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2nd newsletter of the IRINEMA project

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Welcome to the second IRINEMA newsletter

We are pleased to announce the second issue of the IRINEMA newsletters. With this set of publications, we want to share the scientific knowledge derived from the project.

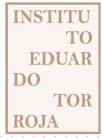
IRINEMA project, "Immobilisation of nuclear-grade ion resins in alkali-activated materials" (2019- T1 / AMB-13672), is financed through grants for the Attracting of Research Talent for the incorporation of research staff and carrying out research activities, awarded by the Community of Madrid. This project is carried out at the Eduardo Torroja Institute of Construction Sciences (CSIC).

The aim of this second issue is to share the main scientific contributions that have been made through IRINEMA in the second year of the project, as well as to highlight the work of the different collaborators who have contributed to it. In this publication, the reader will find an introduction about the studies carried out on new cementitious materials for the immobilisation of spent nuclear grade resins, and a section dedicated to the companies, universities and research centres that collaborate in the project, called "Our collaborators". In this second issue, we wanted to highlight the work of two collaborating centres, the Center for Energy, Environmental and Technological Research, CIEMAT, and the Materials Science Institute of Madrid, ICMM. Finally, there is also a list of news, meetings and conferences where IRINEMA has participated, as well as upcoming events relevant to the project.

We hope that the interested audiences will enjoy the content of these newsletters.









IRINEMA results

During these months, within the framework of IRINEMA, studies have been carried out mainly focused on the design of the base geopolymer. After the preparation and physicochemical characterization of the raw materials (blast furnace slag and fly ash), the proportions of aluminosilicate raw materials and the appropriate alkaline activators have been identified to obtain two types of geopolymers with high and low pH. In addition, the incorporation of superplasticizer admixtures has been considered in the design of the cementitious matrixes to improve the fresh state properties of the developed materials.

The following tests have been carried out with the designed cementitious matrixes:

- 1. Assessment of the fresh state properties of the cementitious matrixes and the influence on them of the use of a variety of admixtures with different nature (vinyl copolymer, melanine base and three polycarboxylates). Automatic Vicat needle test and minislump test were used to determine setting time and fluidity.
- 2. Analysis of the influence of admixtures in the cementitious matrixes and selection of the admixtures that optimize the fresh state properties of the geopolymers.
- 3. Determination of the flexural and compressive strengths of the selected geopolymers at 2, 7 and 28 days of curing.
- 4. Study of the reaction kinetics of the activation process of the selected geopolymers by induction calorimetry tests.
- 5. Determination of the porosimetry of the cementitious matrixes by Mercury Intrusion Porosimetry tests at 28 days of curing.

These advances in the design of cementitious materials for the immobilization of nuclear-grade resins can be extensively consulted in the two scientific publications that have been generated to date within the framework of the IRINEMA project:

- de Hita, M. J., and M. Criado. "Influence of admixtures on the workability and strength of sodium carbonate-activated slag/fly ash cement." *Materials Letters* (2022): 131695. <u>Influence of admixtures on the workability and strength of sodium carbonate-activated slag/fly ash cement ScienceDirect</u>
- de Hita, M.J.; Criado, M. Influence of the Fly Ash Content on the Fresh and Hardened Properties of Alkali-Activated Slag Pastes with Admixtures. *Materials* (2022), *15*, 992. Materials | Free Full-Text | Influence of the Fly Ash Content on the Fresh and Hardened Properties of Alkali-Activated Slag Pastes with Admixtures (mdpi.com)

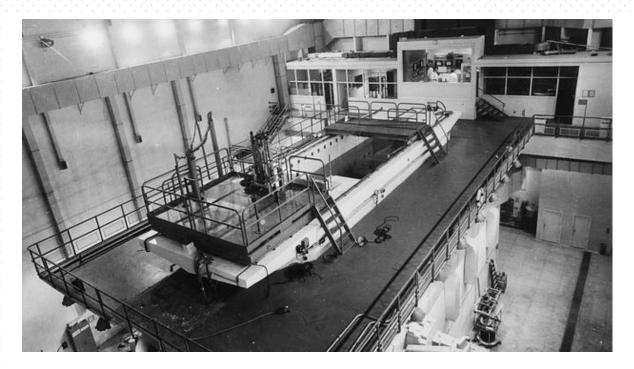
After the design of the base geopolymer, research work has focused on the first tasks concerning nuclear grade ion exchange resins:

- 1. CIEMAT-IRINEMA collaboration with the aim of developing the procedure and methodology to prepare the resin load that will simulate its operation in the PWR nuclear reactor primary circuit systems (dissolution design and preparation), and the way to incorporate the isotopes in the resin (resin loading conditions).
- ICMM-IRINEMA collaboration for the characterization of the cementitious matrixes before and after the incorporation of resin by Nuclear Magnetic Resonance tests of solids.

