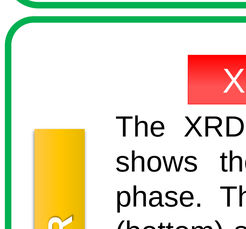
Synthesis and characterization of new mesoporous materials based on organoaluminosilicates

VIII Trobada d'Estudiants de Doctorat
VIII Encuentro de Estudiantes de Doctorado

C. Esteban, U. Diaz
Instituto de Tecnología Química, Universitat Politècnica de València-Consejo Superior de Investigaciones Científicas,
Avenida de los Naranjos s/n, E-46022 Valencia, Spain.
cestbar1@itq.upv.es
Doctorado en Química Sostenible

The development of sustainable and efficient chemical processes is one of the highest priorities in contemporary society. For this reason, the use of heterogeneous catalyst with reusable capability of performing such processes in a single step could avoid the isolation of intermediates as well as the recovery and disposal of by-products and solvents. Herein, we report the synthesis of a new family of mesoporous materials type SBA-15 by co-condensing a previously synthesized organoaluminosilicate with varying amounts of an inorganic silica source.

MESOPOROUS MATERIALS

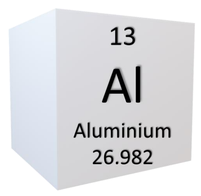


- ✓ High specific area.
- ✓ Large pore volume.
- ✓ Tailorable properties.

ORGANOSILICAS

- ✓ They are used to change the surface polarity and functionality of the materials. [1]

HETEROATOMS

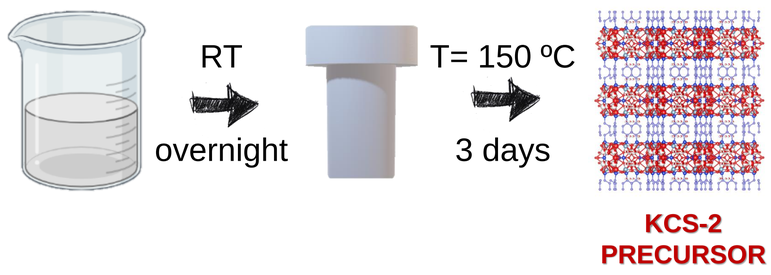


- ✓ Provide acidic or redox sites into pure silica materials according to the desirable application. [2]

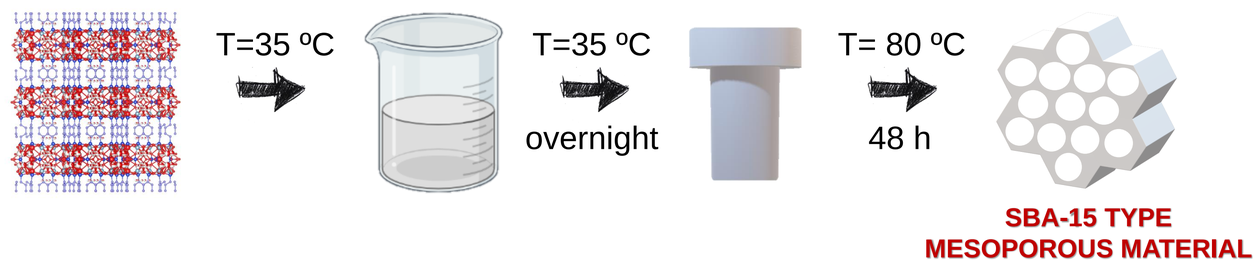
SYNTHESIS

The synthetic protocol is based in a two-step approach:

(1) Synthesis of organoaluminosilicate precursor through hydrothermal synthesis. [3,4]



(2) Preparation of SBA-15 type mesoporous materials by co-condensing a previously synthesized organoaluminosilicate (KCS-2 type) with varying amounts of an inorganic silica source using P123 as template.

