blast furnace slag particles. The behavior, interactions and stability in alkaline solutions are still not clear.

This work is related to the study of polyethylene glycol and polypropylene glycol based SRA' behavior in alkaline environment. The polymers (and corresponding monomers) were mixed with different solutions according to pH and ion composition (H<sub>2</sub>O, NaOH, sodium silicate glass and synthetic pore solution, respectively) with the mass ratio 1:1. The time dependence study was performed on the samples incubated at 25°C in sealed vials. The part of treated samples was separated after 1, 7, 14 and 28 days after mixing and dried for analyses. The Raman and FTIR spectroscopy was used to assess the stability of the chemical structure. The treated samples were also studied in terms of surface tension characterization. Finally, the time dependence reaction has also affected the rheology of solutions, what can extremely influence the workability and casting of final AAS mortars or concretes. So the rheological behavior was examined. The spectra obtained from Raman and FTIR spectroscopy analyses showed the time influence and pointed to ongoing reactions in the high alkaline system. Especially, the rheology behavior was strongly changed within the time and SRA molecular weight from liquid (in case of low weight) to almost solid state (in case of higher ones). The experimental results showed the essential need to study the polyether compound behavior in alkaline environment.

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# NEW INSIGHTS INTO THE ROLE OF HYDROXIDE IONS AND SILICATE SPECIES DURING GEOPOLYMERIZATION

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the gradual release of hydroxide ions resulting from silicate species condensation during the metakaloin

dissolution.

# SYNTHESIS OF GEOPOLYMER EMULSIONS

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Key Words: geopolymer, emulsion, monolith, hierarchical porous network

The understanding of emulsion geopolymer synthesis is a major issue for several industrial applications such as the formation of hierarchically porous material for filtration, lightweight materials for civil engineering or even the conditioning of radioactive mineral oil. Emulsion stability (irreversible coarsening, creaming...) are mainly controlled by the interfacial properties (surface tension and nature of the surfactant) and the viscosity ratio between the dispersed ( $\eta_d$ ) and the continuous phase ( $\eta_c$ ). The aim of this paper is thus to study model emulsions (composed of hexadecane (C16) as dispersed phase and metakaolin based geopolymer as continuous phase) with the highest volume fraction of C16 as possible. Surfactant was added to the mixture to stabilize the C16 droplets and geopolymer emulsion was synthesis under shear stirring. The influence of the viscosity of the geopolymer paste controlled by the water content was studied and results show that emulsions are unstable for a viscosity ratio  $\eta_d/\eta_c$  lower than 0.01. Up to 70% in volume of C16 was incorporated within the geopolymer and hierarchical porous network was thus obtained. Indeed, after removal the C16 phase, the porous network was characterized and a specific surface area of 90 m<sup>2</sup>/g, a mean mesopore diameter of 19 nm, a macropore size distribution ranged between 10-200 µm (fig 1.) and a compression strength of around 0.5 MPa were obtained.



Figure 1 : SEM image for geopolymer emulsion with 70% v/v of C16

### GEOPOLYMER-BASED BRICKS MAY BE THE NEXT GENERATION OF BRICKS: A CASE STUDY OF MINEALITHE AS AN ALKALINE ACTIVATOR

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Key Words: Alkaline activation, Geopolymer, mechanical properties, clay, MINEALITHE, brick;

The objective of this study is to compare the mechanical performance of fired bricks with geopolymer-based bricks by alkaline activation of clays. The alkaline activator considered in the research is called "MINEALITHE" that has a PH within the range of 13 -14. The choice of the solid material and the alkaline activator have a significant impact on the mechanical properties of the final product. Based on literature, the mechanical properties of Geopolymers produced from fly ash, Ground-granulated blast-furnace slag (GGBS) or calcined kaolin exhibits greater strength than Geopolymers based on non-calcined materials such as kaolin. This research challenges this statement by providing evidence in favour of a high mechanical strength of Kaolin thanks to the MINEALITHE Activator. For that, Geopolymer blocks are prepared by mixing clay, sand, MINEALITHE solution and water. Geopolymer samples are prepared according to different clay / MINEALITHE ratios. After curing at room temperature, samples are investigated using compressive and flexural tests. The results show that the obtained Geopolymers have a compressive strength comparable to that of fired bricks. This study gives insights into the potential of Geopolymer materials as a substitute of fired bricks for future constructions.

# **GEOPOLYMERS INCLUDING CDW FOR APPLICATIONS AS A BUILDING MATERIAL**

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Key Words: Construction & Demolition Waste (CDW), Alkali Activation, Geopolymers, Building Materials.

The assessment of metakaolin-slag-potassium-silicate geopolymer mixtures containing concrete and fired clay aggregates from Construction and Demolition Waste (CDW) was carried out within the framework of the H2020 European Project InnoWEE (Innovative pre-fabricated components including different waste construction materials reducing building energy and minimizing environmental impacts). The research aimed at obtaining materials with recycled CDW adequate for applications in building components, i.e. energy-efficient and sustainable prefabricated panels for external insulation, ventilated façades and radiant ceilings.

The potential of those mixtures as a building material, and their suitability for the production at industrial level, were explored through an extensive mechanical and physical characterization. The investigated properties included compressive and indirect tensile strength, drying shrinkage, open porosity and water absorption, leaching of water soluble salts, preliminary freeze-thaw resistance and open time.

Although further investigations are needed, the main parameters affecting the performance were identified. The obtained results showed that geopolymers with CDW can have adequate properties for use in building elements even with 50% and more of aggregates in weight.

According to recipe and aggregates type and amount, the compressive strength at 28 days approximately ranged between 40-90 N/mm<sup>2</sup>, with apparent dry density comprised between 1700-2000 kg/m<sup>3</sup>, open porosity between 10-30% of volume and water absorption between 10-30% of dry weight. Drying shrinkage approximately varied 0.8-3‰ and 1-4‰ after 7 and 28 days, respectively.



Figure 1 – Samples of tested mixtures

Figure 2 – Prototype panel for external insulation

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# GEOPOLYMERS BASED ON NATURAL ZEOLITE AND METAZEOLITE

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Key Words: natural zeolite, clinoptilolite, metazeolite, geopolymer, aluminate activator

Zeolites and geopolymers have similar chemical composition, but differ in their crystallinity. In Bulgaria one of the world's largest resources of natural zeolites is located. The deposit consists of rather pure clinoptilolite (70% - 80% clinoptilolite with 10% - 15% cristobalite and some amorphous phases) and has estimated reserves in the order of  $10 \times 10^9$  tons. With the aim of evaluating the suitability of this abundant raw material to serve as a precursor for geopolymeric materials, and hence applications in the building industry, an experimental study of geopolymerisation of natural zeolite was carried out.

Metazeolite was obtained by calcining the zeolite at 900 °C for one hour, at which temperature XRD analysis confirmed complete destruction of all clinoptilolite without formation of new phases, resulting in an amorphous material with a trace of residual cristobalite. Natural zeolite and calcined natural zeolite (metazeolite) were mixed with KOH, K-silicate and Na-aluminate activator solutions to produce geopolymers. In addition, also a spent Na-aluminate waste solution from the aluminium anodizing industry was used as activator. The compressive strength did increase by a factor 2 to 3 for the calcined zeolite compared to the natural zeolite. The calcined zeolite activated with K-silicate activator showed the highest compressive strength (43 MPa  $\pm$  4 MPa), but also showed high shrinkage (7%). Shrinkage could be reduced to 2% to 3% by activation with the aluminate solutions, while compromising the compressive strength to 33 MPa – 37 MPa.

The geopolymers produced from oven-dried natural zeolite and activated with aluminate solutions showed lower shrinkage (3%) and some strength as well (10 MPa – 11 MPa). Those materials could still be of interest since they contain residual clinoptilolite and newly formed zeolite Na-P and phillipsite and therefore combine the properties of geopolymers (strength) and zeolites (ion exchange and adsorption).

The geopolymer samples were examined with X-Ray Diffraction (XRD), Fourier Transformed Infra Red (FTIR) Spectrometry and Scanning Electron Microscopy (SEM). XRD revealed that the geopolymers from the calcined zeolites remained fully amorphous when activated with KOH and K-silicate, but showed zeolite Na-P and phillipsite as newly formed crystalline phases when activated with aluminate solutions. FTIR confirmed decreasing Si/AI ratios in the geopolymers when activation was done with the aluminate solutions. SEM micrographs displayed that the samples made from the oven-dried natural zeolites had many more macro pores present compared to the geopolymers from the calcined zeolites. Probably this was due to air entrainment in the zeolites.

There was no significant difference in geopolymer product between the use of chemical grade aluminate solutions and industrial waste aluminate solutions, although using waste solutions has both economic and environmental benefits.

# ALKALI ACTIVATION OF MSWI BOTTOM ASH: EFFECTS OF THE SIO2/NA2O RATIO

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Key Words: MSWI bottom ash, NaOH molarity, Na<sub>2</sub>SiO<sub>3</sub> moduli, alkali activated materials, compressive strength.

