MULTIFUNCTIONAL INORGANIC FOAMS BASED ON ALKALI-ACTIVATED MATERIALS



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The fabrication of inorganic foams with a porosity gradient is widely desired from scientists in order to achieve enhanced performances, mainly due to their low density, high strength and specific functional properties. Multifunctional inorganic foams represent innovative systems thought for specific and advanced functions, in which a spatial gradation in structure and/or composition lends itself to tailored properties and for this reason they find application in a broad range of high-tech fields such as energy, building, aerospace, filtration and bioengineering.



Supporting or shaping zeolites can represent a challenge to obtain this kind of systems. In fact, structuring a porous powder, as a zeolite, permit to obtain an optimized structure with high mass transfer, low pressure drops and high mechanical and chemical stability.

This research activity is focused on hybrid foams based on zeolite/geopolymer materials and characterized by hierarchical porosity.

The activity has been focused firstly on the analysis of the state of the art. Then, the research has been focused on the design of a multifunctional porous material combining the microporosity of the zeolites and the mesoporosity of the geopolymer matrix. In particular during the first year, the optimization of the operating conditions, by studying the effect of the foaming agent (silicon) content, relative humidity (RH %), and curing time on zeolite content in the sample, is performed

After the optimization phase and the consequently choice of the best sample, the activity of the second year has been focused on the study of main relevant properties for industrial applications. In particular water sorption capacity, porosity, BET surface area, mechanical properties, Cation Exchange Capacity, water softening capacity are performed.

Collected data demonstrated that it was possible to obtain macroscopic objects characterized by a hierarchical porosity, containing macro-, meso- and micro-pores by a quite simple process named Geopolymer Gel Conversion (GGC) starting from a metakaolin based geopolymer. Macroporosity is obtained by the foaming promoted by silicon,

mesoporosity is typical of the geopolymeric structure and microporosity is assured by the fact that zeolite crystals, grown after geopolymer setting by a low temperature hydrothermal treatment, covered the macro-pores walls. The zeolite crystallization occurred inside the geopolymer matrix starting from a curing time of 3 days, leading to two distinct crystal phases, Na-A [LTA] and 13X [FAU]. The controlled co-crystallization of mixtures of LTA and FAU, which is very interesting for detergent applications, can be obtained by tailoring the synthesis parameters. A positive correlation of both silicon content and relative humidity with the 13X growth was revealed, while, concerning the Na/A, an interdependent influence of both the process parameters was found. The nucleation and growth of both phases seems to be completed after 3 days of curing time. Beyond that period, no big alteration in the sample structure can be detected. The presence of a geopolymer backbone support and shape the zeolitic powder widening its technological application field.

These promising results suggest the possibility of using geopolymer-zeolite hybrid materials as bulk-type adsorbent (both in gaseous and aqueous system), self-supporting membranes, catalysts and gas separation.

The activity of the third year will be the shaping of a membrane-like or column-like geometry for ZEOP/1/100/3 to apply as bulk type adsorbent in aqueous system for continuous applications. Also, we aim to test the removal capacity of heavy metals.

In addition, to obtaining a controlled and homogeneous foaming process thanks to an under vacuum curing process or using a foaming stabilizer.

In order to obtain a zeolite monolith, the choice of zeolite (LTA or FAU) depends of the future specific application, curing parameters, such as temperature, pression, pH, relative humidity and raw materials will be modified individually or simultaneously. In fact, the properties of geopolymer-zeolite composites depend on the quantities and on the type of zeolites that formed in the geopolymer matrix, it seems possible to favour the formation of 13X zeolite [FAU] modifying the curing parameter. In particular, the Si/AI ratio must be greater than 1. For this, the next target could be the choice of raw materials with higher Si content, as silica smoke or fly ash. Also, the increase in the crystallinity of zeolite with increasing curing temperature will be investigate. On the other hand, it could be investigated the possibility of a two steps method to favour the crystallization of only 13X zeolite.

Published papers:

Campanile, A., Liguori, B., Marino, O., Cavaliere, G., De Bartolomeis, V. L., Caputo, D. (2020), Facile synthesis of nanostructured cobalt pigments by Co-A zeolite thermal conversion and its application in porcelain manufacture, Scientific reports, 10(1), 1-9, https://doi.org/10.1038/s41598-020-67282-1

Iucolano, F.; Campanile, A.; Caputo, D.; Liguori, B. Sustainable Management of Autoclaved Aerated Concrete Wastes in Gypsum Composites. Sustainability 2021, 13, 3961. <u>https://doi.org/10.3390/su13073961</u>

Attended conferences, oral presentations, posters:

XIV National Congress of Zeolites Science and Technology,11-14/06/2019, Amantea (CS), "Facile synthesis of nanostructured cobalt pigments by Co - A zeolite thermal conversion and its application in porcelain manufacture", Assunta Campanile, Oral presentation

XII INSTM conference-XV AIMAT conference, 21-24/07/2019, Ischia (NA), "Reuse of waste autoclaved aerated concrete granulates in gypsum-based building materials", Campanile A, Iucolano F, Liguori B, Caputo D, Poster

X Giornata di Studio Geopolimeri: Materiali green per l'ingegneria civile ed industriale-CETMA (Centro di Ricerche Europeo di Tecnologie Design e Materiali), 05/12/2019, Brindisi (BR), "Sistemi espansi geopolimero-zeolite per l'addolcimento delle acque", B. Liguori, A. Campanile, P. Aprea, G. Roviello, C. Ferone, Poster

Current and future challenges in advanced materials, sustainability, health and nanomedicine, Workshop IPCB (Institute for Polymers, Composites and Biomaterials) 14 – 16 Dicembre 2020, "Biocomposites and/or graft copolymers for drug delivery and cosmetics", M. Stanzionea, F. Tescione, B. Galzerano, A. Campanile, B. de Gennaro

Other relevant achievements:

14th "Glauco Gottardi" Degree Award, June 2019

Assunta Campanile, PhD student XXXIV cycle, May 2021