



CONVEGNO NAZIONALE ITALIANO SUI GEOPOLIMERI



Materiali innovativi per uno sviluppo sostenibile:
dal laboratorio alle applicazioni

UNIVERSITÀ DEGLI STUDI DI BARI ALDO MORO

BOOK OF ABSTRACTS

A cura di Marina Clausi, Daniela Pinto e Cristina Leonelli



Patrocini

Sponsor



Materiali innovativi per uno sviluppo sostenibile: dal laboratorio alle applicazioni - Book of Abstracts

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Indice

Relazioni tematiche ad invito

<u>Fernández-Jiménez A.</u> , Palomo A. & Garcia-Lodeiro I. Challenge of AAMs as substitute of cement and concrete.....	5
<u>Luukkonen T.</u> Geopolymers in water and wastewater treatment: current state of the	6
<u>Miccio F.</u> , Papa E., Natali Murri A., Landi E. & Medri V. Development of green and sustainable catalysts based on geopolymers for syngas upgrading, methane reforming and chemical looping gasification	7
Barone G., Belfiore C.M., Bertino A., Caggiani M.C., Coccato A, <u>Finocchiario C.</u> , Fugazzotto M., Lanzafame G., Mazzoleni P., <u>Occhipinti R.</u> , Portale S., Stroschio A., Zafarana S. Advanced Green Materials for Cultural Heritage: the experience of UNICT in the AGMforCuHe project.....	8
Lancellotti I., Dal Poggetto G., Andreola F., Barbieri L., Romagnoli M. & <u>Leonelli C.</u> A chemical perspective in the formulation of alkali activated materials	9

Comunicazioni orali

<u>Occhicone A.</u> , Roviello G., Ricciotti L., Cioffi R. & Ferone C. Alkali-activated building materials based on red mud and blast furnace slag: characterization and environmental analysis.....	10
<u>Masi G.</u> & Bignozzi M.C. Development of sustainable geopolymer formulations for building and industrial sectors: the experience at University of Bologna.....	11
<u>Caggiani M.C.</u> , Occhipinti R., Finocchiario C., Fugazzotto M., Stroschio A., Mazzoleni P. & Barone G. Testing of Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS) as a portable tool to evaluate geopolymerization reaction	12
<u>Campanile A.</u> , Ferone C., Caputo D., Aprea P. & Liguori B. Geopolymer-Zeolite monoliths for water softening	13
<u>Clausi M.</u> , Girardi G. & Pinto D. Alkali activated materials from Apulian region precursors	14
<u>Vitale E.</u> , Costa L.T., Cappelletti P., Graziano S.F., Rispoli C. & Russo G. Use of alkali-activated volcanic ash as binder for soil improvement	15
<u>Ricciotti L.</u> , Occhicone A., Ferone C., Cioffi R. & Roviello G. Geopolymer-based Materials Recycling Porcelain Stoneware Wastes for eco-sustainable Art&Design applications	16
<u>Volpintesta F.</u> , Finocchiario C. Paris E. Barone G. & Mazzoleni P. Role of different types of construction and demolition wastes (CDW)	17
<u>Rovella N.</u> , Ulian G., Moro D. & Valdrè G. Waste Industrial materials and geopolymers: new sustainable perspectives assessable in Conservation of Cultural Heritage	18

<u>Petti R.</u> , Vitone C., Plötze M. & Puzrin A.M. Experimental study on the efficacy of mussel shells as next-generation	19
<u>Portale S.</u> , Occhipinti R., Lanzafame G., Gimeno D., Barone G. & Mazzoleni P. Ash from Cumbre Vieja volcano (La Palma, Canary Islands, Spain) from waste to resource for AAMs technology	20

Comunicazioni orali progetto GeoT-NET

<u>Pinto D.</u> GeoT-NET: the geoscience perspective	21
<u>D'Accolti L.</u> , Casiello M., Nacci A. & Fusco C. Valorization of Waste for the Materials and Energy	22
<u>Amicarelli V.</u> Methods and indicators to measure Circular Economy	23
<u>Mastrodonato G.</u> New circular economy models in the legal framework of waste	24
<u>Matteucci F.</u> , Kalogirou C. & Mouwen F. Geopolymers and Circular Economy: opportunities and challenges	25
<u>Angiuli R.</u> & Tarantino V. New use and frontiers for alkali activated materials in EU founded research projects – the experience of CETMA	26

Comunicazioni poster

<u>Bertino A.</u> , Caggiani M.C., Fugazzotto M., Barone G. & Mazzoleni P. Development of pigmented geopolymers for sustainable conservation interventions	27
<u>Blasi E.</u> , Maqbool Q., Mobili A. & Tittarelli F. Mechanical strength of alkali-activated mortars containing copper mine tailings and metakaolin	28
<u>Brienza M.</u> , Lelario F., Bufo S.A., Modley L.A. & Scrano L. Natural Clay-Based Materials for the Removal of Antibiotics from Contaminated Water	29
<u>Cofano V.</u> , Clausi M., Santoro O. & Pinto D. Geopolymers for municipal wastewater treatment produced by using natural and waste-deriving precursors	30
<u>D'Angelo A.</u> , Viola V., Dal Poggetto G., Leonelli C., Vertuccio L., Piccolella S. & Catauro M. Synthesis and characterization of Coloured Metakaolin-Based Geopolymers	31
<u>Fornari G.</u> , El Chami D., Clausi M. & Pinto D. Vegetal biomass ashes as alternative activator in alkaline activated binders: characterization and feasibility study	32
<u>Gomiero C.</u> , Casamassa E. & Magnacca G. Geopolymer based brake pads	33
<u>Lacalamita M.</u> , Mesto E., Mongelli G., Mameli P., Cerri G., Pinto D., Buccione R. & Schingaro E. Characterization of Red Muds as valuable resource for sustainability	34
<u>Mastrorilli M.</u> , Comparelli R., Dell'Edera M., De Pasquale I., Monno A., Tempesta G., Acquafredda P., Spalluto L. & Curri M. L. Photoactive TiO ₂ nanoparticles for Cultural Heritage protection: investigation in lab and application in situ.	35

<u>Occhipinti R.</u> , Caggiani M.C., de Ferri L., Xu Z., Steindal C.C., Razavi J., Andriulo F., Mazzoleni P. & Barone G. Strength and durability studies on Mt. Etna volcanic precursors-based geopolymers.....	36
<u>Ossoli E.</u> , Volpintesta F., Reggiani A., Stabile P., & Paris E. Recycling of fine industrial waste in geopolymer foam mortar for new insulation materials.....	37
<u>Savino S.</u> , Clausi M., Casiello M., Tommasi I., Fusco C., Pinto D. & D'Accolti L. Pollutants removal in aqueous solutions with geopolymers: a PhotoFenton case	38
<u>Tarantino S.C.</u> , Sturini M., Maraschi F., Occhipinti R., Clausi M., Riccardi M.P. & Zema M. Recyclable Geopolymer Blocks for the Removal of Antibiotics from Wastewater	39
<u>Viola V.</u> , Catauro M. & D'Amore A. Synthesis and characterization of bottom wood ash metakaolin-based geopolymer.	40
<u>Zafarana S.E.</u> , Barone G., Occhipinti R. & Mazzoleni P. The effect of fibres on geopolymers made using Mt. Etna volcanic ash: a preliminary study	41
Elenco dei relatori.....	42