CIEMAT - IRINEMA Collaboration

CIEMAT (Centre for Energy, Environmental and Technological Research) is a Public Research Organization attached to the Ministry of Science and Innovation through the General Secretariat for Research. Its activity is mainly focused on the fields of Energy and Environment, and the technical aspects related to both.

CIEMAT is framed in an intermediate position in the path that goes from the generation of fundamental knowledge to industrial application. In this sense, it works closely with other national R & D & I centres, institutions, universities, and companies in the Energy sector. This collaboration is carried out in order to transfer the knowledge and technology that have been generated and, with it, promote innovation and change in the economic model based on knowledge.



JEN I nuclear reactor at CIEMAT headquarters (1971)

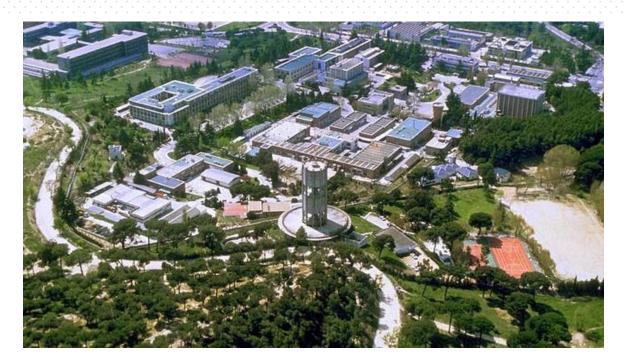
The R & D & I activity carried out by CIEMAT is developed at the national and international level with participation in EU programs and cooperation with intergovernmental organizations and research centres in other countries, especially with Latin America and the Mediterranean Basin. R & D & I actions are complemented by training activities, technology transfer, provision of advice and technical services to different administrations, and the representation of Spain in various international forums.

Since 1951, as the Junta de Energía Nuclear (JEN), and since 1986 as CIEMAT, it carries out R & D & I projects in the Energy area. Currently, the main lines of action are the study, development, promotion, and optimization of different energy sources: renewable, fusion, fission, and fossil fuels, as well as the study of their impact on the environment.

CIEMAT has extensive experience in R & D & I projects related to the management of radioactive waste, ranging from the radiological, physical, and chemical characterization of the waste, its conditioning, the durability of the barrier materials or the selection, characterization, and monitoring of sites for waste storage and disposal.

In particular, the Applied Environmental Geology Unit (Unidad de Geología Ambiental Aplicada) has as one of its priority lines of research the study of the compatibility of the different types of materials used in multi-barrier systems for the storage of radioactive wastes, as well as their durability under representative conditions of operation.

The collaboration of CIEMAT with the IRINEMA project arises from the common interest in exploring the improvement in the performance offered by the new formulations of alkali-activated cements compared to the current ones, based on Portland cements, for the conditioning of organic radioactive wastes. In particular, the main aim of the CIEMAT-IRINEMA collaboration focuses on the preparation and characterization of a surrogate radioactive waste of interest, in this case, spent ion exchange resins. With this purpose, protocols have been developed for the doping of the ion exchange resins according to representative water chemical conditions in the primary circuit of the reactor as well as its physical and chemical ones.



Aerial view of the CIEMAT headquarters in Ciudad Universitaria, Madrid.

ICMM – IRINEMA Collaboration

The Solid-state Nuclear Magnetic Resonance laboratory of the Materials Science Institute of Madrid collaborates actively with the IRINEMA project. The Materials Science Institute of Madrid (ICMM) is an Institute of the Spanish Research Council (CSIC), belonging to the Area of Matter. The aim of ICMM is to generate new basic and applied knowledge in innovative materials and processes and its transfer to the productive sectors of local, national and European scope (the true value of the materials is in their use), the training of new professionals in the materials field and the dissemination of scientific knowledge.

ICMM aims to become an international reference centre in the area of materials with high technological impact, which contributes effectively to the knowledge-based society. At the same time, ICMM should be a national benchmark for industry based on innovative technology with a special focus on the nano-scale and other cutting edge materials issues.