Due to its high mineral content, the valorization of bottom ash from municipal solid waste incineration (MSWI) as potential precursor in the application of alkali activated materials is attracting attention. In literature there is a large variation on using of the activator solutions to activate MSWI bottom ash. In most studies, the bulk composition rather than reactive fraction of MSWI bottom ash is considered in the alkali activation design. However a large part of the Si present in MSWI bottom ash is in the form of non-reactive quartz. In this study, mainly slag fraction was considered, the glass, ceramic and natural stony materials were removed before MSWI bottom ash was used as precursor. An efficient activator solution test. Alkali activator was made of NaOH solution with concentration varying from 4M to 8M and Na<sub>2</sub>SiO<sub>3</sub> solution with moduli of 0.75 to 1.5. The effects of SiO<sub>2</sub>/Na<sub>2</sub>O ratio, where the oxide ratio for SiO<sub>2</sub> consisting of the reactive Si contributed by MSWI bottom ash slag and by the Na<sub>2</sub>SiO<sub>3</sub> in the activator solution, on the compressive strength of alkali activated MSWI bottom ash were studied. XRD was used to determine the reaction products. SEM was used to observe the morphology of synthesized binder phase and EDX will be used to determine the binder chemistry.

## PVA FIBER/MATRIX INTERFACE CHARACTERIZATION IN ALKALI-ACTIVATED SLAG/FLY ASH SYSTEM: EFFECT OF SIO<sub>2</sub>/NA<sub>2</sub>O RATIO

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Key Words: PVA fiber; Alkali-activation; interface properties; fiber pullout

Bond behavior between fiber and matrix plays crucial role in the nonlinear properties and performance of fiber reinforced composites. However, the available information on the fiber/matrix interface properties in composites made of alkali-activated materials is still quite limited which have hindered the scientific design and production of these materials.

This study presents an experimental investigation for evaluating the bonding characteristics of PVA fiber/alkaliactivated matrix interface by performing single fiber pullout tests. Four SiO<sub>2</sub>/Na<sub>2</sub>O ratios (0.8, 1.0, 1.2 and 1.5) within the alkaline activator were used for mixture preparation to investigate the effect of chemistry of the matrix on the bond performance. All the specimens were tested after 28-days curing. The interface characteristics including chemical bonding energy, interfacial frictional force as well as the slip-hardening properties were determined from the fiber pullout tests. It was found that the chemical bonding energy increased initially and then decreased with increment of SiO<sub>2</sub>/Na<sub>2</sub>O ratio, indicating an optimal alkaline activation condition at SiO<sub>2</sub>/Na<sub>2</sub>O ratio of 1.0-1.2. At the same time, the interfacial frictional strength and the slip-hardening properties did not change significantly by increasing the SiO<sub>2</sub>/Na<sub>2</sub>O ratio. These changes with SiO<sub>2</sub>/Na<sub>2</sub>O ratios were further correlated with the chemical nature of the reaction products to provide preliminary insight into the interfacial bonding properties of PVA fibers to the alkali-activated slag/fly ash matrices.

# MECHANICAL PROPERTIES AND CREEP BEHAVIOUR OF AN ALKALI-ACTIVATED CONCRETE

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Key Words: Geopolymer, alkali-activated materials, concrete creep, mechanical properties.

Alkali activated materials (AAMs) have received great interest for production of eco-friendly concrete as an alternative for OPC-based concrete. AAMs usually have comparable or better mechanical properties and durability compared to that of OPCs, and at the same time help in reduction of CO<sub>2</sub> emissions and utilization of industrial wastes. In general, AAMs have a high resistance to very high and low temperatures and show a rapid strength gain which makes them an excellent option for rapid construction.

As a potential structural material, AAMs face some obstacles for application in construction including the lack of knowledge on long-term performance and stability as well as the lack of standardized procedures for production and design alkali activated concrete. The problem of control and increase of the material performance is the main challenge for AAMs researchers.

Despite the recent interest and intensive research on the performance of AAMs, the time-dependent deformation and creep of these materials have not been fully investigated yet. The creep phenomenon depends on many environmental (like temperature and humidity) and structural (the mix design and curing conditions) parameters and therefore it is expected have differences in AAMs compared to OPC. In this work, the authors present a preliminary experimental investigation of this phenomenon in concrete made of AAMs. The presented experimental data, including mechanical properties and creep tests results, are a step towards understanding of the creep mechanism in these materials.

The test results shows the increase of compressive strength, splitting strength, and Young's modulus during the first 30 days of curing. From 30<sup>th</sup> to 90<sup>th</sup> days, the mechanical properties have insignificant changes. The creep tests are performed for five months. The creep curves are similar to OPC and show a creep coefficient of about 5.5 for total creep and 4 for basic creep.

# METAKAOLIN BASED GEOPOLYMERS AS SOIL STABILIZERS

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Key Words: metakaolin, geopolymer, sand, clay, soil stabilizer, erosion

In many kinds of engineering constructions, wind-swept soils and soft weak soils are often stabilized and strengthened with ordinary Portland cement (OPC) and lime, to increase soil strength and durability. Furthermore, such soil stabilizing can also prevent erosion and dust generation. However, OPC is known to leave an enormous environmental footprint on planet Earth as its production processes are significant energy consumers with high CO<sub>2</sub> emissions. Therefore, the development of a novel generation of cements with high durability and environmental sustainability is essential. One of these novel binders is the alkali-activated binder based on aluminosilicates materials as metakaolin or industrials by-products such as fly ash or slags, commonly referred to as geopolymer. It has been found that geopolymers can exhibit high compressive strength and higher chemical and thermal resistance than cement-based materials [1]. Therefore, due to their high strength, low cost, low energy consumption and CO<sub>2</sub> emissions, geopolymers offers a promising alternative to OPC [2]. Geopolymers also exhibit excellent adhesion to aggregates [3], therefore it is reasonable to assume that they can serve as an effective soil stabilizer.

In this study, a new composite alkali-activated material was developed and designed to serve as a soil stabilizer for wind-swept and soft soils as well as a waterproof layer, as replacement for OPC, the conventional soil stabilizer. This new developed material can also be considered as a soil cover layer for engineered barrier system (EBS) for waste disposal facilities. The composite material was designed to have extended antierosion/abrasion resistance, high mechanical strength and high resistance to water infiltration. The materials used in the current study include local clays and silica sandy soil, molded with metakaolin as the geopolymeric binder, activated with a solution of sodium silicate and sodium hydroxide with mass ratio of 1.3:1. Six different mixtures were studied differing in their clay/sand/metakaolin content.

The different geopolymer composites were examined for their compression properties, physical degradation, chemical and morphological characterization. Increasing the sand content in the mixtures was found to increase compressive strength and mixture workability. The best mechanical properties were obtained for the mixture with sand/metakaolin mass ratio of 1:0.3 and Solids/activator mass ratio of 1:0.33 reaching a compressive strength of 33.4 MPa, with abrasion resistance meeting the Israeli standard [4].

Two-layered samples were prepared composed of a silica-sand based upper layer designed to serve as a mechanical anti-erosion barrier and a clay based lower layer designed to serve as an infiltration barrier (see Figure 1a). The adhesion between these two layers was examined by four-point flexural test along with scanning electron microscope (SEM) observation, showing a very dense and compacted interface as clearly presented in Figure 1b.



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### INVESTIGATION ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF FA/GGBS -BASED GEOPOLYMER AFTER EXPOSURE AT ELEVATED TEMPERATURE

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Key Words: Geopolymer, fly ash, GGBS, microstructure, shrinkage

Fly ash (FA) and ground granulated blast -furnace slag (GGBS) are industrial wastes and have been widely utilized as construction material due to the environmental impacts. The use of these by-products is not only for the sustainable construction but also for reducing the emission of CO<sub>2</sub> due to the use of Portland cement. This study presents the microstructure and mechanical properties of geopolymer paste made from different ratios of low -calcium FA and GGBS after exposure at elevated temperature (500- 950°C). Compressive strength tests for both heated and unheated cylindrical specimens (using 50 mm x 100 mm mold) after the age of 28 days have been carried out. The results showed that the compressive strength of unheated specimens is increasing with increasing amount of GGBS in the mixes (up to 84 MPa). However, exposure at high temperature caused dehydration of water inside the specimen, resulting in strength loss. It was also found that exposure at 500°C caused a shrinkage, while a thermal expansion event was observed to occur at approximately 750°C. Moreover, scanning electron microscopy (SEM), X-ray diffraction (XRD), and mercury intrusion porosimetry (MIP) methods have been used to investigate the microstructure, phase composition, and pore distribution, respectively.

## EFFECT OF MICROSTRUCTURE ON PHYSICAL PROPERTIES OF SLAG AND FLY ASH BASED ALKALI ACTIVATED MATERIALS

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Nowadays cementitious materials such as concrete and mortar are widely used in many fields. One of the examples is an application for construction of buildings. However, there is one problem about using cement. In producing cement, CO2 emissions are unavoidable. In order to reduce the amount of CO2 emissions in the process of cement production, blended cements that are partly replaced by by-products are recommended. By-products that are used for blended cements production are mainly blast furnace slag which is by-product of steel production, and fly ash which is generated from thermal power plant. Even blended cements include cements in them, there is still a problem of CO2 emissions. Therefore, the production of geopolymer concrete have been increased for reducing CO2 emission.