Challenge of AAMs as substitute of cement and concrete

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The PC production contributes between 2-3% of global energy use and 8%-10% of world-wide carbon dioxide emissions (CO₂). According to estimates, in 2050, the CO₂ emissions associated with the manufacture of PC could increase between 240-260%, compared with those produced in 1990. Seeking alternative cementitious materials (ACMs) is crucial to reduce the carbon footprint caused by the cement industry. A pragmatically option is to apply alkaline activation technology to produce alkaline cements and hybrid alkaline cements. This document pursues a critical analysis of the different factors that show that: 1) the raw materials, needed to produce Alkali Activated Binders (AAB) and activators, are very versatile and abundant supply everywhere on the Earth's crust (materials that would contribute to instituting a circular economy, raw materials or waste) and that 2) given their diversity and versatility, AAB production processes can draw from technologies that are already in place, call for no huge investment, ensure end product uniformity (and therefore quality) and are characterized by a much lighter environmental impact than generated by PC manufacture.

Keywords: geopolymers, alkaline cement, hybrid alkaline cement

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Relazione tematica ad invito

Geopolymers in water and wastewater treatment: current state of the art

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Geopolymers, and alkali-activated materials as a wider group, have attracted a great deal of interest in water and wastewater treatment over the last approximately 15 years [1]. They can be used in a variety of ways: as adsorbents, catalyst supports, membranes and filters, pH adjustment agents, disinfecting materials, and matrix to immobilize solid wastes (such as sludges or spent ion exchangers) to name just a few examples [2]. The main properties enabling these applications include ion-exchange capacity, intrinsic mesoporosity, simple, extremely versatile, and low-energy synthesis, low cost, and lower environmental impact in comparison to some alternative materials (e.g., synthetic zeolites or high-temperature ceramics) [1]. Moreover, the raw materials to prepare geopolymers can be based on silicate, aluminosilicate, or alkaline waste materials, which is important from the viewpoint of circular economy. In this contribution, an overview of the preparation and modification, properties, and applications of geopolymers is provided in the context of water and wastewater treatment. A summary of a currently ongoing research project AshCycle (funded by Horizon Europe), in which geopolymer-based adsorbents are studied, is also provided.

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Relazione tematica ad invito

Development of green and sustainable catalysts based on geopolymers for syngas upgrading, methane reforming and chemical looping gasification

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Several processes in chemical industry need the utilization of suitable solid materials, usually in granular form, for different purposes: catalysis, reforming, chemi-sorption, element carrying, heat transport, etc. Chemical looping (CL) combustion allows the inherent separation of CO₂ during the combustion of fossil fuels [1] thanks to the use of regenerable oxygen carriers. In gasification, chemical looping gives rise to a more energetic syngas, likewise oxygen enriched gasification, avoiding the need of an expensive air separation unit. Novel oxygen carriers based on Mn, Fe and Cu oxides have been developed and produced via geopolymerization [2]. They were characterized in thermogravimetric apparatus and fixed bed reactor. The tests conducted in the temperature range 800-900 °C revealed the good performance of the developed oxygen carriers, which also exhibited the ability to release O₂ in inert conditions. Efficiencies in CO conversion up to 99% were achieved and some synergies between Fe and Mn oxides were beneficial toward the oxygen yield.

Upon biomass gasification, a thermochemical conversion step allows the upgrading of undesired tar into valuable gases via heterogeneous catalysis at intermediate temperature [3]. Noble metals as Rh, Ru, and Pt demonstrated high activity for the tar conversion, however their high cost is hardly compatible with applications at likely small scale. Ni-based catalysts are the most commonly used ones since they combine high activity with more competitive cost, but are easy deactivated by carbon deposition and toxic. Non critical elements (Fe, Mn, Al) have been used for producing structured and granular catalysts for tar conversion through a one step, low cost and green process (geopolymerization). These were tested by TPR in H₂ atmosphere and in a double fixed bed with real syngas from biomass, providing good and comparable performances with those reported in literature.

The paper reports the developments and the results of the research in these different applications and the future perspectives.

Keywords: geopolymer, catalytic composites, chemical looping.

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Relazione tematica ad invito

Advanced Green Materials for Cultural Heritage: the experience of UNICT in the AGMforCuHe project

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AGMforCuHe project is aimed at developing new strategies for the conservation and fruition of Sicilian Cultural Heritages by the production of alkali activated materials based on natural and industrial wastes.

Structural and decorative elements of ancient Sicilian masonries, from black volcanic stone to white calcarenites, from red bricks to mortars and plasters, reflect the availability of local resources. In this view, Sicilian natural and industrial wastes have been used as resources to synthesize advanced materials in accordance with the EU policy referred to circular economy. All these materials have demonstrated a good, chemical, mineralogical, mechanical and aesthetic compatibility with the original substrates, as required by good conservation practices.

The established partnership (led by UNICT and composed by 7 Universities and 5 companies), the sharing of competences and the quality of the involved expertise, has allowed the creation of a multidisciplinary network able to support also the management of local cultural, social and economic policies.

Moreover, the project has promoted the fruition of cultural sites during the restoration campaign, allowing the general public to become familiar with sustainable restoration practices and technological advances in the field.

Finally, the project has guaranteed an advanced technological knowledge transfer launching a semi-industrial scale-up through the collaboration with the local companies of the partnership.

Keywords: geopolymers, cultural heritage, circular economy.

Relazione tematica ad invito

A chemical perspective in the formulation of alkali activated materials

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Highly reactive metastable aluminosilicates, containing variable amounts of Ca and Fe compounds, can be opportunely alkali activated to form a cementitious material with high mechanical performance. This is indicated nowadays as a novel class of engineered binders: the alkali activated materials-AAMs.

The experience at the DIEF-UNIMORE started in 2008. Metakaolin, unfired clays, volcanic ash and laterites from Cameroon, industrial by-products containing aluminosilicates were successfully activated to produce “ceramic-like” solids. Foaming agents were tested to produce lightweight panels, and fillers of different nature were added to reinforce the most fragile structure. The aluminosilicate source characterization, the AAMs fresh pastes rheological behavior and the densified products performance are the field of expertise. A particular attention has been posed on the chemical bonding and reticulation process. With the addition of heavy metals and hazardous cations, the chemical frame of the AAMs was completed and patented. Urban solid wastes (recycled glass containers, bottom ash or fly ash from incinerator plants) have been studied as replacements for the aluminosilicate source or the sodium silicate solution. All these processes have been checked for a balanced and stable aluminosilicate chemistry in alkaline media.

