

New Hybrid organochlorinated xerogels

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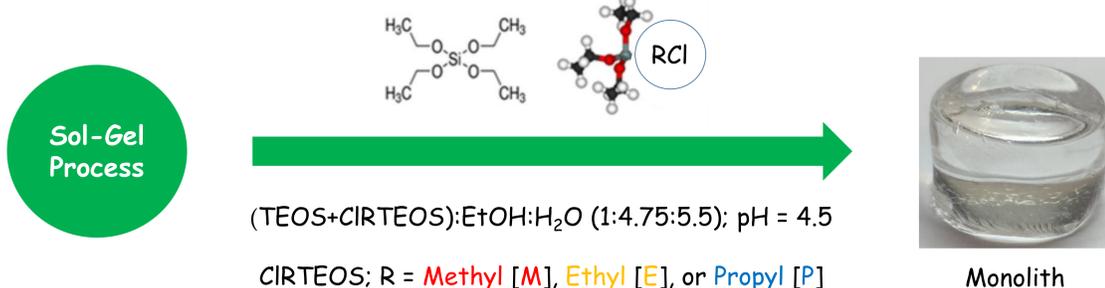
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Introduction

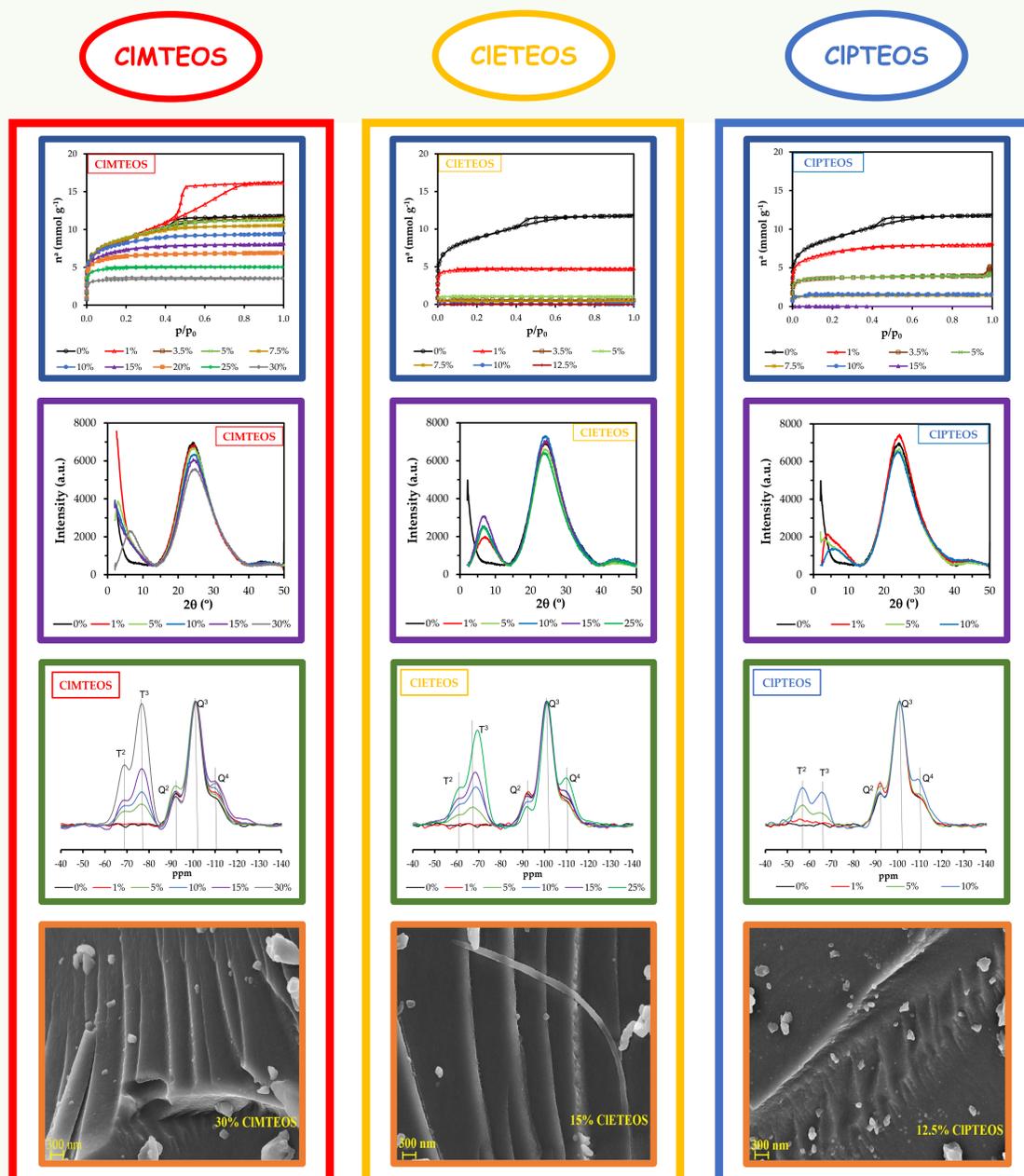
Hybrid silica xerogels (HSXG) combine the properties of organic and inorganic components in the same material, which makes them promising and versatile candidates for multiple applications. These materials can be easily prepared by the sol-gel process, which offers the possibility to obtain different morphologies. The incorporation of organic precursors plays an important role in their properties, hence, allowing the design of materials for specific applications such as coatings for optical fibers [1]. The aim of this work was to study the influence of the alkyl chain and chlorine atom on the morphological and textural properties of various hybrid materials produced by co-condensation. For this purpose, three series of hybrid xerogels were prepared by co-condensation of TEOS and a chloroalkyltriethoxysilane (TEOS:CI RTEOS, R = methyl, ethyl or propyl) at different molar ratios. The influence of the precursors on the structure and textural properties of the xerogels was studied by means of N₂ adsorption, XRD (X-ray diffraction), ²⁹Si NMR (nuclear magnetic resonance) and FE-SEM (Field Emission-scanning electron microscope) [2].