Geopolymer concrete is required to have the same performance such as compressive strength, elastic modulus, durability of chloride ion and resistance to sulfate attack as cement concrete. There are numerous researches putting focuses on these properties, but the effect of chemical composition and microstructure on physical properties are not clear, because blast furnace slag and fly ash are by-product and their compositions are difficult to control. Take this situation into account, in our study, physical properties and microstructure were investigated to evaluate the effect of microstructure on physical properties. Specifically, in order to identify the microstructure, we focused on degrees of reaction of blast furnace slag and fly ash which are rarely taken into consideration in previous studies.

In our study, we conducted experiments for identifying physical properties and microstructure. In order to identify physical properties, we conducted uniaxial compression test and measured electric resistivity and transport of chloride ion. For investigating microstructure, degrees of reaction of blast furnace slag and fly ash were measured by scanning electron microscope (SEM) with backscattered electron mode. Degree of reaction of slag was calculated from brightness level of backscattered electron image and degree of reaction of fly-ash was calculated by point-counting method of backscattered electron image. Specific surface area was also calaculated by BET theory from the result of N2 gas adsorption method. The chemical composition of reaction products was measured by energy dispersive X-ray spectroscopy (EDX). In EDX, we focused on the elements of Ca, Si, Al and K.

As a result, compressive strength and electric resistivity of geopolymer paste increased with the increase of replacement ratio of blast furnace slag and specific surface area. Diffusion coefficient of chloride ion decrease with the increase of replacement ratio of slag. Degree of reaction of slag and fly ash in this study were almost same despite of different replacement ratios of slag and fly ash, it means that the amount of reaction products from slag increase with replacement of slag. It became clear that calcium concentration in reaction product had an influence on the compressive strength considering relationship between compressive strength and calcium concentration in reaction product. In addition, a good correlationship was seen in the amount of reaction product and compressive strength. Therefore, we could see that the amount of reaction products and concentration of calcium in reaction products is significantly important in evaluating physical properties of geopolymer paste.

#### ALKALI ACTIVATION OF FLY ASHES PART II: MECHANOCHEMICAL PRE-PROCESSING AS WAY TO OPTIMIZE THE REACTIVITY

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Key Words: mechanochemical treatment fly ash

Good quality fly ash is in demand in the cement and concrete industries due to its ability to replace clinker Portland in blended cements and Portland cements in concrete. In this paper the authors show how by mechanochemical activation of fly ashes it is possible to increase the amount of fly ashes added to cement and concrete. The objective pursued in this study was to determine the mechanical properties and mineralogical characteristics of some mortars and concretes containing a high percentage of fly ash additions (50%): A=original fly ash; B= mechanically activated fly ash, C= mechanochemically activated fly ash. The mechanical strength of mortars prisms (4x4x16cm) were determined at early and long ages (1, 2, 7, 28, 90 and 180 days) at room temperature; also some concrete cubes (15x15x15cm) were analysed. The setting time and the heat flow released in the hydration process were additionally determined.

## STRENGTH AND ELASTIC BEHAVIOR OF METAKAOLIN-BASED AND BAMBOO FIBER REINFORCED GEOPOLYMERS

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Key Words: Sustainable composites, compressive strength, elastic modulus.

Amazonian metakaolin-based and bamboo fiber reinforced geopolymers were studied by means of the strength and elastic aging behaviors for construction materials applications. Scanning electron microscopy and energy dispersive x-ray fluorescence were used to investigate the microstructure of the composite materials. X-ray diffraction was used to confirm the reliability of the samples as being geopolymers. The geopolymer matrix (GP) and the GP reinforced with bamboo fiber (GPBF) samples were aged-dried at room conditions for the periods of 1-7-28 days. The GP and GPBF ultimate compressive stress increased with age from 1-day to 28-day, while elastic modulus decreased with age. The GPBF samples ultimate compressive stresses and elastic moduli were lower than the GP samples values, but still can be suitable as sustainable construction materials.

# **GEOPOLYMER-ZEOLITE COMPOSITES FOR CO2 ADSORPTION**

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Key Words: zeolite, composite, adsorbent, CO<sub>2</sub> capture, porosity

Geopolymer-zeolite composites were produced mixing different geopolymer matrices with a synthetic commercial Na13X zeolite, to combine the functional microporosity of the zeolite with the mesoporosity of the geopolymer matrix, with the further possibility to consolidate the zeolite powder. The new materials were designed and produced in forms of monoliths to be used as adsorbents for low temperature  $CO_2$  capture applications.

A potassium or sodium silicate activating solution was used to produce the metakaolin-based geopolymer matrices, then mixed with the synthetic zeolite used as a filler. As geopolymers can be regarded as the amorphous counterpart or precursor of crystalline zeolites, it is important to underline the chemical affinity between these two constituents. As a matter of fact, the morphological characterization evidenced the presence of geopolymer nanoprecipitates covering zeolite particles for the K-based composite, while in the Na-based composite the formation of a NaA zeolite phase was evidenced (Fig. 1).



Figure 1 – High resolution TEM image of newly formed NaA phase.

The composites were deeply characterized in terms of macroand microstructure, structural composition, porosity, specific surface area and  $CO_2$  adsorption capacity and selectivity. The most promising formulations gave compact porous monoliths with a mechanical resistance up to 17 MPa and a 3-dimensional structure of interconnected open pores with a wide range of porosity (from 5 Å to 2 mm).

The surface area analysis showed that the geopolymer-zeolite composites have characteristics of Type I isotherms of microporous solids with specific surface area values quite high in the order of 200 m<sup>2</sup>/g.

The adsorption performances were determined through a dedicated pressure-decay apparatus and by thermogravimetric analysis. The results highlighted as the  $CO_2$  capacity of Na-based composite resulted significantly larger (2-3 times) than K-based sorbents, approaching the values obtained for pure synthetic Na13X zeolite.

Na-based composite revealed also a synergistic effect, indeed the

CO<sub>2</sub> capacity was approximately 20% larger that the value expected by averaging those of the constituents. Such effect is mainly attributed to the large interaction among the phases (zeolite NaA and Na13X and the geopolymer matrix) obtained by the effective chemical mixing achieved during the geopolymerization reaction.

# GEOPOLYMER OXYGEN CARRIERS FOR CHEMICAL-LOOPING COMBUSTION

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Key Words: Chemical looping combustion, CLC, Geopolymers, Iron oxide, Manganese Oxide

One of the best alternatives to reduce the economic cost of  $CO_2$  capture is represented by the chemical looping combustion (CLC). This technology accomplishes indirect fuel combustion by use of a solid oxygen carrier (OC), generally a metal oxide having the capability of transporting the oxygen needed for the combustion from an air reactor to a fuel reactor, usually designed as two coupled fluidized beds. The combustion takes place in the fuel reactor through the reaction between the fuel and the solid OC, which is consequently reduced to a lower oxidation state. The reduced OC is then transferred to the air reactor, where it is regenerated by oxidation in air at high temperature. Therefore, the CLC process enables the inherent separation of the produced  $CO_2$ , the stream exiting the fuel reactor being only composed of  $CO_2$  and  $H_2O$ , easily separable by water condensation.



Figure 2 – SEM images of novel geopolymer-composite OCs

An innovative class of OCs are here reported, which documented the application of geopolymer-based materials for CLC.  $Mn_2O_3$  and  $Fe_2O_3$  were mixed to the geopolymer matrix, in order to investigate their interaction in the composite OC. The novel geopolymer-composite OCs (Fig 1) were tested in a combined gasification-CLC laboratory scale plant, consisting of two interconnected fixed bed reactors, fueled with coal char and operated at 800, 850 and 900 °C.

Only the Mn-based OCs were reduced at temperature below 900 °C. Furthermore, they were able to release oxygen in inert atmosphere, thereby demonstrating potentialities for the chemical looping combustion with oxygen uncoupling (CLOU), where free oxygen is produced by dissociation under inert atmosphere at high temperature, thus allowing direct combustion of fossil fuels (e.g. char, coal). The best performances in terms of CO conversion and oxygen rate were given by Mn based geopolymer OCs at 900 °C, even though mixed Mn-Fe geopolymer OCs provided highly comparable results. Both the systems were stable to the temperature and to multiple CLC cycles, since similar behavior in

the reactor tests were observed and no relevant modifications to their macro- and micro-structure were detected after the process.

XRD analyses confirmed the OCs phase evolution predicted by the theoretical analysis. In particular, the formation of MnFe<sub>2</sub>O<sub>4</sub> demonstrated the synergic action of the mixed oxides, and was reasonably related to the positive performances of the sample, by promoting the redox behavior. Similarly, the formation of Mn-silicates, known to enhance the CLOU behavior of Mn-based OCs, pointed out that the interaction of the metal oxide with the GP matrix could potentially improve the CLC performance of the system.