The Research Group has reached out to local, national, international partners, working together on research projects and collaborating in international committees.

Keywords: Alkali activation, waste, encapsulation.

Comunicazione orale

Alkali-activated building materials based on red mud and blast furnace slag: characterization and environmental analysis

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Red mud (RM) is an iron-rich, highly alkaline hazardous and toxic residue produced during the Bayer process for alumina production. The aluminosilicate nature of the RM and its basic nature makes it potentially suitable as a precursor for the production of alkali-activated materials. In the present work, the best conditions were found for the production of alkali-activated pastes and mortars from RM and blast furnace slag (BFS) using sodium silicate (SS) as activating solution at different $\text{SiO}_2/\text{Na}_2\text{O}$ ratios. The obtained samples were characterized from chemical, physical, morphological and mechanical points of view. Very high values of compressive strength, even for the samples with a low amount of BFS in the mixture, were obtained. The higher compressive strength was measured for cubic samples containing 50 wt.% of RM (>70 MPa) [1]. Mortar samples were prepared by alkali activation of RM with various percentages of BFS and inert construction and demolition waste aggregates. Again, the highest values were obtained for the sample with 50% wt. of RM. A formulation with a reduced amount of sodium silicate and 75% wt RM in the powder mix showed particularly interesting mechanical strength (>37 MPa). Comparative life cycle assessment (LCA) was performed with commonly marketed materials [2]. Obtained samples are characterized by a lower environmental impact compared to commercial products and can be used as an eco-sustainable alternative in the construction industry.

Keywords: alkali-activated materials, waste recovery, LCA.

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Comunicazione orale

Development of sustainable geopolymer formulations for building and industrial sectors: the experience at University of Bologna

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One of the most interesting properties of alkali activated materials and geopolymers is their versatility in their applications. In addition, the possibility of activating a wide range of precursors sourced by industrial by-products is fundamental to enlarge the material sustainability in the building and industrial sectors. However, to obtain promising material performances, the mix design optimization is a critical step and shall be pursued with special attention depending on the applied precursor. For this reason, this contribute reports the results of research activity performed at University of Bologna in the last three years on the optimization of mix design of alkali activated materials starting from metakaolin and different types of industrial by-products.

Results on the performances of a geopolymeric membrane starting from flash calcined metakaolin have been obtained in light of wastewater treatment applications. In the field of building sector, ground granulated blast-furnace slag, a commonly used precursor for geopolymers, was applied for high-mechanical-performance concrete, while waste obtained from the rectifying process of porcelain stoneware tiles (locally and largely available in Emilia Romagna region due to the ceramic industrial cluster in Sassuolo and Fiorano Modenese close to Modena) was used to produce light-weight materials for thermal insulation in buildings.

Keywords: alkali activated materials, sustainability, mix design.

Comunicazione orale

Testing of Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS) as a portable tool to evaluate geopolymerization reaction

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One of the most used techniques to monitor the development of molecular reticulation in geopolymers is Fourier Transform Infrared spectroscopy (FTIR). However, to apply this method directly *in situ* during outdoor tests monitoring, a portable instrumentation would be necessary: infrared spectroscopy in Diffuse Reflection (DR), operating with a mobile device with a non-contact head could be an opportunity. It is known that spectral modifications generated in DR mode can make the correct interpretation of data particularly challenging, therefore, different materials databases are necessary to properly exploit the benefits of this technique. Very few information is found in literature about the potentiality of Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS) for geopolymers study.

For these reasons, this work [1] is aimed at testing DRIFTS efficiency in the detection of the occurred geopolymerization process. Two groups of geopolymers were studied, based on clay sediments and ceramic waste precursors, with the possible addition of metakaolin. The combination of DRIFTS with Principal Components Analysis (PCA) was used to search for criteria discriminating the successful products from those requiring a correction in their formulation.

Keywords: diffuse reflectance infrared spectroscopy, portable, geopolymerization degree.

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Comunicazione orale

Geopolymer-Zeolite monoliths for water softening

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The total concentration of magnesium and calcium in water, known as water hardness, represents a great problem for all the industrial processes which use water for steam generation. Direct feed of hard water to the boiler reduces the steam production due to the presence of metal ions, which, forming scale and sludge, priming and foaming, can greatly reduce the heat transfer efficiency [1]. Hence the necessity to soften the water prior to use in such applications. In this research, the design of a monolithic softener obtained by geopolymer gel conversion is proposed. The softener used consists in a geopolymeric macroporous matrix functionalized by the co-crystallization of zeolite A and X in mixture. The dual nature of the proposed material promotes a softening process based on the synergistic effect of cation exchange and alkaline precipitation. A softening capacity of 90% and 54% for Ca^{2+} and Mg^{2+} respectively was attained in 24 h. In fact, the softener reported a Cation Exchange Capacity value of 4.43 meqg^{-1} . Technical features such as density, porosity and mechanical resistance were also measured. The use of this monolithic softener can improve performance and sustainability of hardness removal from tap water, reducing the production of sludge and adding the possibility to partially regenerate or reuse it [2].

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Comunicazione orale

Alkali activated materials from Apulian region precursors

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With a view to pursue economic and environmental benefits from the recovery and reuse of resources locally, the contribution illustrates results of a study regarding the production of alkali activated materials (AAMs) using water potabilization sludge (WPS) from Apulian plants as main waste-deriving raw material. The work involved three steps: i) WPS characterization by different techniques XRF, XRPD, TG-DTA, FTIR, SEM-EDS; ii) evaluation of the most suitable activation conditions for producing WPS-based AAMs; iii) compatibility assessment of alkali activated systems composed by WPS and an Apulian carbonate-rich illitic clay (LCR) [1].

WPS resulted aluminosilicate-rich, with illite and kaolinite as clayey crystalline phases and a large amount of amorphous phase (up to 76 %) in which aluminium occurs mainly in amorphous or low crystalline phases. 6M NaOH solution and room temperature curing was selected for untreated and for 700 °C heated WPS. A widely characterization from the mineralogical, microstructural and chemical point of view of WPS-LCR systems demonstrated that up to 25 % of untreated and 75% of heat-treated sludges could be substituted to clay (compressive strength values < 10 MPa and physical integrity after 28 days in water) to produce novel binding materials.

Keywords: water potabilization sludges, carbonate-rich clay, alkali activation.

