DEVELOPMENT OF INNOVATIVE COMPOSITES VIA SOL-GEL PROCESS

Jessica Passaro – Advisors: Prof. Francesco Branda, Prof. Giuseppe Mensitieri

Curriculum: Ingegneria dei Materiali e delle Strutture

Material science represents a branch of the natural sciences that becomes crucial in a world dominated by the importance of the choosing of suitable material. New materials are required for a wide variety of applications, which have to be extremely specialized and at the same time sustainable and economically affordable. High technology materials are required in every technological field, ranging from opto-electronics or bio-materials for medical application to adsorbent materials, and they must be designed or invented with great care.

In this scenario the use of hybrid materials opens new routes to the production of innovative materials, whose properties can be tailored depending on the demands for the different applications. Their properties, in fact, can be chosen so as to obtain the best performances of the organic materials and the inherent stability of the inorganic ones. In particular, when the two phases are structured on a micro- and nanometer scale, the result can be considered as a new material with desired properties and a structure that can diverge from those of the starting components.

The sol–gel synthesis route has been extensively exploited since the 1970s, in combination with polymer synthesis methodologies, to produce not only inorganic materials (glassy or ceramic) but also hybrid organic/inorganic (O/I) composites in the form of aerogels, monoliths, coatings, fibers, and particles. The strategy takes advantage of the fact that almost all the important oxides MOn (where M is a metal or semimetal and n is not necessarily an integer), as well as many mixed oxides, have been prepared by the sol–gel process through reactions occurring at low temperatures starting from precursors that are commercially available at high purity. Very mild reaction conditions and low reaction temperatures are particularly useful for incorporating inorganic filler into organic materials or organic materials into inorganic matrices.

In this PhD work the sol-gel methodology has applied to solve some technological problems inherent to the use electropsun polymer composites for industrial applications.

These electrospun mats can show some limitations:

- low resistance to organic solvents and thermal instability (i.e. shrinkage) of the electrospun fiber;
- easy flammability of the polymer matrix that can significantly restrict the application fields of these materials, especially when the possibility of the use of the mats is strictly related to specific regulatory fire tests that have to be passed, hence ensuring public safety (e.g., in the aerospace industry).

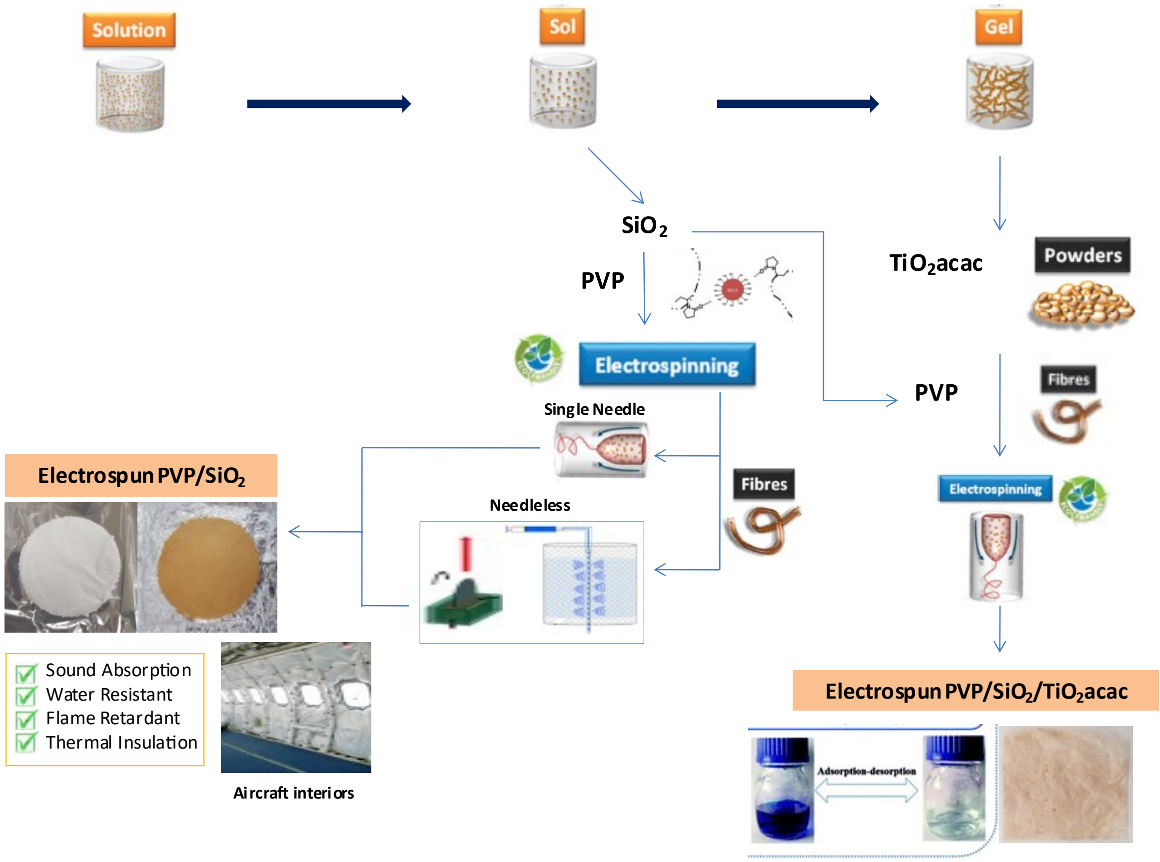
Sol-gel methodologies can improve the fire behavior, the resistance to organic solvents and thermal instability of electrospun composites through the use of sol-gel particles added into polymer solution.

The first and second years of the PhD was primarily focused on the study and characterization of PVP_silica electrospun composite. The silica particles, 250 nm in diameter, were produced through the Stöber method. Thin disks of Silica/Polyvinylpyrrolidone (PVP) composite were obtained through electrospinning. A second series of disks was obtained by submitting the electrospun mats to proper thermal treatment. The heat treated samples became highly water resistant without suffering shrinkage. Moreover, they passed the 12-s Vertical Bunsen Burner Tests specified in Federal Aviation Regulation (FAR) 25.853 and FAR 25.855 and the smoke density test described in the Aircraft Materials Fire Test Handbook. The experimental results prove that the large addition of silica (66% of fiber mass) does not change the general features of sound absorption behavior of the mats. Also in the present case, for each set of disks (from a minimum of 6) the sound absorption coefficient changed with the frequency (in the range 200–1600 Hz) following a bell shaped curve with a maximum (where the coefficient is greater than 0.9) that shifts to lower frequencies by increasing the piled disks number. A tunable acoustical response was therefore also obtained in the presence of silica

Then, various techniques were used to characterize the samples: SEM, ATiR, Dynamic Light Scattering, Vertical Burner Bunsen Test, Smoke Density test, DSC, Acoustic Impedance.

In this thesis, great attention was laid on the presence and role of sol-gel particles as filler of composite electrospun fibers. For this reason, the research continued in the third year with the production of new electrospun composite material adding hybrid titania sol-gel particles to polymer solution. In particular, TiO2-acetylacetonate (TiO2acac) amorphous xerogels have shown remarkable performance in the oxidative degradation of different aqueous organic pollutants (phenanthrene, 2,4-dichlorophenol, chlorophenoxy herbicides) without any need of light irradiation. This uncommon catalytic activity makes these hybrid materials potentially promising for water decontamination, abatement of air pollutants and antimicrobial coatings.

Then, various techniques were used to characterize the samples: SEM, TEM, TGA, DSC, Zeta Potential, EPR, Dye removal Test.



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