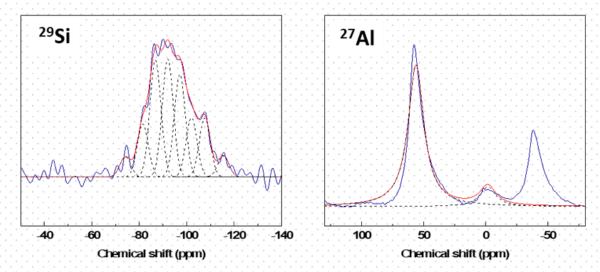


Left: Image of the ICMM, Cantoblanco university campus, Madrid, Spain. Right: 400 MHz NMR spectrometer for solid-state samples.

ICMM has a wide range of research support services necessary for the characterisation of materials. One of them is the laboratory of solid-state Nuclear Magnetic Resonance (NMR). It was the first laboratory in Spain dedicated to NMR of solids that started its work in 1986. From the beginning, it was a service open to the entire scientific community, which has promoted the establishment of collaborations between ICMM researchers and other research centres and companies. This has contributed to an enrichment of its own research activity and, at the same time, to an increase in the visibility of the institute and its groups within the national and international research community, holding the scientific leadership in the study of disordered systems.

The use of high-resolution NMR in solids is one of the main objectives pursued when characterising ceramic and glass materials, catalysts, cements, polymers, ionic conductors, etc. since it allows to study the structural properties of a wide variety of amorphous or little crystalline materials. Studies of amorphous precursors of cements based on SiO₂-CaO y Al₂O₃-CaO has been carried out at the ICMM. In this regard, the researchers responsible for the NMR service, Dr. Jesús Sanz and Dra. Isabel Sobrados,

have been collaborating for many years and very actively with researchers from the IETcc of the CSIC, which has led to numerous articles with a large number of citations. The analysis of the ²⁷Al and ²⁹Si spectra of different systems makes possible to study the evolution of the crystalline and amorphous phases of the raw materials, as well as the hydration and alkaline activation and their reaction mechanisms.



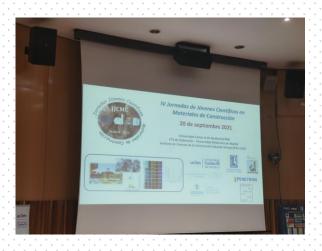
²⁹Si and ²⁷Al MAS NMR spectra of alkali-activated ashes made at NMR of solids laboratory of the ICMM by Dr. Criado.

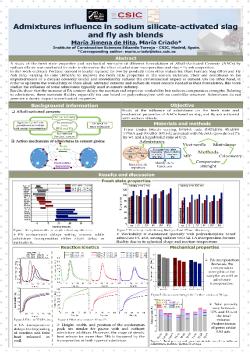
In particular, IRINEMA-ICMM collaboration aims to carry out Nuclear Magnetic Resonance studies at ICMM of the materials developed at IRINEMA. The purpose of these studies is the determination of the local structure of the gels formed in the alkaline activation of blast furnace slag and fly ash as immobilisation matrixes of spent ion exchange resins. These studies are closely related to the mechanical behaviour and the final properties of the cementitious matrixes that ensure the nuclear waste immobilisation and its safe long-term management.

News and IRINEMA participations

IV Conference of Young Scientists in Construction Materials

For the fourth consecutive year, the Carlos III University of Madrid, the ETS of Building of the Polytechnic University of Madrid, and the Eduardo Torroja Institute of Construction Sciences (IETcc-CSIC) have carried out the Conference of Young Scientists in Construction Materials, which this year took place at UC3M on September 20, 2021. IRINEMA has had the pleasure of participating in this fourth edition through the presentation *Diseño de matrices cementantes con menor huella de carbono para la inmovilización de residuos sólidos orgánicos de grado nuclear*.





75th RILEM Annual Week

From August 29 to September 3, 2021, the 75th week of RILEM was held in Mérida, Mexico, during which meetings of RILEM's permanent committees and several meetings of the technical committee were held. The International Conference on Advances in Sustainable Building Materials and Structures also took place during this event, intending to bring together the latest developments to improve quality assurance in construction materials and tests and in which IRINEMA has participated through the poster presentation Admixtures influence in sodium silicate-activated slag and fly ash blends

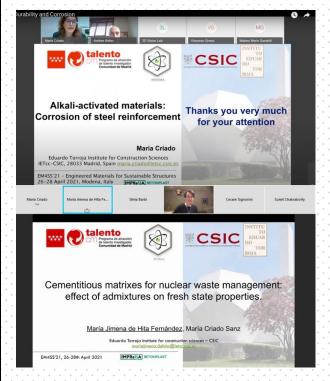
Within the framework of the RILEM week, different PhD courses were also held, which we have attended: *Alkali Activated Materials* and *Hydration and microstructural characterization of cementitious systems*.