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Use of alkali-activated volcanic ash as binder for soil improvement

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The valorization of waste materials, such as natural and artificial pozzolanas, for the synthesis of alkali activated binders represents a potential sustainable alternative to the use of traditional binders in the field of soil stabilization. Alkali activated binders are synthesized from the chemical reaction between an amorphous precursor, which is rich in alumina (Al_2O_3) and silica (SiO_2), with a sodium or potassium-based activator. There are several secondary by-products which are commonly used as precursor for the synthesis of alkali activated binders for geotechnical purposes [2,3]. Conversely, practical application of natural pozzolanic materials in soil stabilization has been quite limited. Valorization of natural alumino-silica sources (i.e., volcanic pyroclastic deposits) is of practical interest since there is no need for natural pozzolans to be treated (i.e., calcination) before their use, with advantages in terms of lower cost, lower carbon dioxide emissions and easy access to the available resource. In the present study, an experimental investigation on alkaline activation of volcanic ash from Mount Etna has been analysed. Reactivity of the volcanic ash has been investigated considering an alkaline activator with different molar concentrations (i.e., 8M and 12M NaOH solution). Mineralogical evolution of binders has been monitored at increasing curing times by means of X-ray Diffraction and Scanning Electron Microscopy. The experimental evidence at microscale has been related to macroscopic behavior of binders. Ultrasonic wave velocities and Uniaxial Compressive Strength tests have been performed and interpreted considering the mineralogical evolution of the binders. From a mineralogical point of view, volcanic ash activated with 8M solution of NaOH shows a high reactivity to promote the formation of secondary minerals (i.e., zeolites and smectite) from the volcanic ash, generally formed as result of alkaline activation reactions. An increase of unconfined compressive strength over curing time is consistent with observed mineralogical evolution of the binder. Conversely, the use of a NaOH solution with higher molarity (i.e., 12M) affects the alkaline activation process, inhibiting the formation of zeolite crystals and thus promoting the precipitation of sodium carbonate hydrate. The occurrence of carbonatic compounds has a detrimental effect on the physical and mechanical features of the binder in terms of efflorescence formation and compressive strength reduction.

Keywords: alkaline activation, volcanic ash, soil improvement.

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Comunicazione orale

Geopolymer-based Materials Recycling Porcelain Stoneware Wastes for eco-sustainable Art&Design applications

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The preparation and characterization of new geopolymer-based materials obtained from recycling waste deriving from the production process and the “end-of-life” of porcelain stoneware products are proposed. Structural, morphological, and mechanical studies carried out on mortars prepared by using several types of by-products (i.e., pressed burnt and extruded ceramic waste, raw pressed and gypsum resulting from exhausted moulds) point out that these systems can be easily cast also in complex shapes and show a more consistent micro-structure than the geopolymer paste, with a reduced amount of microcracks [1]. Moreover, the excellent adhesion of these materials to common substrates such as pottery and earthenware, suggests their use in the field of technical-artistic value-added applications, such as restoration, conservation, and/or rehabilitation of historic monuments, or for building revetments [1].

A comparative “cradle to grave” Life Cycle Analysis between the production processes of ceramic stoneware products and geopolymeric materials based on ceramic wastes confirmed the effectiveness of the Eco-design approach that represents a strong contribution to the environmental and economic sustainability of the Italian ceramic industry [2].

The proposed materials could represent valuable candidates to overcome some problems experienced in the cultural heritage sector concerning the selection of environmentally friendly materials that meet art and design technical requirements.

Keywords: Geopolymer; Sustainability; Life Cycle Assessment.

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Comunicazione orale

Role of different types of construction and demolition wastes (CDW) in geopolymeric mortars

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CDW accounts for more than one third of the total amount of waste produced in the EU with huge large disposal areas and costs. Its recycling is a problematic issue due to own strong compositional heterogeneity. The latter depends on many factors, such as the architectural style of the buildings, the geographical area and lithology, creating problems for finding a generalized approach to this waste material.

In this study, CDW samples, coming from two different Italian areas (Camerino and Catania), were used as aggregate in geopolymer mortars. Six typologies of binders, obtained using metakaolin, fly ash, volcanic ash, Na- and K-silicate were tested to assess how the CDW aggregates, after standardizing the grain size distribution, interacted with the geopolymeric matrix. Mortars have been produced with the aim to introduce 50 wt.% CDW in specimens. Moreover, considering waste materials used as precursors in the formulations, mortars containing up to 80 wt.% waste were produced, reaching an interesting proportion of waste incorporation.

Physical-mechanical tests showed a porous structure with good compressive strength, especially in the mortars containing CDW with low Ca contents (Catania), reaching values up to 45 MPa.

The results revealed that CDW can be recycled in the geopolymer synthesis and how the compositions of aggregates and precursors play a fundamental role for the production of alternative materials, suitable for promoting circular economy in the building sector.

Keywords: geopolymer, construction and demolition wastes, mortars.

Comunicazione orale

Waste Industrial materials and geopolymers: new sustainable perspectives assessable in Conservation of Cultural Heritage

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The sustainable conservation of built Cultural Heritage (CH) has become crucial because of the faster evolution of the degradation phenomena and the growing attention to environmental safeguard. Thus, different materials are combined and tested for applications in CH; geopolymers are included in this new frontier as a possible component in consolidants or concretes. They show features in compliance with the prerequisites requested for modern materials such as a lower carbon footprint than traditional concrete, the versatility and sustainability suggested by the possibility to be mixed with industrial waste in the preparation of restoration mortars and the reduction of water amount in the synthesis of the products. In this work, Napolysialate(siloxo) geopolymer concretes were formulated according to the method [1] mixing different amounts of metakaolin as precursor, a waste industrial silt as additive and an alkali activating solution of NaOH and sodium disilicate. Uniaxial compressional test, XRD analysis, and SEM-EDS determined the mechanical properties and the evolution of the polymerization in the concretes. Data indicate that the combinations tested provided different geopolymer concretes, where the water amount has a key role in the strength reached. The results encourage the research on geopolymers in issues related to the restoration and conservation of CH.

Keywords: Consolidation, restoration.

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Comunicazione orale

Experimental study on the efficacy of mussel shells as next-generation soil stabiliser

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The exponential growth in demand for natural resources requires an original thinking about innovative strategies for the transformation of waste into new resources to be used for other applications. In this context, soil improvement is an important subject in civil and environmental engineering and searching for effective solution of admixtures for soil stabilisation is a crucial issue. In recent years, research studies focused on treatment strategies for the reuse of soft soil waste, such as dredged sediments (in Europe about 200 million m³ of sediments are dredged every year [1]), by means of natural additives in substitution of traditional hydraulic binders [2], [3].