### MIXTURE OPTIMIZATION OF AN ALKALI-ACTIVATED STEEL SLAG TO MAXIMIZE BINDER STRENGTH USING OPTIMAL DESIGN OF EXPERIMENTS

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Key Words: Alkali-activated, binder, BOF-slag, optimization, optimal design of experiments

The diversity of precursors suitable for alkali activation demands a flexible methodology to study the properties of alkali-activated binders. Optimal design of experiments (ODOE) [1] allows a systematic and efficient exploration of effects and interactions among mix components and processing conditions, a situation commonly found during proportioning studies. Moreover, the ODOE algorithms provide sets of experiments of an optimized size that consider all the factors studied at the same time, a key feature to detect absolute maximums (or minimums) of a response. In this case, the strength-optimized proportioning for basic-oxygen-furnace (BOF) slag specimens activated with NaOH solutions was determined. The impact of solution molarity ranging from 0M (only water) to 0.5M and the additions of gypsum (2 to 6 wt%), Portland cement (0 to 10 wt%) and 0.2 wt% of a commercial plasticizer (polycarboxylate-based dispersant) were mapped. Proportions tested were

selected running ODOE software using an I-optimality criteria algorithm, which minimizes the average variance of model prediction. A response surface model (RSM) for 28-day strength was defined. Paste and mortar specimens were produced with the predicted proportioning of highest strength and its binding matrix was characterized and compared with low-strength samples using X-ray diffraction (XRD), secondary electron microscopy (SEM) and infrared spectrometry (FTIR). The results obtained confirm that the methodology generates a model able to predict mechanical response, detecting general trends, high impact factors and interactions. More important, the optimal experimental design can be used to effectively study changes in the binding matrices and link them to the binder's mechanical performance.



[1] Goos P, Jones B. Optimal Design of Experiments: A Case Study Approach. Wiley; 2011.

Figure 3. RSM model for 28-day compressive strength of paste specimens produced with BOF slag as precursor, (a) behavior with 6wt% addition of gypsum, and (b) behavior once plasticizer is included and 2wt% gypsum is added.

Poster Number 17

# IMPACT OF SODIUM ALUMINATE ON THE HYDRATION PROCESS OF ALKALI-ACTIVATED GROUND GRANULATED BLAST FURNACE SLAG

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In the present investigation, the effect of the presence of highly soluble alumina source (sodium aluminate, NaAlO<sub>2</sub>) on Ground Granulated Blast furnace Slag (GGBS) hydration was considered. The GGBS was alkaliactivated with sodium hydroxide (NaOH) or sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>). The impact on the evolution on the setting time, hydration kinetics and strength development was first considered. In the case of NaOH-activated mixes, the presence of the alumina source led to the delay in setting and an extension of the induction period. The alumina additive led then to an extension of the workable time of the binder. For instance the initial setting time was extended from 30 min to more than 6 hours when adding 1% by weight of NaAlO<sub>2</sub>. The strength development was also significantly improved with addition of NaAlO2, both at early age and long term. In contrast, addition of NaAlO<sub>2</sub> to the Na<sub>2</sub>CO<sub>3</sub>-activated mixes led to almost complete cessation of hydration (no measurable strength) within the whole dosage rate interval considered (0.1% to 2% of NaAlO<sub>2</sub>). Different techniques were used to investigate the impact of NaAlO<sub>2</sub> on the hydration products development, including XRD, SEM image analysis and <sup>27</sup>Al/<sup>29</sup>Si/<sup>23</sup>Na high-resolution NMR spectroscopy. It was found that the presence of the highly soluble alumina source led to rapid precipitation of Afm phases. It is suspected that, due to the low pH level in the case of the Na2CO3-activated mixes, the calcium ions are mainly distributed near the slag grain surfaces. The Afm phases may then mainly precipitate on the slag grains leading hydration blockage. Actually only Afm phases and Gaylussite were detected in the case of NaAlO2 blended Na2CO3-blended mixes.

# REINVENTING THE STRUCTURAL FIRED CLAYEY BRICKS THROUGH THE GEOPOLYMERISATION OF LATERITES

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Keywords: Laterites, structural clayey products, iron silicates, geopolymerisation

Fired clayey products have been successfully used as structural materials for many engineering applications as building and construction all around the world. In the tropical area, however, the most available raw clayey materials are laterites (kaolinite with associated iron minerals). The kaolinite present in the laterites based concretes is amorphous or metastable prompt to be activated with alkaline solution. In this work, the results of the investigations regarding the geopolymerisation of laterites are presented. It was found that in the presence of amorphous silica, the iron minerals of laterites reacts to form low temperature iron silicates with particularly good mechanical properties (15-35 MPa) as the results of the combination of polysialates, ferrosialates and ferrosilicates. Composites obtained can be valorized as products of substitution of structural fired clayey products.

# DIFFERENT FIBER MATERIALS AS REINFORCEMENT FOR GEOPOLYMER COMPOSITE

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For the last two centuries, Ordinary Portland Cement (OPC) is the most popular building material in the world due to its high mechanical properties, ease of handling and low cost. However, the concrete industry is known to leave an enormous environmental footprint. Therefore, the development of sustainable materials that could replace the OPC is essential. One of such recent developments is an aluminosilicate based material that can be activated in an alkaline medium to form a hardened sustainable product, known as 'Geopolymer'. Geopolymers exhibit equal or better engineering properties as compared to conventional concrete with better environmental foot print. However, geopolymer's main disadvantage, as concrete, is its brittleness and low tensile properties. One way to overcome this limitation is by addition of fibers, as they can control cracking by crack bridging, resulting in an increase of the tensile properties of the geopolymeric composite.

The purpose of this research was to develop a high performance geopolymer composite by addition of short fibers. Three different types of fibers were added to the matrix with two different fiber contents (0.5% and 1%). The idea was to add fibers of significant difference in their chemical nature and tensile properties: PP and Carbon fibers which are both hydrophobic, but have significant differently tensile behavior, and PVA which is hydrophilic like the geopolymeric matrix, and has moderate tensile properties. Their influence on the geopolymer flexural behavior was examined. The microstructure of the composite at the fracture surface was also studied to better understand the role of the fibers.

The results of this research showed that all fibers improved the ductility and toughness of the matrix. Geopolymeric composites with 1% carbon fibers showed the highest flexural strength, +216% compared to plain matrix, followed by the PVA fiber composites. Different failure modes were observed – fiber pull-out for the PP and carbon composites, and fiber rupture for the PVA fiber composite. This can be explained based on the different chemical nature of the fibers which produce a different matrix-fiber interface.

# EFFECT OF SULFIDES IN THE PASSIVE LAYER OF STEEL REINFORCEMENT IN ALKALI-ACTIVATED SLAGS

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Key Words: alkali-activated slag; simulated pore solution; passive film; electrochemical measurements; XPS

Steel reinforcing elements (rebars) embedded in Portland cement concretes are protected from corrosion by a thin passive film that is formed and maintained on rebar surfaces due to the high pH level of the surrounding concrete. Corrosion of reinforcing steel is frequently induced by its interaction with chloride ions, leading to local destruction of the passive layer. The nature and stability of this layer change when the surrounding concrete is produced with different cementitious materials, as the permeability of the matrix as well as the chemistry of pore solution can vary significantly. This is particularly the case when using non-Portland cements, such as alkaliactivated slags (AAS). Ground granulated blast furnace slag can contains sulfur at levels between 1-2 wt.%, mostly in a reduced state. The sulfide ions are released during the alkali-activation of vitreous slag into the alkaline aqueous solution, and subsequently can alter the nature of the passive film formed on a steel surface.

In this study the influence of sulfide on the stability, chemical composition and morphology of the passive layer forming in steel embedded in alkali-activated slags mortars, and in simulated alkali-activated slag pore solutions were investigated. The potential influence of sulfide in corrosion induced in the presence of chlorides was also assessed. This was carried out by combining electrochemical measurement with a detailed inspection of the rebars specimens using different analytical techniques. The outcomes of this study revealed that in absence of sulfides, corrosion initiation is governed by localised breakdown of the passive film, followed by metastable/stable pit growth. However, in sulfide containing pore solutions localised pitting induced by chlorides was not identified. The presence of sulfides in these systems alters the mechanism of corrosion initiation, and its influence is strongly dependent on sulfides concentration at the steel/solution interface.

### PERFORMANCE EVALUATION OF METAKAOLIN CLAY GEOPOLYMERS SYNTHESISED WITH BAMBOO WOOD ASH AS AN ACTIVATOR

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Keywords: Bamboo wood ash, biomass ash activator, Geopolymer, Ikere-Ekiti kaolin clay, Nigeria

Global concern about substantial CO<sub>2</sub> emissions during manufacturing of Portland cement has led to studies on alternative low cost green binders. Geopolymer, an inorganic binder, which is produced from reaction of an alkaline activator and aluminosilicate mineral is receiving attention as an alternative cementitious material. However, one of the drawbacks of geopolymer production is the cost of chemical activators used in its synthesis. Studies are scanty on the use of Bamboo Wood Ash (BWA) as a less expensive alternative biomass ash activator in the synthesis of metakaolin clay geopolymers. Possibility of replacing a part of the alkaline commercial activator with alkaline-rich biomass ash activator was investigated. Metakaolin clay-based geopolymers were synthesised from kaolin clay obtained from Ikere-Ekiti, Ekiti state, Nigeria and calcined at 700°C for 6 hours. Bamboo wood was obtained as a waste from a construction site in Ibadan, ashed and used as alkaline source in place of NaOH. The chemical composition of BWA and calcined clay were characterised by X-ray fluorescence spectroscopy, Geopolymers with BWA to calcined clay ratios of 5:95, 10:90, 20:80, 30:70 and 40:60 were synthesised with sodium silicate solution. Setting time, compressive strength (at 7, 14, 21 and 28 days), water absorption and density of the geopolymers were determined and compared with 100% clay geopolymers synthesised with the reported 8M NaOH/Na<sub>2</sub>SiO<sub>3</sub> activator. The BWA contained 22.9% K<sub>2</sub>O. Setting time of geopolymers increased with increasing ash/clay ratio, enhancing ease of handling and workability. At 28 days, the compressive strength values of the geopolymers containing less than 30% BWA are higher than the 28.9 MPa of the reference geopolymer. The 10% BWA:-calcined clay geopolymer exhibited the highest compressive strength (45.5 MPa), followed by 20% BWA geopolymer (38.8 MPa). Water absorption capacity values of BWA-metakolin clay geopolymers are lower than that of the reference geopolymer. Increase in the proportion of BWA resulted in increase in geopolymers bulk density. Bamboo wood ash is proposed as an alkali activator in geopolymer synthesis.