2nd VitroGeoWastes

From May 24 to 26, 2021, the second VitroGeoWastes Congress was held, organized by the University of Andalusia and which took place in Baeza, Jaén, Spain. The topics of the congress were vitrification, geopolymerization, waste management, "green" cement, and circular economy. IRINEMA has participated in this second edition of VitroGeoWastes through two presentations, one given by Dr. María Criado, Design of alkali-reactivated fly ash matrixes to encapsulate ion exchange resin radioactive wastes, and the other from PhD student M. Jimena de Hita, Effect of admixtures on the fresh state properties of sodium carbonate-activated slag/fly ash cement.



News and IRINEMA participations



EM4SS'21 – Engineered Materials for Sustainable Structures

The Engineered Materials for Sustainable Structures Congress, organized by the University of Modena within the framework of the IMPReSA project and held from April 26 to 28, 2021, aimed to provide a unique forum for researchers, companies, and professionals to exchange ideas on new materials and structures. The six thematic sessions addressed different aspects in the field of engineering materials, with the main objective of establishing a meaningful dialogue between the various scientific communities and the business world. IRINEMA has participated in the EM4SS'21 through two presentations: Dr. María Criado presented the talk Alkali-activated materials: Corrosion of steel reinforcement and PhD student M. Jimena de Hita presented the talk Cementitious matrixes for nuclear waste management: effect of admixtures on fresh state properties.

Life Hypobrick - IRINEMA collaboration

Collaboration between scientific projects contributes beneficially to the development of knowledge. For this reason, IRINEMA and Life Hypobrick projects have held networking sessions to promote collaboration between the researchers of both projects. The main objective of Life Hypobrick project is to demonstrate the reliability of the manufacture of waste-based building products (bricks and blocks) using an extremely low CO2 emissions process.

The minutes of the meetings can be consulted here:

Meetings from 10 to 15 February 2021

Life Hypobrick - IRINEMA networking

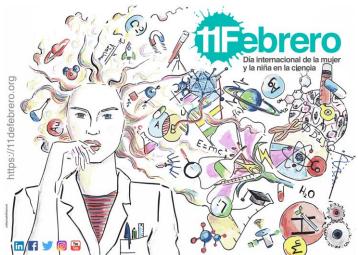
Meeting 14 September 2021

Life Hypobrick - IRINEMA 14 September 2021



Upcoming events

February 11th – International day of Women and Girls in Science February 11th - International day of Women and Girls in Science 11th February | Madrid, Spain



Picture by @mariadelalamort for February 11th

February 11 is a citizen initiative to make visible the work of women who are dedicated to STEM (Science, Technology, Engineering and Mathematics) areas, thus creating female references for childhood that can contribute to the choice of these areas as professional careers and know the different factors that affect the current situation of women in STEM areas to promote practices that lead to their elimination and achieve gender equality in the scientific field.

Alkali Activated Materials And Geopolymers: Sustainable Construction Materials And Ceramics Made Under Ambient Conditions

ECI Conference - Alkali Activated Materials and Geopolymers

From 29th May to 3rd June 2022 | Calabria, Italy

The 3rd ECI International Conference, "GEOPOLYMERS and Alkali Activated Cements: Sustainable construction materials and ceramics made under ambient conditions", planned for the year 2022, aims to collect scientific and industrial contributions, to find technical solutions enabling the application of geopolymers and related materials, to reduce waste and greenhouse gas emissions in ceramic and cement manufacturing, and to provide high-performance, scalable, ceramic materials.

XVI National Congress of Materials CNMAT22

Congreso Nacional de Materiales 2022 From 28th June to 1st July 2022 | Ciudad Real, Spain



The 16th National Congress of Materials CNMAT22 organized by the University of Castilla La Mancha will take place in Ciudad Real and will be a meeting forum around the latest advances in processing, properties, characterization, and behavior of materials with applications in multiple technological fields; structural and functional materials, sustainable materials, materials for ICTs, energy, health or construction, among many others.