The main objective of this study, with applications of industrial interest, is to assess the effectiveness of mussel shells, a highly impacting waste in aquaculture industry, as original additive for the hydro-mechanical stabilisation of fine-grained marine sediments to be used in partial replacement of cements.

The present note shows some of the results of the research carried out at the Politecnico di Bari in collaboration with Italcementi HeidelbergCement Group and ETH Zurich. In detail, the experimental results, obtained from multidisciplinary and multiscale analyses on both natural and treated samples of dredged marine sediments, show the effects of the use of the new treatment solutions on the chemical, physical and hydro-mechanical properties of the sediments.

Keywords: eco-sustainability, recycling, innovative solutions

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Comunicazione orale

Ash from Cumbre Vieja volcano (La Palma, Canary Islands, Spain) from waste to resource for AAMs technology.

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In this work, the feasibility of using Cumbre Vieja (La Palma Island, Spain) volcanic ash to produce Alkali Activated Materials (AAMs) was investigated for the first time. Last, recent (2021) eruption caused extensive damages due to lava flow invasion and the discharge of huge amount of pyroclasts, which volume amounted to $45 \times 10^6 \text{ m}^3$ [1] that blanketed the entire surface of the island. Although pyroclasts are considered as a waste material, previous studies showed excellent results of their use as raw materials in the AA-process [2,3]. For this reason, different formulations using Cumbre Vieja ash were prepared, adding small amount of metakaolin and using sodium hydroxide (8M) and sodium silicate solution as activators. After a curing period of 28 days at room temperature, the samples were investigated by XRD, FT-IR, SEM-EDS, microCT, pH and electric conductivity measurements. Results indicate that Cumbre Vieja ash-based AAMs proved to be highly satisfying in terms of the quality of the binders obtained.

Keywords: Alkali Activated Materials, volcanic ash, La Palma.

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Comunicazione orale progetto GeoT-NET

GeoT-NET: the geoscience perspective

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Natural clays represent the primary source materials used for geopolymerization reaction owing to their relatively high Si and Al content, with kaolinite clay being commonly the most used one in geopolymer synthesis. However, natural clay deposits generally consist of a mixture of different clay and associated minerals, which are strongly affected by the nature of the parent rocks. The research interest of the geoscience team from Bari is focused on the study of common local deposits containing complex clay mineral assemblages, rather than only kaolinite, as source material for geopolymers and alkali activated materials (AAMs). Special attention is paid on the investigation of pretreatment methods for clay activation which greatly influence the successful of the geopolymerization reaction. The evaluation of several waste materials as substitute of natural clays and chemical industrial activators for the preparation of AAMs is also field of interest of our team. In the last times the research interests on geopolymers and AAMs of our team met the expertise of researchers from different research areas in the University of Bari, materializing in the multidisciplinary research projects “WASP- water as sustainable products” and “GeoT-NET – Geopolymer technology for a clean and sustainable future”, which explore the applicability of geopolymeric materials in heterogeneous solid catalysis for removing organic and inorganic pollution from air and water.

Keywords: clays, alkali activated materials, multidisciplinary research.

Comunicazione orale progetto GeoT-NET

Valorization of Waste for the Materials and Energy

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The research is currently intensively focused on use of renewable resources to produce energy and for new innovative materials. Particular attention is paid to wastes as raw materials for producing biodiesel, lubricants, surfactants, polymers, solvents, and fine chemicals. [1-3].

The development of efficient and sustainable, catalytic processes is the main to achieve these transformations, and heterogeneous catalysts play the dominant role for applications at an industrial level. This speech will offer a survey our recent findings in this field with special attention to up-scalable catalytic processes without “critical raw materials”, using also the geopolymers as catalysts, capable of producing chemicals, fuels and cellulose-based products, from waste materials.

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Methods and indicators to measure Circular Economy

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It has been a long time since the concept of “circularity” has been introduced by several economists intending the interconnection between the environment and economic system with the final aim to achieve, in a given period, resources consumed, and waste released zero balance. This approach indicates the capacity to implement integrated closed-loops able to assure constant recycling process and secondary raw materials availability to prevent and/or minimize virgin resources utilization. At global level and during the time, the circular economy model has assumed growing interest in contrast with the classic linear model (take-make-dispose). In this regard, the European Union has addressed a wide range of actions and policies to support, with huge capitals, the transition from linear to circular model until March 2020, when the European Commission adopted the “New circular economy action plan”, one of the European Green Deal main building blocks. Despite all these efforts, one of the still open crucial points is how to measure circular economy and how to standardize appropriate methodologies and indicators to make comparable different performance, to identify best practices and to replicate them. National and international organizations for the standardization are working hard to achieve these goals and, at the end of November 2022 the first Italian standard UNI/TS 11820:2022 was published, which provides a set of definitions and indicators for measuring the circularity of a product and/or a service organization. UNI/TS 11820:2022 key role will be presented.

Keywords: circular economy, UNI/TS 11820:2022, circularity indicators.

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New circular economy models in the legal framework of waste

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At an early stage of development, the structure of the production system has not developed the ability to absorb and reuse waste: only later was realised that the adoption of a circular economy development model could achieve the goals of sustainable development. EU action in the waste sector dates back to the mid-1970s, as a uniform waste policy would have improved the functioning of competition.

EU action in the waste sector dates back to the mid-1970s, as a uniform waste policy would have improved the functioning of competition. Although the Waste Framework Directive 2008/98 was already very modern, the 2018 Circular Economy Package seems to go much further: it introduces new quantitative recycling targets and the terms waste or disposal now seem to be marginal notions, to leave room for the concepts of circular economy, such as the productivity of resources, efficiency in the use of materials and energy, renewable resources, industrial symbiosis, the sharing of the economy and, last but not least, the concept of end of waste.

Keywords: waste, sustainability, circular economy.

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Comunicazione orale progetto GeoT-NET

Geopolymers and Circular Economy: opportunities and challenges

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The EU has set clear ambitions for decarbonisation, with a target to reduce GHG emissions by at least 55% by 2030. Cement production is currently responsible for around 7% of global and 4% of EU CO₂ emissions [1]. To mitigate the approximately 60% of cement emissions stemming from the calcination of limestone into calcium oxide there are two main solutions: carbon capture storage and utilisation technologies and alternative chemistries [2]. Besides, improved cement recycling and high clinker efficiency in new cement mixes would appear to make sense as part of a broader portfolio of solutions and could theoretically be implemented today. Anyway, besides technical and supply chain issues, current regulations, and standards, that play a crucial role in the transformation of the cement sector towards climate neutrality, currently hinder the use of innovative technologies reflecting in part the conservatism of the construction sector [3]. The breakthrough technologies needed to decarbonise cement production are the results of decades of R&D in the sector. The European Commission (EC) has been and is instrumental in supporting early-stage R&D projects in the cement sector focussing on CO₂ emissions reduction. Within Horizon Europe is providing the means to further develop breakthrough deep or science-based technologies and progress on the TRL scale, funding (large and small) demonstrators and first-of-a-kind facilities via the European Innovation Council and the EU innovation fund.