# ANALCIME GEOPOLYMERS AS SORBENTS IN WATER TREATMENT

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Key Words: analcime, geopolymers, sorbent, water treatment

Zeolite mineral, analcime [Na16(Al16Si32O96)-16H2O], is formed in mining industry as lithium carbonate is produced from spodumene (LiAlSi<sub>2</sub>O<sub>6</sub>) using sodium pressure leaching process (1). Zeolite structure possess aluminosilicate framework and pores occupied by water and exchangeable cations (2). Zeolites act as cation exchangers due to which they have been applied as sorbents in water treatment (2). Geopolymers (GP) consist of an anionic framework of corner-sharing SiO<sub>4</sub> and AIO<sub>4</sub>, and the exchangeable cations are located in the voids in the same way as in zeolites (3). However, GP are x-ray amorphous unlike zeolites. Also, GP are excellent sorbent materials in water treatment for e.g. As<sup>3+</sup>, Cu<sup>2+</sup>, NH<sub>4</sub><sup>+</sup> Ni<sup>2+</sup>, Sb<sup>3+</sup> (4-6). The ion-exchange capacity of analcime at room temperature is quite low (Querol et al. 2002) and therefore analcime was geopolymerized with a 1:1 mixture of 10 M sodium hydroxide (NaOH) and sodium silicate (SiO<sub>2</sub>:Na<sub>2</sub>O = 3.1-3.4) to improve the sorption properties of analcime. Then analcime GP were applied as sorbents in the removal of different anions and cations (e.g. ammonium, nitrate, potassium). Before sorption experiments, the GP were crushed and sieved to a particle size <500 µm and washed with deionized water until pH was stable. Experiments were conducted in batch mode with synthetic wastewater. The effect of sorbent dosage, sorption time, temperature, and anion/cation solution concentration on the anion/cation removal efficiencies of analcime GP were studied. The results indicate that analcime GP could be used as sorbents in water treatment. The use of the analcime GP in water treatment could lead to cost savings in water treatment as a low-cost by-product based GP are used instead of the commercial ion exchange resins.



Figure 4 – Ammonium removal efficiency of analcime GP

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# ADSORPTION OF COPPER AND ZINC WITH ALKALI-ACTIVATED BLAST FURNACE SLAG FROM MINE WATER

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Key Words: alkali-activated material, water purification, metal contamination, mine water

Metal contamination is an alarming problem near mining areas all over the world. Released wastewaters and mining water loose different metals to environment affecting lakes, rivers and other water sources (Jain and Das, 2017). In this study, alkali-activated blast furnace slag was used as an adsorbent for mine effluent treatment. Alkali-activation was conducted by reacting ground granulated blast furnace slag and a mixture of sodium hydroxide and silicate. Water samples are obtained from the last pumping point of infiltration water. Metal content of this water is still above the environmental safety level and the water should be recirculated and repurified. The aim of this work is to find a method to purify the mine water at this testing point to reach the environmental safety level. Then water will be releasable back to the lake.

Alkali-activated materials are widely tested and used in different kind of purification applications. These adsorbent materials are known since beginning of 1900 century but interest towards this kind of research has grown during the few last decades. There are a lot of possibilities for water research and purification processes with alkali-activated materials due to their strong and insoluble form and wide range of feasible materials available (Provis, 2014). Alkali-activated blast furnace slag was selected to be an adsorbent material for this work because it is cheap and easy to produce. It has also relatively good metal removing capacity. Same kinds of adsorbent materials have been tested for metals like nickel successfully (Luukkonen et al., 2016). This encouraged us to study more specific mining waters containing copper and zinc.

Based on our preliminary study, the removal of copper and zinc with alkali-activated material seems to be effective when handling the mine water from the metal mine. The studied water samples had the pH typically under two. When applying alkali-activated blast furnace slag adsorbent the pH of the mine effluent pH raised strongly. The metal removal mechanism was then likely to be a combination of adsorption and precipitation. With pH adjustment with acid, the removal of copper and zinc occured mainly via adsorption (Figure 1)



Figure 5 – Removal of copper and zinc from mine water with alkali-activated blast furnace slag with respect to contact time.

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# ZEOLITE-BASED CERAMIC COMPONENTS THROUGH HYDROTHERMAL DRY SYNTHESIS

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Key Words: geopolymer, zeolite, hydrothermal synthesis,

Zeolites are three-dimensional, microporous, crystalline solids with well-defined structures that contain aluminum, silicon and oxygen in their regular framework. Zeolites are generally formed in strong alkali solution (Na, K) and in hydrothermal conditions.

In this work, inorganic compacts were produced using an innovative approach, where kaolinite was directly converted into a zeolite structure through a hydrothermal synthesis without the addition of any water, and therefore in dry conditions. Zeolite-based components reinforced with fillers were also produced. XRD analyses were conducted to confirm the formation of the desired phase. Strength and microstructure were evaluated to optimize the composition of the composites.

The zeolite-based components could replace fiber reinforced plastic in terms of thermal resistance. Moreover, this class of ceramic composites can be process with the same technology of thermosetting based composites.

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#### 3D PRINTED GEOPOLYMERIC LATTICES: EFFECT OF DIFFERENT FILLER MATERIALS ON MECHANICAL PROPERTIES

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

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 glass and plastic fibers, as well as fillers like sand, to produce innovative 3D printed geopolymeric composites. The influence of these materials on the mechanical properties was evaluated.

#### ELECTROCHEMICAL CHARACTERISATION OF CEMENT HYDRATION AND PROPERTIES BY ALTERNATING CURRENT IMPEDANCE SPECTROSCOPY

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Key words: Impedance spectroscopy, cement hydration, isothermal calorimetry, inductance, white Portland cement

The complexity of the chemical and microstructural evolution of cement during the hydration process can be evaluated using many different characterisation techniques. One of these techniques which has been demonstrated to be useful, but not yet fully accepted by the cement research community, is Alternating Current Impedance Spectroscopy (ACIS). However, although ACIS is a non-destructive, rapid, and easily implemented technique, it has been found in the past that it has several limitations such as electrode contact, electrode area dependence, ground coupling effects, complications due to inductance at high frequencies, and a lack of mathematical and physical rigour in much of the data interpretation.

This study assesses ACIS as a characterisation technique to evaluate the early hydration process of white Portland cement (WPC) pastes blended with ground-granulated blast-furnace slag (GGBS), by comparison with data obtained from isothermal calorimetry and other analytical techniques. Points addressed include the electrochemical cell setup, the inductance behaviour and the resistivity changes observed during the impedance measurements, and the impact of the addition of GGBS throughout the WPC hydration process, from the fresh to the hardened state. The results show a correlation between the heat of hydration observed in the calorimetric curves and the impedance measurements, which could be used as a new approach to evaluate the early cement hydration process. Inductance behaviour observed in the impedance measurement during the early cement hydration process needs further investigation.



Fig 1. Impedance response for WPC over 24

Fig 2. Impedance response (decreasing curve) against hydration curve (increasing curve) for WPC over 24 hours.

### THE EFFECT CALCINATION METHOD AND CLAY PURITY ON THE PERFORMANCE OF METAKAOLIN-BASED GEOPOLYMERS

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Key Words: Geopolymers, Metakaolin, Calcination, Waste Immobilisation, Rheology

The calcination of kaolinite clay to produce metakaolin can be achieved using a range of processes, including rotary, fluidised bed and flash calcination. Rotary calcination was the most popular of these processes for many years as it takes place in a rotary kiln, which is readily available, at easily attainable temperatures of 650 – 800 °C. However, in recent years' flash calcination processes have become more widely used, and the technology has advanced to a point where commercial flash metakaolin-based geopolymers are now available. Flash calcination involves the rapid heating of clay at temperatures of around 1000 °C for less than a few seconds. The differences in these calcination methods can have a notable effect on the structural ordering of the metakaolin itself, as well as playing an important role in defining the chemical and physical properties of metakaolin-based geopolymers. The purity of the clay also plays a key role in the chemistry of the geopolymers produced.

Calcined clay-based geopolymers can be used as construction materials or for the immobilisation of problematic wastes, among other applications, as they can offer desirable performance characteristics. The chemical and physical properties of these geopolymers, and thus the influence of the clay source on key performance parameters, will need to be fully understood when deciding how they can be used for many different applications.

This study demonstrates the effect of the calcination method on the properties of calcined metakaolin geopolymer systems for waste immobilisation applications. A main focus of this study is the rheological properties, as the flow properties of these systems are one of the most important parameters for many geopolymer applications. The porosity, heat evolution and mineralogical development of these systems is also presented, with a view towards assessing performance in targeted applications for the immobilisation of nuclear waste.