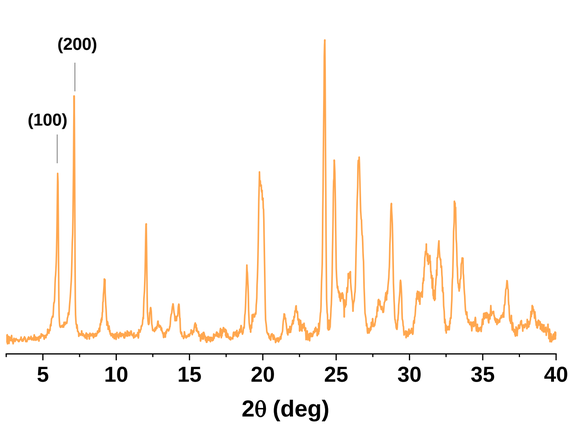


CHARACTERIZATION RESULTS

KCS-2 PRECURSOR

X-RAY DIFRACTION

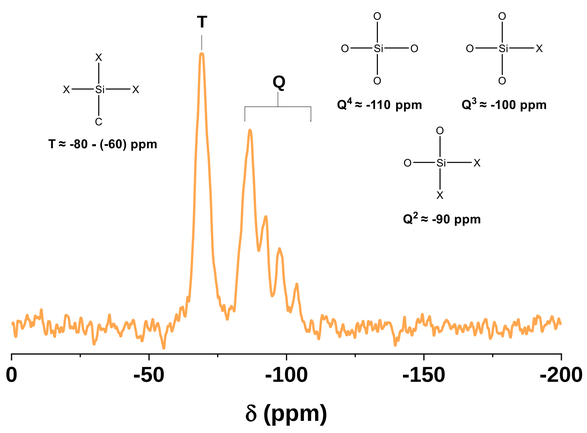
The XRD pattern of the precursor (top) shows the peaks associated to KCS-2 phase. The XRD pattern of the material (bottom) shows that the structure is similar to SBA-15.



SOLID STATE NMR

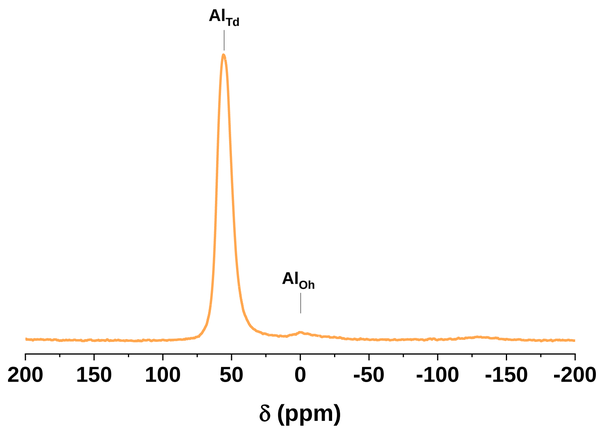
^{29}Si-NMR

The ^{29}Si-NMR spectra confirm the presence of two types of silicon, one organic (T) and the other one inorganic (Q).



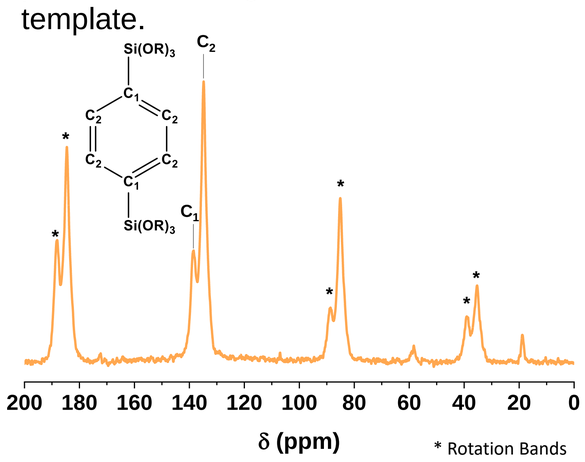
^{27}Al-NMR

The ^{27}Al-NMR spectra confirm the incorporation of aluminium in the framework.