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New use and frontiers for alkali activated materials in EU founded research projects – the experience of CETMA

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CETMA has been working for over 20 years on sustainability of building materials for applications such as cement-free binders and the reuse of different type of waste as aggregates in concrete mixtures. AAM represent a valid alternative to the use of cement-based binders [1], possess enhanced mechanical and durability properties [2] and can be used for the 3D printing of building and components [3].

Some applications for AAMs still unexplored and CETMA has recently been engaged in EU research projects and open calls on the AAM theme.

MAREWIND - development of AAM mixtures for the construction of more durable marine foundations of offshore wind farms with a smart FRP system with dual function of reinforcement and structural monitoring system.

EXPLOIT4INNOMAT - implementation of a pilot line to produce building components with the use of AAM-based mixtures and recycled aggregates from CDW or from other sectors.

METABUILDING - development of innovative mixtures with AAM binder and natural aggregates (rice processing waste and hemp fibres) to be used to produce insulating blocks for green building.

The research work in this area is very active and constantly looking for new solutions and applications. Thanks to these works, increasingly sustainable, economic, and high-performance mixtures will be available in the future for use in the construction sector.

Keywords: Alkali Activated Materials, recycling, building.

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Development of pigmented geopolymers for sustainable conservation interventions

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Recently, innovative alkaline-activated materials are gradually entering the field of Cultural Heritage conservation.

These categories of eco-friendly materials are very promising and, due to their versatility, are suitable for various applications, as shown by recent studies on application in the field of conservation with different functionalities, both structural and of reintegration.

Thanks to recent scientific researches, involving leading companies in the field of Italian and international Cultural Heritage conservation, they have proven certainly to have great potential, which this research attempts to investigate, to become of primary importance in the field of conservation, with a view to modern and innovative restoration, characterized by low environmental impact and aimed at the protection of our Cultural Heritage with a view to protect the Planet.

The present study is part of a PhD project involving the development of pigmented geopolymers for sustainable restoration of a particular category of goods: polychrome surfaces.

Preliminary geopolymers with different kind of inorganic pigments, added directly into the geopolymer mixture were tested by means of colorimetric analysis.

Keywords: pigmented geopolymers, conservation interventions, sustainability.

Mechanical strength of alkali-activated mortars containing copper mine tailings and metakaolin

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AAMs (alkali-activated materials) were obtained by the polymerization reaction of a solid aluminosilicate precursor, based on copper mine tailings (CMT) from Chile and commercial metakaolin (MK), as more sustainable alternative binders to Portland cement for building applications. The precursor was mixed with activating solutions composed of sodium silicate (SS) and potassium hydroxide (KOH). At first, to test the potential reactivity of CMT, preliminary AAMs pastes were prepared by replacing MK with 0%, 25%, 50%, 75% and 100% by mass of CMT. The 5 blends were activated by adopting different (Na+K)/Al molar ratios: equal to 1 (group 1), equal to 2 (group 2), and decreasing from 100% CMT to 100% MK content (group 3), whereas the Si/Al ratios varied between 1.50 and 3.5. The most promising mixtures in terms of hardness and absence of efflorescence were chosen, namely those containing 75% and 50% of MK belonging to groups 1 and 2, to prepare mortars by adding a calcareous sand ($d_{max} = 8\text{mm}$, aggregate/binder ratio equal to 3 by weight) and by optimizing the water/binder ratio. Mortars were cured at room temperature and characterized by mechanical strength tests after 1, 7, 14 and 28 days. Results show that the highest compressive strength was obtained by mortars belonging to group 3 which, after 28 days of curing, reached compressive strengths up to 21 MPa and 44 MPa when the 75% and 50% of MK was replaced by CMT, respectively.

Keywords: AAMs, copper mine tailings, metakaolin, mechanical strength.

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Natural Clay-Based Materials for the Removal of Antibiotics from Contaminated Water

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The release of antibiotics into the environment has increased remarkably due to the extensive use of these pharmaceuticals worldwide. Sulfamethoxazole (SMX) and trimethoprim (TMP), two antibiotics included in the 3rd Watch List and often prescribed together, were selected as representative pollutants. In this study, we investigated volcanic ashes collected on Monte Vulture (PZ, Italy) as a material tested to remove SMX and TMP from wastewater. X-ray photoelectron spectroscopy (XPS) showed that volcanic ash was composed mainly of pyroxene and olivine. Preliminary tests revealed that this material was an excellent TMP adsorbent but not for SMX. The presence of metals like Fe and Al makes it capable of activating oxidizing agents such as sodium persulfate (PS). Indeed, experiments showed that the SMX was efficiently degraded under the test conditions. A systematic study was performed to evaluate the influence of the most critical factors, such as initial antibiotic and PS concentrations, liquid-to-solid ratio, and reaction time, on the removal efficiency. Factor levels were chosen to cover a range of values of practical interest. The main effects and interactions between variables were estimated through a statistical analysis performed on the data collected. The results of this study suggest that the proposed approach could represent a valuable strategy for *in-situ* and *ex-situ* remediation of antibiotic-contaminated waters and soils.

Keywords: antibiotics, adsorption, catalysis, volcanic ash.

Geopolymers for municipal wastewater treatment produced by using natural and waste-deriving precursors

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The main goal of this work is to evaluate the physical, chemical and mineralogical properties of geopolymers designed for the wastewater treatment. In addition to a commercial kaolin, geopolymerization reaction was tested on wastewater treatment sludge (WWTS), resulting from the tertiary treatment [1] and subsequent dehydrated in phyto-depuration bed at the wastewater treatment plant of Lago Forcatella (Fasano, Italy). Both precursors were heat-treated at 700°C to promote the kaolinite dehydroxylation in the former case, and the organic matter removal in the latter [2][3], then activated by using a solution of 6M NaOH and sodium silicate and cured at 40°C for 24 hours and at 85°C for 5 hours. Silica fume and different foaming agents were also added in order to improve the geopolymers features.

Analyses, carried out on monoliths, showed that geopolymerization occurred in all samples and it was influenced by the amount and type of additives used. More in general, the incorporation of geopolymers in the current wastewater management can help in waste materials recovery and the production of add-values products, thus contributing at the end-of-waste idea.

Keywords: wastewater treatment sludge; metakaolin; geopolymers.

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Synthesis and characterization of Coloured Metakaolin-Based Geopolymers

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Over time, several coloured materials have been employed in buildings, paintings, ceramics, and mosaic restoration. In recent years, geopolymers, due to their high chemical and mechanical resistance, have attracted great attention to be used in many areas, including restoration [1].