### MECHANICAL PERFORMANCE OF ALKALI ACTIVATED MIXTURES BASED ON ELECTRIC ARC FURNACE SLAG

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Key Words: Alkaline activation, mechanical strength, electric arc furnace slag, aluminium anodizing sludge, phosphate sludge.

Nowadays there is a challenge to introduce different waste materials, generated by several areas of the industrial sector, in a valorisation chain, allowing it to play a more useful role, which is one of the premises of the circular economic paradigm. The main aim of the present work was to evaluate, through uniaxial compression and flexural strength tests, the mechanical performance of mixtures composed by electric arc furnace slag, aluminium anodizing sludge and phosphate sludge. The mixtures were activated with an alkaline solution, containing sodium hydroxide and sodium silicate, and were cured for 20 hours, at 80°C. The results of the investigation, which included a thorough characterization of the wastes, show that the addition of aluminium anodizing sludge and phosphate sludge decrease the compression strength, relatively to the mixtures prepared only with slag. Nevertheless, the strength values are still encouraging, justifying further research and, inclusive, opening the possibility of using such mixtures in specific applications, less demanding in terms of mechanical performance. The results obtained are seen as a key step to define the framework associated with this type of mixtures, in the development of applications that could be compatible with the strength levels achieved.

# POTENTIAL OF IGCC SLAG AS AN ALKALI ACTIVATED MATERIAL

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Key Words: IGCC, slag, alkali-activated material, strength, potential

Integrated gasification combined cycle (IGCC) is a next generation energy production technology that converts coal into syngas with enhanced power generation efficiency and environmental performance. IGCC produces coal gasification slag as the solid by-product. Recycling of IGCC slag is still in the early stages, but the recycling process has been around the cement and concrete industry. We calculated the reactive Si/AI ratio of IGCC slag which is generated from a pilot plant in South Korea, and evaluated the potential of it as an alkali-activated material. Samples which were activated with the combined activator of sodium silicate solution and caustic soda had an average compressive strength of 4.5 MPa, showing swelling on the top free surface. Expansion of the alkali-activated slag was possibly caused by free CaO and MgO in the slag. While the samples that were activated with the combined activator of sodium aluminate and caustic soda had an average compressive strength of 10 MPa. Hydroxy sodalite and  $C_3AH_6$  were found to be the new crystalline phases. IGCC slag can be used as an alkali-activated material, but the strength performance should be improved with proper mix design approach which can alleviate the expansion issue at the same time.

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### BENEFITS OF ADDING CALCIUM HYDROXIDE TO METAKAOLIN-BASED GEOPOLYMERS ON FAST SETTING AND STRENGTH ENHANCEMENT

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Key Words: calcium hydroxide, fast setting, metakaolin, geopolymers

Ca compounds are used in geopolymers and alkali-activated materials to accelerate setting. The effectiveness, however, can be erratic and the mechanism of fast setting is not clearly understood. It might be because the unstandardized, different combination of feedstocks in each case and the reaction products still include substantial amount of amorphous phases. We investigated the effect of some calcium compounds on the setting time in metakaolin-based geopolymers having the fixed ratio of ingredients (Na<sub>2</sub>O : Al<sub>2</sub>O<sub>3</sub> : SiO<sub>2</sub> : H<sub>2</sub>O = 1 : 1 : 4 : 10). Calcium compounds were added at dosages from 1% to 4% of the total mix weight. The final setting time of the 1:1:4:10 composition geopolymers was presumed to be over 24 hours at ambient temperature. Ca hydroxide was vastly superior to Ca oxide and carbonate in accelerating setting, and Ca carbonate even retarded the setting of geopolymers. Adding 2% of Ca(OH)<sub>2</sub> concurrently increased the compressive strength by about 12% from 68 MPa to 76 MPa. Calcium hydroxide probably decreases the modulus of the alkaline activator at low dosage and resulted in gaining higher strength. An overdose of calcium, a glass modifier, might depolymerize the molecular structure of geopolymers and cause dehydration cracking.

# GEOPOLYMER ULTRAHIGH PERFORMANCE CONCRETE: MATERIAL AND PERFORMANCE

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Key Words: ultrahigh performance concrete, geopolymer, compressive strength, durability, alkali-activated materials

During the last two decades, considerable progress has been made in the development of ultra-highperformance concrete (UHPC) with ordinary Portland cement (OPC). UHPC represents a major development step over high performance concrete (HPC), through the achievement of very high compressive strength (over 20,000 psi or 140 MPa) and superior durability due to very low permeability compared to high-performance concrete; in some cases, fibers are included to achieve improved ductility. Despite these performance advantages, deployment of Portland cement-based UHPC has been slow, in part due to the relatively high compared to that of conventional concrete components. In addition, the higher content of Portland cement in UHPC, high temperature steam curing, and use of relatively large amounts of superplasticizers increase the cost and CO<sub>2</sub> footprint. Geopolymer-based UHPCs have the potential for significant advantages over comparable OPC-based materials. We have developed a range of low-cost, low-CO<sub>2</sub> footprint, geopolymer UHPC (GUHPC) formulations. The main characteristics of these GUHPCs include: 1) Increased homogeneity by excluding aggregates >9.5mm, 2) Increased packing density through use of micro- and nano-particles, 3) Very low water-to-binder ratio through chemically tailored activator compositions and use of intensive mixing; 4) Composite binders yielding hybrid calcium aluminosilicate hydrate (C-A-S-H) and alkali aluminosilicate hydrate (A-A-S-H) gels to improve product properties; and 5) Regulation of set times using a very effective inorganic retarder.

Figure 1 shows compressive strength growth curves of several of these GUHPCs, indicating rapid strength gain and very high final strength. Samples have been studied for freeze-thaw resistance, shrinkage, alkali-silica reactions, acid resistance (up to 10%  $H_2SO_4$ ), rebar corrosion, Young's modulus and Poisson's ratio, expansion in sulfate solution and water, natural carbonation, and bond strength by slant shear. Effects of curing temperatures on materials performance were also investigated. Field testing now extends to five-year outside weathering exposure (-15°C to +45°C, ice, rain, and snow) showed no deterioration of mechanical durability. In this paper, we will review the development, characterization, and properties of these materials and prospective applications.



Figure 6 – Compressive strength as a function of curing time at room temperature

## **GEOPOLYMER WASTE FORMS FOR RADIOACTIVE WASTES**

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Key Words: Geopolymer, waste form, radioactive waste, solidification and stabilization, alkali-activated material

Geopolymer formulations, referred to as 'DuraLith', have been developed as candidate waste forms for nearsurface disposal of a range of radioactive waste streams in the United States. Examples of these radioactive waste streams include Hanford Low-Activity Waste (LAW), Hanford Secondary Waste (HSW), Sodium Bearing Liquid Waste (SBW) at the Idaho site, and Tank 48H waste at the Savannah River Site. These waste streams exhibit an extremely wide variation in chemical composition and radionuclide content, which pose significant challenges for their solidification and stabilization. In this paper, we will review the development, characterization, and properties of DuraLith geopolymer waste forms for various radioactive waste streams. Metakaolin (MK), blast furnace slag (BFS), and Class F fly ash (FFA) were selected as reactive aluminosilicate materials to produce DuraLith waste forms for these wastes. Numerous composite geopolymers have been investigated, such as FFA/BFS. MK/BFS, and MK/BFS/FFA. The alkaline activator is a tailored solution of the simulated waste stream into which alkali hydroxide and silica fume are dissolved. The testing included key radionuclides such as Tc, I, and Cs, which dominate the risk to the environment. Various enhancers such as tin fluoride and Ag-modified zeolites were employed to improve fixation of radionuclides such has Tc and I. The process of solidification of these radioactive waste streams through geopolymerization was monitored by isothermal calorimetry, rheology, and Vicat needle penetration. Cured geopolymer waste forms were characterized for compressive strength and phase composition and microstructure by XRD and SEM/EDS. Selected samples were tested for leachability of heavy metals and radionuclides after 28 days of curing at ambient temperature according to the ANSI/ANS 16.1 and TCLP leach test procedures. Effects of BFS grades and FFA incorporation on the properties of fresh and hardened waste forms were investigated.

The results show that the leachability indices for key radionuclides exceed the U.S. waste performance requirements and that U.S. EPA limits for retention of hazardous elements were successfully met. The compressive strength exceeded the minimum requirement by factors of up to 20. The results also demonstrate that the waste loading, which is a key factor in the economics of any waste form, can be extremely high for



DuraLith geopolymer waste forms as compared to conventional cementitious materials. For example, 16.5 wt% waste loading of HSW on a dry solids basis was achieved without compromising the waste form properties. The results further demonstrate that the fresh properties of DuraLith waste forms, such as set time and workability time, can be regulated by judicious use of multiple binders by, for example, incorporating FFA or using BFS of a specific grade. In scale-up testing, the feasibility of manufacturing DuraLith geopolymer waste forms has been demonstrated successfully at an engineering scale. The results indicate that DuraLith is a promising alternative waste form for immobilization and stabilization of various radioactive waste streams, providing superior performance in mechanical and chemical durability.