Methods

All the hybrid xerogels were prepared with 1:4.75:5.5 molar ratio of (TEOS+CI RTEOS):EtOH:H₂O (TEOS= tetraethoxyorthosilicate; CI RTEOS= Chloroalkyltriethoxyorthosilicate, being R= methyl, M; ethyl, E; propyl, P), and the pH was adjusted to 4.5. After stirring for 10 min, the alcogel was introduced in an oven at 60 °C until gelation, cured for one week with EtOH and dried at room temperature until there was not significant change in the weight of the monolith.



Results



Conclusions

The study reveals that the properties of the synthesised materials greatly depend on the organic precursor.

As a way of example, the material prepared with 5% molar percentage of CIMTEOS is mesoporous, whereas its counterparts synthesised with the same percentage of CIETEOS and CIPTEOS remain microporous. In addition, the microporosity of the three series increases with the percentage of organic precursor (N₂ Adsorption isotherm and FE-SEM).

The chlorinated precursors also promote the formation of ordered domains within the silica matrix, as is evidenced by: i) the increase of more condensed species observed on the ²⁹Si NMR spectra, and ii) the XRD peak at 2θ < 10° associated with the formation of ordered structures composed of T₈ cages or ladder-like structures. This peak is observable at lower molar percentages of chloroalkyl precursor than in those materials prepared with non-chlorinated analogues (MTEOS, ETEOS and PTEOS) [2].

The synthesis of these new locally nanostructured hybrid materials with tuneable porosities opens up a promising path for their use in a wide range of applications such as membranes, conductive films, absorbents, catalysts, optoelectronics, and coatings for optical sensors.

References

[1] Echeverría, J. C.; Calleja, I.; Moriones, P.; Garrido, J. J. Fiber Optic Sensors Based on Hybrid Phenyl-Silica Xerogel Films to Detect n-Hexane: Determination of the Isosteric Enthalpy of Adsorption. *Beilstein J. Nanotechnol.* **2017**, *8*, 475 - 484.

[2] Cruz-quesada, G.; Espinal-Viguri, M.; López-Ramón, M. .; Garrido, J. Novel Organochlorinated Xerogels: From Microporous Materials to Ordered Domains. *Polymers*, **2021**, *13* (09), 1415.