In this work, coloured geopolymers are realized starting from white metakaolin paste and pH indicators (i.e. bromothymol blue, cresol red, phenolphthalein, and methyl orange) as dyeing agents. The geopolymers, cured at 25 and 40°C are chemically analysed at different ageing times (from 7 to 56 days) through the Ionic Conductivity and pH measurements and FT-IR. Eventually, the colour hues are assessed in the CIELAB colour space before and after immersion in water.

The results confirm the chemical stability of the consolidated materials, while FT-IR analyses prove the occurrence of the geopolymerisation process. Finally, the retention of the dyes for a short period of time is demonstrated suggesting the possible utilization of these materials in indoor environments.

Keywords: Coloured Geopolymers; Synthetic dyes; FT-IR.

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Vegetal biomass ashes as alternative activator in alkaline activated binders: characterization and feasibility study

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One of the main obstacles to industrial scale up of Alkaline Activate Binders (AABs) consists in the difficulty of handling high corrosive alkaline activating solutions for their preparation. Furthermore, the use of very expensive alkaline activating solutions (i.e. Na and K silicates) produced by the conventional chemical industry, badly affects the economic and environmental feasibility of AABs. In the perspective of reducing the environmental impact of AABs, it is useful to find alternative alkaline activators, as well as to implement new strategies for reducing the amount of activating solution during the preparation. Recent studies suggest that biomass ashes can be evaluated as interesting alternative activators owing to their high calcium, potassium and silicon content [e.g.1,2]. In this study, eight samples of vegetal biomass ashes, consisting of four fly ashes and four bottom ashes from different cogeneration plants producing electricity through the combustion of agro-forestry residues, were characterised from the chemical and mineralogical point of view to evaluate their suitability as alternative activators in the formulation of AABs.

Results show the occurrence of high proportions of K_2O , CaO , Na_2O in biomass fly ashes and their chemical and mineralogical composition indicate a good potential as alkaline activators. Bottom ashes samples show a high content of CaO and SiO_2 , with significant amount of amorphous phase and several crystalline phases such as Ca, K-sulfates and -silicates; they can be evaluated both as solid precursors or a source for generating alkaline activating solutions.

Keywords: alkali activated binders, vegetal biomass ashes, alkaline solutions

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Geopolymer based brake pads

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The development of products with reduced carbon footprint is of major interest also in the automotive industry. Geopolymers (GPs) are eco-friendly materials and RAICAM^[1] is studying the replacement of phenolic resin (PR) with GPs as binder in brake pads.

The considered GP was obtained by reacting metakaolin with a potassium silicate solution. Both PR and GP were fully characterized (i.e. by SEM-EDS, DSC, TGA) and their comparison highlights a significantly major thermal stability of GP. Indeed, the PR degradation starts around 350°C and leads to 85-90% of total weight loss, whereas GP has a total weight loss of 20-22% mainly ascribable to water loss.

To verify the application of GP as binder, P1 and P2 prototypes were prepared with the same formulation but adding GP to the first one and PR to the second one. P1 appears weaker in terms of hardness and shear strength, but TG analysis shows a major degradation till 600°C for P2 due to the binder thermal instability.

Considering the performance, it was evaluated on a bench dynamometer by AK Master test^[2] (involving brakes at different pressures, speeds and temperatures) and by a Block Wear test (temperature steps at 100, 300 and 500°C). P1 shows improved friction stability and lower pad wear at 500°C.

As a conclusion, the tests suggest GP is a promising substitute for PR. Further studies are needed to confirm the results and optimise the formulation.

Keywords: Geopolymer, brake pads, eco-friendly

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Characterization of Red Muds as valuable resource for sustainability

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Residues of the Bayer process developed on bauxite ores (Red Muds, RM) represent an environmental challenge due to their high alkalinity and storage problems. This study aims at a geochemical and mineralogical characterization of RM stored in Porto Vesme (Sardinia) disposal sites with the objective to enhance the knowledge on the processes promoting the concentration of critical metals (CMs) in such matrixes. In turn this is essential in the perspective of applying a strategic CMs recovery and an efficient reuse of the residues obtained from the CMs recovery for the preparation of low cost and environment friendly construction materials. The characterization was carried out through a combination of X-ray powder diffraction (XRPD) and Inductively Coupled Plasma Mass Spectrometry (ICP-MS).

XRD results evidence that main phases are: hematite, α -Fe₂O₃; gibbsite, Al(OH)₃; boehmite, AlO(OH); anatase, TiO₂; cancrinite, (Na, Ca, □)₈(Al₆Si₆)O₂₄(CO₃, SO₄)₂·2H₂O; sodalite, Na₄(Si₃Al₃)O₁₂Cl; quartz, SiO₂. Mayor oxide composition (wt %) from ICP-MS analysis includes: SiO₂ (11.9 ÷ 22.6), Al₂O₃ (17.4 ÷ 24.9), Fe₂O₃ (22.2 ÷ 30.3), MgO (0.7 ÷ 4.7), CaO (2.6 ÷ 5.9), Na₂O (3.5 ÷ 11.5), K₂O (0.2 ÷ 0.7). Relevant concentrations of Zr, Cr, Ce, V, Sr, Ni, Zn, Ga and Y were detected. Abundance of Ce (93 ÷ 258 ppm) is a notable feature of the analysed samples.

The reuse of RM for the above purposes could provide sustainable and environmental friendly alternative to its mere disposal.

Keywords: bauxite residues; critical metals.

Photoactive TiO₂ nanoparticles for Cultural Heritage protection: investigation in lab and application in situ.

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Nanoparticles (NPs) of TiO₂ have been found to be particularly effective in the field of Cultural Heritage thanks to their photocatalytic activity which limits the formation of black crusts, saline efflorescence and prevents biodeterioration [1]. In this work rod-shaped TiO₂ nanocrystals (TiO₂ NRs) capped with oleic acid molecules (OLEA), were investigated for their application as a coating with self-cleaning, hydrophobic and photocatalytic properties for the conservation of limestone monuments [2] [3]. For this purpose the application of a nanocrystal dispersion was tested on a defined stone type known as Calcare di Bari in order to evaluate the physical properties of the stone before and after the treatment. The obtained results demonstrated the effectiveness of TiO₂ NRs as an active component in formulations for stone protection. This innovative treatment was applied, for the first time on cultural heritage, over the surface of the sarcophagus that has been monitored for the last year.

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Strength and durability studies on Mt. Etna volcanic precursors-based geopolymers

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In recent years, volcanic pyroclastic residues from Mt. Etna (ash and paleosoil named “ghiara”) have been widely employed as feedstocks in the alkali activation process [1, 2].