Figure 1. Typical microstructure of a BFS-based DuraLith waste form for HSW. The arrow indicates an iodine loaded zeolite particle. The EDS analysis suggests the matrix phase is an alkalimodified C-A-S-H gel.

#### FREEZING AND THAWING RESISTANCE OF SLAG ALKALI ACTIVATED CONCRETE WITH DIFFERENT ACTIVATORS

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Key Words: activator, frost resistance, slag.

The frost resistance of alkali activated materials is often considered very good. But some opposing results have also been recorded. Despite the good mechanical properties of some slag-alkaline concretes their frost resistance was found bad for field application. One of the reason for the explanation of the discrepancy of the results may be a different composition of the alkaline activator. Very often the content of Na<sub>2</sub>O (N) or K<sub>2</sub>O (K) is mentioned as the basic characteristic of an activator. But if water glass is used as an activator the content of SiO<sub>2</sub> (S) is also very important.

Alkali activated mortars were designed with water to slag ratio 0.50 and (N+K) content 6  $\%_{mass}$  with regard to slag content. Sodium water glass was used with M<sub>s</sub> = 2, which contains 34  $\%_{mass}$  of N and 66  $\%_{mass}$  of K - in this paper, the ratio N/S is expressed as 34/66. The composition of water glass was modified with an addition of KOH for the next ratios (N+K) / S = 40/60, 50/50, 60/60, 70/30 and 100/0 (only KOH). The level of N+K was kept at the value 6 %, which means that the dry content of the activator was 17.6  $\%_{mass}$  for only water glass and this decreased continuously to 6% for KOH only. The mechanical properties and frost resistance were tested. The frost resistance index is the ratio of the bending strength of prisms after 125 F-T cycles to the bending strengths of comparative prisms. One F-T cycle represents 4 hours in the freezer in temperature -20°C and 2 hours in water +20°C. Before testing F-T resistance the prisms were stored in a laboratory enveloped with PE foil, at the start of F-T testing, the comparative prisms were put into water 20°C.

Other mortars were prepared for ratios 34/66, 60/40 and 100/0 in order to have all of the mortars with dry mass content 17.6, 14 and 10%.







Figure 2 – F-T resistance indexes for mortars with different content of dry mass of activator and (N+K)/S = 34/66, 60/40 and 100/0

The results of the frost resistance for 6 % of (N+K) are presented in Figure 1. It shows very good results for F-T resistance, especially for mortars with the ratios of 40/60, 50/50 and 60/40. Prisms with ratio 100/0 were disintegrated during the first 45 cycles. It is interesting that F-T indexes are higher than 100 % in some cases. It means that frosted prisms show better strengths than those cured in +20°C. This phenomenon is also known from Portland cement based high performance concrete. The explanation may be in terms of a better structure of C-S-H gel which arises in a lower temperature, but there will probably also be some other influences. Figure 2 shows the results of F-T resistance of mortars with (K+N)/S ratios 34/66, 60/40 and 100/0 for dry mass of activator content 10 %, 14 % and 17.6 %. Water to slag ratio was 0.50. The results are interesting and they do not show any simple course except for bad frost resistance for ratio 100/0 (disintegration of prisms for 10 and 14%).

The results of this paper show that the frost resistance is not only influenced by the amount of activator or its composition, but probably the type of hydration products plays an important role. The general opinion about good or excellent frost resistance of alkali activated materials is not correct.

## GEOPOLYMER MATRIX FOR THE INERTIZATION OF GOLD MINE TAILINGS

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Key Words: geopolymers, inertization, mine tailings, wastes, heavy metals

The mining industry produces a huge amount of solid waste materials during mining's lifetime. Solid mine tailings typically contain many sulfide minerals and heavy metals. These fine-grained residues are usually deposited in impounding lakes near mining sites. Sulfides are oxidized in contact with water, which decreases the surrounding pH, and metal oxides are leached into the environment. This leachability causes short- and long-term environmental problems, such as contamination of surface and ground water. There is increasing interest in discovering new methods to manage mine tailings more effectively in the future. This interest is mainly focused on developing low-cost treatment or confinement processes. The possibility of immobilizing several heavy metals from gold mine tailings by reactive geopolymerization technique has been investigated in the present study. The chemical stability of geopolymers synthesized by the alkali activation of metakaolin and blast furnace slag with the addition of 40 to 50 wt% gold mine tailings is demonstrated. The geopolymers were cured at room temperature, and the effects of different Si/AI and Na/AI molar ratios and curing times were investigated. The inertization effectiveness was evaluated by means of leaching tests carried out according to standard EN 12457 after 7 and 28 days and after 18 months. The samples were immersed into the water for 1 day, and the leachable metals in the test solution were determined by ICP-OES.

Table 1. Geopolymers containing 40 wt% ot mine tailings (MT) rele	lease test results after 7 and 28 days and 18
months of curing compared to as received MT.	*QL = Quantification limit

ppm	ppm	ppm	Zn ppm	v ppm	As ppm	Sb ppm	Mn ppm
74	120	100	71	59	1520	32	1500
0,3	0,2	0,1	0,5	1,3	68	0,1	4,2
0,7	1,7	0,3	0,6	63	133	0,2	1,4
0,6	0,1	0,3	0,1	5	24	0,7	3,5
	ppm 74 0,3 0,7 0,6 0.11	ppm         ppm           74         120           0,3         0,2           0,7         1,7           0,6         0,1           0.11         0.05	ppm         ppm         ppm           74         120         100           0,3         0,2         0,1           0,7         1,7         0,3           0,6         0,1         0,3           0,11         0.05         < QL	ppm         ppm         ppm         ppm           74         120         100         71           0,3         0,2         0,1         0,5           0,7         1,7         0,3         0,6           0,6         0,1         0,3         0,1           0.11         0.05         < QL <ql< td=""></ql<>	ppm         ppm         ppm         ppm         ppm           74         120         100         71         59           0,3         0,2         0,1         0,5         1,3           0,7         1,7         0,3         0,6         63           0,6         0,1         0,3         0,1         5           0.11         0.05         < QL <ql< td="">         1.3</ql<>	ppm         ppm         ppm         ppm         ppm         ppm           74         120         100         71         59         1520           0,3         0,2         0,1         0,5         1,3         68           0,7         1,7         0,3         0,6         63         133           0,6         0,1         0,3         0,1         5         24           0.11         0.05         < QL <ql< td="">         1.3         0.12</ql<>	ppm         ptm         ptm

The results show that various elements (Cr, Cu, Ni, Zn and Mn) from gold mine tailings have been immobilized almost completely by alkali activation with proper co-binder material. The problematic oxyanions (As, Sb, B, and V), after 18 months of curing time, show leaching values very low in line with the disposal in landfill for no dangerous wastes. These results are very important because confirm the improvement of the consolidation degree with time for the geopolimeric matrix.

## ACID GEOPOLYMER MATERIALS BASED ON DIFFERENT ALUMINOSILICATE SOURCES

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Key Words: metakaolin, phosphoric acid, geopolymer, composites.

Geopolymers synthetized from the reaction of metakaolin (or alumino-silicate) in an alkali medium are studied since decades [1]. However, in order to develop composites, alkali medium is undesirable due to the detrimental effect of alkali ions on fibers. As an alternative, the use of an acid medium seems promising as some authors demonstrated an increase of mechanical properties with acid-based geopolymers [2, 3]. The aim of this work is to investigate the various formulations and to understand the reactional mechanisms.

The work is based on the activation of an alumino-silicate source with phosphoric acid to obtain geopolymer The various formulations consist to determine the Al/P ratio leading to consolidation at various temperatures (from 20 to 70°C). The samples were characterized by Fourier Transform Infrared and Nuclear Magnetic Resonance spectroscopies and X-Ray Diffraction measurements in order to study their structure. The thermal and mechanical properties were correlated with the microstructure [4].

Consolidated geopolymers were synthetized with different Al/P ratios. The consolidation time decreases with increasing Al/P ratio. For instance, the material realized with Si/Al=1,17 consolidates at 20°C in 15 and 8 days with Al/P=4 and 1, respectively. Tow behaviors can be distinguished samples presenting good fire resistance (Si/Al=1,17 and Al/P=1) or presenting poorly water resistant (Si/Al=1,17 and Al/P=4). Consequently, the impact of the reactivity of the alumino-silicate source on the geopolymerisation kinetics leads to different types of structures (secondary and metastable phases) in relation with the phosphor content.

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#### DURABILITY OF LIGHTWEIGHT GEOPOLYMERS FOR PASSIVE FIRE PROTECTION: STEEL CORROSION BEHAVIOR IN CHLORIDE-RICH ENVIRONMENT

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Key Words: lightweight geopolymers, fireproofing materials, steel corrosion, adhesion.

Different technologies are currently developed as promising passive fire protective coatings, due to the fact that fire protection of steel structures is an important requirement for structural components for several civil and industrial applications. Among the others, geopolymers have attracted lot of attention as promising materials suitable for high temperature applications. An optimized mix-design makes their amorphous structure more stable, when exposed to direct fire or heating from high temperatures, compared to ordinary Portland cement-based materials (OPC). However, the durability of a fire protective coating strongly depends on its adhesion on steel and its ability to prevent and/or mitigate steel corrosion phenomena. For these reasons, the understanding of the corrosion behavior of steel coated with geopolymer-based fireproofing coatings is necessary for ensuring the service life of the structure.

