# INNOVATIVE PROCEDURE FOR THE PREPARATION OF SILICA FILLINGS WITHOUT SHRINKAGE

PATENTED TECHNOLOGY

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## ABSTRACT

The research group Carbon Materials and Environment which belongs to the Inorganic Chemistry Department and the University Materials Institute of the University of Alicante has developed a procedure to prepare silica fillings preventing their shrinkage by the deposition of a thin film prior to the synthesis of the filling. Modifying the said thin film (microporous or mesoporous) the filling properties are modified, obtaining highly satisfactory results in the case of the mesoporous thin films in all case studies (different supports). This method also allows obtaining silica fillings with different diameters and their incorporation into different types of supports such as glass, steel or honeycombing cordierite monoliths.

Silica fillings are nowadays employed in applications as for example stationary phase in chromatography columns, support for the immobilization of bioactive molecules, catalysts and/or catalyst supports, polymer fillers, etc. Following this procedure the shrinkage of the fillings is avoided, which is a common occurrence with the established preparation protocols.

## INTRODUCTION

From the first synthesis of a mesoporous silica in 1991, the interest in this solid has grown steadily, as well as the number of applications in which it could implemented. Mesoporous silica is an inorganic compound based on silicon oxide which, due to the synthesis conditions in which it has been prepared, presents a type of porosity centered in the mesopore range (pore size between 2 and 50 nm).

The success of mesoporous silicas either as filling, powder, or thin film mainly derives from the simplicity of their synthesis, in the variety of materials on which they may be supported, and their physico-chemical properties (controlled porosity, high surface area, tunable surface chemistry, mesopore morphology…). There are already applications in which it has demonstrated its worth as a material. Among the various applications in which it has already been implemented, we may mention the following:

- Stationary phase in HPLC (High Performance Liquid Chromatography) columns.
- Support for the immobilization of bioactive molecules.
- Catalysts and/or catalysts support.
- Other applications where mesoporous silicas are important is as polymer fillers or as structure-directing agents for the formation of other materials.

## TECHNICAL DESCRIPTION

This new synthetic procedure is based on the incorporation of a porous silica thin film on the walls of the employed supports prior to the synthesis of the filling, with the objective of improving the adhesion of the silica avoiding the shrinkage taking place during the last stages of the sol-gel process.

The development consist on a procedure for the preparation of silica fillings with meso- and/or macroporous structure (hierarchical structure) in the inner volume of different supports made up of different materials and with different inner diameters completely avoiding the shrinkage of the silica, which is divided into the following steps:

a. deposition of a silica thin film on the walls of the support used for filling,
b. preparation of the filling precursor solution with the necessary porogen precursors,
c. synthesis of the silica material which serves as filling in the inner volume of the support.

The employed supports for the filling with hierarchical silica are, a) honeycomb cordierite monoliths, b) glass tubes, c) stainless steel tubing, and d) fused silica capillaries. All the materials present a wide range of compositions (cordierite, glass, fused silica, and steel) and a wide range on internal channel diameter (between 0.25 and 7.28 mm).

*Figure 1. Selected materials, from left to right: Fused silica capillary (i.d. 0.25 mm), stainless steel tubing (i.d. 1.6 mm), glass tube (i.d. 7.6 mm), and multichannel cordierite monolith (i.d.channel 1.2 mm).*

## ADVANTAGES AND INNOVATIVE ASPECTS

### MAIN ADVANTAGES OF THE TECHNOLOGY

Previous deposition of a thin film allows:
- Avoiding shrinkage, thus reducing the detachment of the monolith provoked by this effect.
- Increasing the adherence of the monolith to the employed support walls.
- Process can be applied to different supporting materials.

### INNOVATIVE ASPECTS OF THE TECHNOLOGY

- Method based on sol-gel methodologies, modified to overcome shrinkage and detachment phenomena.
- Use of simple silica precursors which are furthermore common to all supports.
- Use of environmentally sustainable chemicals.
- Extractions with organic solvents are avoided.
- Supports with inner diameters larger than those found in the literature.
- Superior mechanical stability of the prepared filling.

## CURRENT STATE OF DEVELOPMENT

The developed procedure has been tested successfully at laboratory scale with the different aforementioned supports having different diameters, obtaining highly satisfactory results in all cases.

## MARKET APPLICATIONS

This technological development may be applied in:
- Development of standard or HPLC columns.
- Production of catalytic reactors and microreactors.

## COLLABORATION SOUGHT

The research group seeks companies interested in acquiring this technology for its commercial exploitation through different technology transfer pathways:

- Patent licensing agreements.
- Development of joint R+D Projects for the adaptation of the developed technology to the industry needs.

- Technical cooperation, subcontracting and R+D consultancy.

## INTELLECTUAL PROPERTY RIGHTS

This technology is protected by the following patent application.

- Title: Procedimiento de preparación de rellenos de sílice que evita el encogimiento de éste mediante depósito previo de una película delgada de sílice
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## MARKET APPLICATION (2)

Materials and Nanotechnology
Chemical Technology