In the optic of building and restoration applications, strength and durability of volcanic ash and ghiara-based geopolymer (binders and mortars) have been investigated in terms of structure degradation, efflorescences development and water moisture transfer after being exposed to natural weathering [2].

Ultrasound Pulse Velocity, specific weight, Brazilian Disk and Digital Image Correlation together with Dynamic Vapor Sorption (DVS) techniques have been used in order to understand the structural properties of geopolymers before and after the atmospheric exposure. In addition, DVS results of pure salts, (i.e. sulphates and carbonates) which can often occur as efflorescence, were compared to the results obtained on geopolymers. Overall, by comparing the results obtained on unexposed and exposed samples, it was found that volcanic ash-based geopolymers have shown a better reaction to weathering in the hot summer Mediterranean climate zone.

Keywords: geopolymers, durability, sustainability

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Recycling of fine industrial waste in geopolymer foam mortar for new insulation materials

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Recently, the interest of companies in recycling waste from production operations has grown up, in the perspective of an increasingly sustainable and circular economy. Among the waste produced during the manufacture of building materials, the least recyclable fractions are the finest ones, obtained from cutting and polishing operations, which are usually landfilled.

In this study we tested the possibility to recycle finest waste fractions from companies dealing with ornamental stones and quartz composites. The aim is to obtain a lightweight geopolymer mortar for applications in the construction sector as insulation materials, thanks to the properties of geopolymer materials, as good mechanical properties, low thermal conductivity and environmental impact.

The industrial fine wastes (< 0.16 mm) were characterized by Powder X-Ray Diffraction and then added to a geopolymeric binder (metakaolin and K-silicate) and a foaming agent, to form the geopolymer foam mortar. The waste fraction accounts for about 30% wt. of the total sample and no natural materials were used.

Preliminary results indicate that sample density is between 1.09 and 1.24 g/cm³ (at 7 days) reaching 0.98 to 1.00 g/cm³ at 28 days, well below the limit for lightweight mortar products (< 1.5 g/cm³). Flexural and compressive tests vary in the ranges 1.4-2.7 MPa and 5.8-7.3 MPa (at 7 days), respectively. Compressive strength is 4.86-5.1 MPa at 28 days, showing properties interesting for lightweight insulating materials.

Keywords: fine industrial waste, geopolymers, lightweight materials.

Pollutants removal in aqueous solutions with geopolymers: a PhotoFenton case

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Environmental pollution is a serious threat to human health and the natural environment and has aroused widespread concern. One of the most effective processes in the removal of pollutants from wastewater is the Fenton reaction. This process is based on the production of highly reactive $\bullet\text{OH}$ radicals due to the rapid reaction between iron ions and hydrogen peroxide under acidic conditions [1].

The hydroxyl radical has a high oxidation potential of $E^\circ(\bullet\text{OH}/\text{H}_2\text{O}) = 2.8 \text{ V/SHE}$ at acidic pH, so they are extremely reactive and non-selective oxidizing agent towards organic contaminants in wastewater.

In order to avoid the drawbacks of a standard Fenton reaction, a solar photoFenton has been tested working at neutral pH in water in the removal of refractory pollutants.

Concerning the catalyst, a heterogeneous system was experimented, impregnating porous metakaolin-based geopolymers, obtained by using hydrogen peroxide as foaming agent in different ratios [2], with iron working as photocatalyst.

Keywords: photoFenton, geopolymers, wastewater treatment

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Recyclable Geopolymer Blocks for the Removal of Antibiotics from Wastewater

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The widespread diffusion of antibiotics in the environment is a compelling issue, as they have been widely detected, mainly in their unmetabolized form, in wastewater, surface water, drinking water and soils. In the search of efficient procedures for water decontamination, various methods have been applied. Adsorption processes are a promising option due to their characteristics of high efficiency, cost effectiveness, easy handling and integration to conventional WWTPs.

This research investigates the applicability of porous metakaolin-based geopolymers as adsorbents for the removal of antibiotic residues. Foamed geopolymers (see [1] for a review) with different porosities were obtained by adding commercial olive oil and H₂O₂ solution to enhance permeability to liquids and thus favour the absorption process. Samples were characterised by means of XRD, FTIR, gas pycnometry and BET analyses. Ofloxacin and ortisone were selected as model molecules to test the efficiency of foamed geopolymer samples.

Kinetic and equilibrium studies were performed in tap water to evaluate the adsorption properties of such materials towards the two antibiotics. The removal efficiency was also evaluated under representative conditions, viz. a few micrograms per litre river water. Satisfactory recoveries were obtained for all samples.

Keywords: porous geopolymers; antibiotic removal; wastewater.

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Synthesis and characterization of bottom wood ash metakaolin-based geopolymer.

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The waste left over after burning wood and wood products is known as wood ash (WA). 6 to 10 % of the ash produced by burning wood is disposed of in landfills[1], even though some wood ash may be recycled [1]. One of the alternatives to give this waste a second life is to use it as filler in geopolymeric materials. In this study, geopolymers (GP) are realized using metakaolin, NaOH, sodium silicate, and different percentages of WA (10, 20, 30%). The characterization of samples occurred at different aging times (7, 14, 28, and 56 days) to assess their mechanical, biological, and chemical properties. The article's research techniques include integrity, weight loss, water boiling test, pH and conductivity measurements, antibacterial activity, FT-IR analysis, moss growing test, and mechanical strength. The synthesized geopolymers are compact and solid. The pH and conductivity tests and the integrity and weight tests demonstrated the stability of the materials. The FT-IR study and boiling water test confirmed the successful geopolymerization in all samples. The antibacterial assay and the moss growing test showed antibacterial activity and gave a first idea about the durability of the materials. Finally, mechanical tests demonstrate how adding 10 and 20% of WA can improve the material's overall strength.

Keywords: Geopolymers; Wood Ash; Wastes.

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The effect of fibres on geopolymers made using Mt. Etna volcanic ash: a preliminary study

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The awareness of environmental issues has led to the development of eco-friendly materials that could combine high performance products with the possibility of the re-use of waste materials [1].

Geopolymers have favourable properties such as low curing temperature, recyclability and low cost of the precursors, making them a valid alternative to traditional OPC.

Despite these excellent qualities their brittle behaviour imposes constraints in structural design. To enhance the strength of the geopolymer matrix, both organic and synthetic fibres can be added to the geopolymeric matrices [2].

In this study two types of fibres were used to reinforce volcanic ash-based geopolymer.

Flexural and compressive strength tests were carried out in order to compare the mechanical properties of geopolymer composites. Moreover, samples were analysed by electron microscope to evaluate the gel formation and the adhesion of the fibre net to the geopolymeric matrix.

Preliminary results have shown that the addition of fibres enhances the mechanical strength and reduces the shrinkage.

Keywords: fibre-reinforced geopolymers, organic fibres, carbon fibres

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