This study aims at characterizing the corrosion behavior of carbon steel coated by different geopolymeric mortars applied as passive fire protection systems. In particular, fly ash-based geopolymeric mortars were applied as coatings on carbon steel plates. They were lightened by the combination of lightweight aggregates, e.g. expanded perlite, and chemical foaming agents, such as hydrogen peroxide ( $H_2O_2$ ), in order to ensure good properties at high temperatures. In addition, geopolymeric paste and mortar containing quartz aggregate were also prepared as reference samples.

The corrosion process was evaluated using an electrochemical approach. The samples have been tested by accelerated ageing methods, such as exposure to salt spray chamber to simulate a chloride-rich environment, such as marine aerosol. The monitoring process has been done applying non-destructive techniques and it is still ongoing. In particular, open circuit potential (OCP) and linear polarization resistance (LPR) have been recorded during the exposure. In parallel, polarization curves have also been carried out at different stages of the ageing exposure to better characterize the corrosion condition of the steel substrates. In addition, adhesion between the different geopolymeric coatings and the carbon steel plates has been evaluated before and after the artificial ageing in the salt spray chamber. Finally, density and porosity measurements were also carried out to better characterize the physical properties of the geopolymers.

In this contribute, preliminary results are reported about short-term exposure. They show that in absence of any aggressive species, fly ash-based geopolymeric mortars provide a highly alkaline environment in the early curing time, enabling the passivation of carbon steel. Finally, steel corrosion behavior has been analyzed as a function of the pore structure of the geopolymeric matrix.

## AMORPHOUS, SELF-HEALED (ASH-G) GEOPOLYMER AND (ASH-C) CERAMIC COMPOSITES

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Basalt is a common volcanic rock found all around the world and on Mars. The abundance of basalt has attracted attention from construction firms and material researchers as an alternative reinforcement source. Potassium geopolymer in the stoichiometric composition K2O • Al2O3 • 4SiO2 • 11H2O was produced from fumed silica, deionized water, potassium hydroxide, (i.e. water glass) and metakaolin. The geopolymer matrix was fabricated in an IKA® high shear mixer. <sup>1</sup>/<sub>2</sub>" chopped basalt fibers from Kameny Vek in Moscow were added to potassium geopolymer in amounts of 7.5 wt %. The basalt fibers and 7.5 wt % glass frit (900°C) were then dispersed in KGP using a planetary high shear Thinky mixer and the samples were allowed to set under applied pressure at ambient temperatures for 1 day followed by 1 day at 50°C to complete the reaction. A low melting temperature fine glass frit (Tm = 900°C) was added to produce self-sealing/crack filling in a dehydrated but uncrystallized geopolymer composite (900-1000 °C). Sample geometries were 1" x 1" x 6" in dimensions. Six samples from each basalt weight class were heated to 400, 800, 900, 1000, 1100, and 1200 °C. The ramp up and down rates were 7 °C/min with a 1 hour soak time at each set temperature. SEM/EDS data indicated that melting and bonding of the glass phase dispersed into the surrounding KGP matrix, produced a self-sealing effect on the dehydrated and cracked matrix. The chopped basalt fibers melted after the KGP matrix crystallized into leucite, providing a network/glass filling system in a ceramic (1200 °C). At intermediate temperatures the geopolymer was converted to a ceramic, but the basalt fibers remained intact. The amorphous self-healing effect of the glass frit significantly improved to the flexure strength of the geopolymer and ceramic composite.

# THE USE OF WASTE BRICKS AND TILES AS A PRECURSOR FOR ALKALI ACTIVATED BINDERS

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Key Words: Alkali Activated Binders, Construction and Demolition Waste, Performance, Workability

Currently, the most common treatment of construction and demolition waste (CDW) in Europe, other than disposal, is backfilling with a very small amount being effectively reused. In an attempt to optimize the use of construction and demolition waste (CDW), potential recycling and reuse routes exist, with the most popular method being to use CDW as recycled aggregates. Another viable route, however, would be to use bricks and tiles (BT) waste, collected from CDW, as a precursor for alkali activated binders as they can make up a larger proportion of CDWi. The work was performed in the framework of RE<sup>4</sup>, "Reuse and Recycling of CDW materials and structures in energy efficient prefabricated elements for building refurbishment and construction", a European project founded by the European Commission in the framework of H2020 Research and Innovation Program (call H2020-EEB-04, GA n. 723583 I project website: www.re4.eu)

Two sources of recycled waste have been collected from Northern and Southern Europe. They were ultimately sorted and were found to contain 14 % and 27 % by weight of bricks and tiles waste respectively. Upon separation, the BT waste from both sources were ground together to form a fine powder to be used as a precursor for alkali activation.

To assess the potential use of BT waste as a precursor, mortars were prepared to measure workability and strength evolution (measured on 50 mm cubes), fixing the sand to binder ratio at 2.75. The activating solution made use of both NaOH and Na<sub>2</sub>SiO<sub>3</sub>, varying the alkali dosage M+ (M+ = Na<sub>2</sub>O/BT) and alkali modulus AM (AM = Na<sub>2</sub>O/SiO<sub>2</sub>). The original water/solids (w/s) ratio was fixed at 0.37 and was increased in increments up to 0.45 to assess its impact on strength and workability. Mortars, prepared replacing up to 80 % of BT waste with GGBS by weight, were also tested.

It was found that mortars, containing BT as the sole precursor, cured at room temperature did not set after one day. In order to accelerate reaction, subsequent mortars were cured at 70°C. Mortars prepared with a low alkali dosage ( $M+ \le 5.5\%$ ) reached low to moderate strengths after 28 days of curing; the strongest mixes reached strength values of 15 MPa. Increasing the M+ up to 7.5 % led to higher strength, up to 30 MPa. However, the strength plateaued, and even reduced marginally, at higher M+ values. Interestingly, varying the AM ratio had very limited effect on strength.

Partial substitution of BT with GGBS led to the possibility of room temperature curing. Strength also increased as the GGBS content increased. Mortars containing 20 % by weight of GGBS of precursor reached a modest strength value of 28 MPa, whereas mortars containing 80 % by weight reached an ultimate strength of 79 MPa.

The mortars were found to be workable, albeit very cohesive. When measured using the flow table test mortars prepared with a w/s = 0.37 spread to an average diameter of 14 mm. The value was near constant regardless of the AM value, ranging from 0.5 up to 1.5, for a fixed M+ = 7.5%. Only mortars prepared with NaOH as the sole activator (AM =  $\infty$ ) showed a reduction in workability. Increasing the water content of the mortars led to more workable mortar. When the w/s was increased up to 0.45, the spread reached an ultimate diameter of 20 mm. The increase in w/s, from 0.37 up to 0.45, however, resulted in a 25 % drop in strength.

Work to date suggests the potential use of BT as an alkali active binder. However, more work is needed in order to understand the reaction mechanisms in an attempt to further optimize BT as a precursor for alkali activated binders, including microstructural analysis.

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## MECHANICAL AND FRACTURE PERFORMANCE OF CELLULOSE FIBERS BASED GEOPOLYMERIC COMPOSITE INCORPORATING WASTES

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Geopolymers drew a lot of attention among scientific communities in the last decades for being a cement-like material, sustainable and eco-friendly. Its mechanical properties are well comparable to those of Portland cement and it has been demonstrated that these materials are so chemically stable that they can yield almost infinite durability compared to concrete [1]. While showing these attractive features, geopolymer nonetheless involves high cost due to refined primary materials utilization and low resistance to crack propagation (fracture toughness). On one hand, cost of production can be sensitively reduced by using wastes as source materials, among all fly-ash, a power plant by-product, and borosilicate glass, recycled glass from pharmaceutical vials. On the other, fracture toughness can be improved by producing composites from geopolymeric matrix. In this work we present a study on mechanical and fracture behavior of composite materials based on geopolymer compound made through alkali activation of fly-ash and borosilicate glass, following the formulation presented in previous studies [2], and then dispersing cellulose fibers in the matrix. Dispersion of fibers was carried out by means of ultra-sonication of the powders and fibers mixture prior to chemically activation in sodium hydroxide (12M NaOH). 4x3x16 mm samples for three-point bending (3PO) tests and chevron notch (CVN) tests were produced in a set of at least 10 samples per test. SEM observations were carried out to



Figure 1 – SEM fracture surface image of 3PO bending sample

examine the grain size of the raw materials (both powders and fibers) and to inspect the fracture surface of the broken samples (Figure 1), and measure CVN depth. Then the critical stress intensity factor was calculated from CVN results. Grain size evaluation of the raw powders was also performed by laser diffraction particle sizing technique. In figure 1 it is evidenced a well-defined dispersion of fibers, and no fiber bundles and/or agglomerates were observed. After testing, it was recorded an improvement of up to 162% and 54 % in bending strength as compared to geopolymeric compound made of the same formula of previous studies and Portland cement respectively [2]. Still, as compared to the same materials, improvement of fracture toughness was up to 50% [2,3]. These results are in line with late developments in geopolymeric composites [4]. Image analyses of 10 captured SEM pictures of the fracture surface were also instrumental to evaluate the porosity of the

geopolymeric components, giving an average value of the relative density of 91%.

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