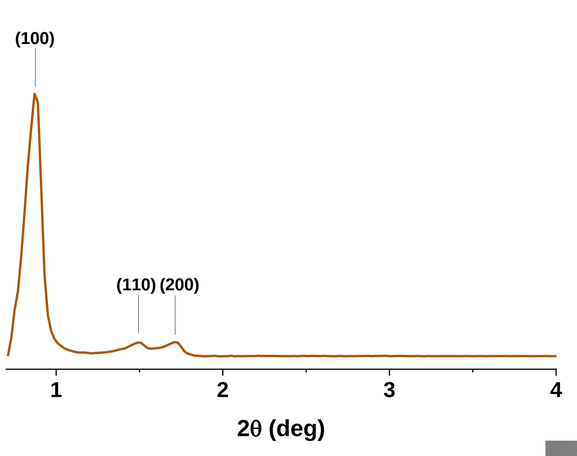


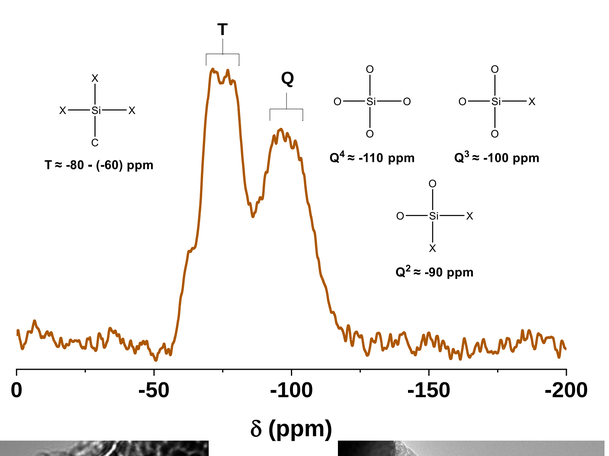
^{13}C-NMR

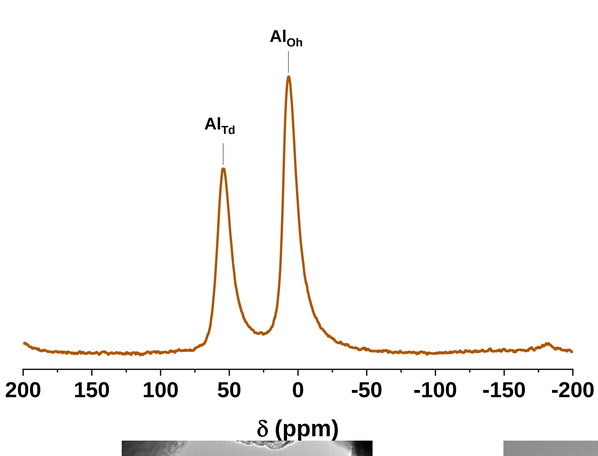
The ^{13}C-NMR spectrum of both materials shows the aromatic carbon signal. Furthermore, the SBA-15 material presents the signals correlated to the template.

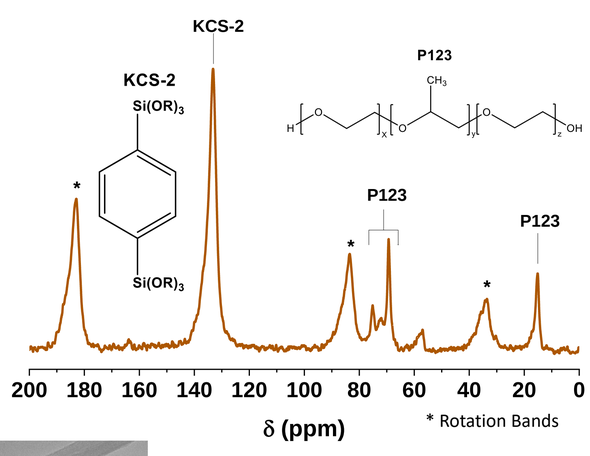


MESOPOROUS MATERIAL SBA-15 TYPE

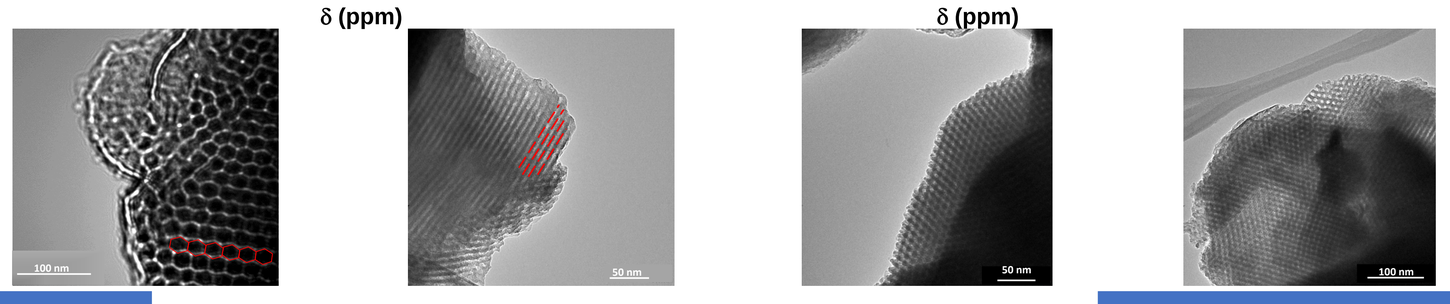








HR-TEM



The HR-TEM images show the channels and hexagonal pores of the material.

CONCLUSIONS

- ✓ Development and characterization of a new family of hybrid mesoporous materials by co-condensing a previously synthesized organoaluminosilicate used as precursor together with an inorganic silica source.
- ✓ Due to the presence of framework heteroatoms, these materials are expected to have interesting catalytic applications.

REFERENCES

[1] H. Moon, S. Han and S. L. Scott, *Chem. Sci.*, 2020, 11, 3702–3712.
[2] I. Rakngam, N. Osakoo, J. Wittayakun, N. Chanlek, A. Pengsawang, N. Sosa, T. Butburee, K. Faungnawakij and P. Khemthong, *Microporous Mesoporous Mater.*, 2021, 317, 110999.
[3] K. Yamamoto, A. Irisa, M. Kawano and T. Ikeda, *Chem. Lett.*, 2014, 43, 376–378.
[4] T. Ikeda, N. Hiyoshi, S. Matsuura, T. Kodaira, T. Nakaoka, A. Irisa, M. Kawano and K. Yamamoto, *Angew. Chemie*, 2015, 127, 8105–8109.

ACKNOWLEDGMENTS

The authors are grateful for financial support for Spanish Ministry (project PID2020-112590GB-C21). C. Esteban has been partially supported by the Spanish Government by FPI pre-doctoral scholarship number PRE2019-089547 under project SEV-2016-0683-19-3.