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Hygro-thermal properties of silica aerogel blankets dried using microwave heating for building thermal insulation

Kevin Nocentini[a,*], Patrick Achard[a], Pascal Biwole[a,b]

[a] MINES ParisTech, PSL Research University PERSEE – Centre procédés, énergies renouvelables et systèmes énergétiques, CS 10207 rue Claude Daunesse 06904 Sophia Antipolis Cedex, France
[b] Université Clermont Auvergne, CNRS, SIGMA Clermont, Institut Pascal, F-63000 Clermont-Ferrand, France

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ABSTRACT

Silica aerogels are highly porous and open-cell materials made of amorphous silica nanoparticles, interconnected in a 3D random network. Silica aerogel-based materials have a great potential as thermal insulation in building thanks to their very low thermal conductivity. However, pure silica aerogels are fragile with low mechanical moduli. Making aerogel composite materials by combining fibers with a pre-gel mixture of a gel precursor or by impregnating a fiber network by such a mixture seems to be a promising way to enhance the mechanical properties of such materials. After drying, the resulting composite is called aerogel blanket. The aerogel blanket is mechanically strengthened, flexible and still has a very low thermal conductivity. Aerogel blankets are usually dried using supercritical process but it is considered as a main drawback for large scale industrialization. The present study uses an innovative micro wave drying. The purpose of this work is to analyze and characterize a handy, light, super-insulating aerogel blanket dried in ambient conditions and see if it could be suitable for building thermal insulation. Two types of blankets have been investigated: the first one with a glass fiber web and the second one with a PET (polyethylene terephthalate) fiber web. Hygro-thermal characterizations were done and show that the aerogel blankets have an excellent thermal conductivity (0.015 $\mathrm{W\,m^{-1}\,K^{-1}}$) and a hydrophobic behavior. The studied aerogel blankets obtained using a new ambient drying process show practically the same characteristics as their counterpart dried with a supercritical process and mark a step forward in the aerogel blanket industrialization.

1. Introduction

The current global energetic context is characterized by a need to reduce our energy consumption, to delay the fossil fuel shortage, and to slow down greenhouse gas emissions. Recently, there has been a tendency of the national governments to release more energy saving policies, especially in the energy-intensive sectors. According to the Directive 2012/27/EU of the European Parliament and the Council efficiency (2012) [1], the building sector is responsible for 40% of the primary energy consumption in Europe. Increasing thermal insulation of the building envelope remains the most efficient way to reduce this consumption [2,3]. Thermal insulation permits reducing the heat exchange from inside to outside during heating and cooling periods while maintaining comfort for the occupants. Conventional insulation materials for buildings are mineral or organic wools and polymer foams, with thermal conductivities between 0.030 and 0.060 $\mathrm{W\,m^{-1}\,K^{-1}}$ [4]. To improve the overall thermal insulation, one can either increase its thickness or decrease its thermal conductivity. Indoors insulation's thickness is typically limited to a few centimeters, especially in urban areas where the living space is scarce and expensive. Decreasing insulations' thermal conductivity has been studied for years, and we have seen the emergence of a new kind of insulation materials called "super-insulating" materials, with a thermal conductivity lower than that of still air (0.025 $\mathrm{W\,m^{-1}\,K^{-1}}$).

Vacuum insulation panels [5] and ambient pressure aerogel blankets [6] are the two main types of super insulating materials and some commercial products already exist [7–11]. However, they are not yet competitive on the building insulation market because of their manufacturing costs, and they are mostly used for particular insulation needs. The latest research efforts are focused on the reduction of time and cost of the manufacturing process of such super-insulating materials.

Silica aerogels were historically elaborated by Kistler in 1932 [12] by using the sol–gel process, and by removing the solvent via supercritical drying. The resulting material was mesoporous,

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* Corresponding author.
E-mail address: kevin.nocentini@mines-paristech.fr (K. Nocentini).

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nanostructured and formed of silica nanoparticles in an open-cell network, with pore sizes from 2 nm to 100 nm. These tiny pore dimensions are responsible for the Knudsen effect and allow obtaining thermal conductivities down to 0.014 $W\,m^{-1}\,K^{-1}$ at ambient conditions. However, the methods used at that time were very tedious and time-consuming. Silica aerogels experienced a revival in the 1960s with the progress of sol–gel chemistry. Peri's works [13] permitted replacing the $NaSiO_3$ precursors initially used by Kistler by alkoxide precursors and was the first to open the route with tetraethyl orthosilicate (TEOS) precursor which is at present mostly favored. More extensive works lead by Teichner group [14] were focused in this direction with the replacement of water by an organic solvent. These previous works have made it possible to further simplify the synthesis of the material. In the 1980's, the alkoxide sol-gel process was made less toxic by using a safer alkoxide compound and the supercritical drying technique was made safer by replacing supercritical alcohol with supercritical carbon dioxide thanks to the works of Tewari et al. [15]. With these developments, interest in commercializing aerogels emerged in many sectors, including thermal insulation. However, the supercritical drying step has been considered as a major drawback for large-scale industrialization because it is a long discontinuous process that can only dry a batch of material at each operation. Nevertheless it is essential to avoid capillarity stresses in pores during the solvent extraction which would lead to the collapse of most of the pores. At this time, the importance of finding an ambient pressure drying technique for industrial production appeared in the collective consciousness. In the early 1990s, researchers at the University of New Mexico [16] found how to avoid the pore collapse during the ambient drying by grafting uncondensable groups (silylation) on the surface of the material and by taking advantage of the spring back effect [17], meaning that the material returns to its initial dimensions after the relaxation capillary stress. However, the resulting global material is divided (granular beads or powder) because of the brittleness of the silica network. This technique was later patented by Cabot Corporation [11] which commercialized in 2001 the first silica aerogels for thermal insulation in the form of translucent granules under the tradename Nanogel. The same year, Aspen Aerogels [9] developed a novel flexible aerogel composite blanket by casting silica gel onto a fibrous padding then supercritically drying the blanket. The resulting material, so-called "aerogel blanket" was robust unlike monolithic silica aerogel thanks to the fibrous network which permits getting a global cohesion and macro flexibility in the material. Both companies have developed a range of optimized products for thermal insulation needs and share almost exclusively the aerogel market today.

For the present study, we consider aerogel blankets obtained thanks to an innovative ambient drying process based on microwaves, patented by the Enersens Company [18]. Microwave energy helps to control the drying process more accurately to achieve greater yields and better quality products in a shorter time. In classical evaporative drying, heat is first transferred to the surface of the material by conduction, convection or radiation and afterward into the interior of the material by thermal conduction. The solvent is initially flushed off from the surface, and the remaining liquid diffuses to the surface. The process time is restricted by the rate of the heat flow into the body of the material from the surface as determined by its physical properties. This technique removes surface solvent very fast, but it is ineffective when it comes to removing the solvent confined inside the material. Microwaves are not forms of heat but rather forms of energy that are manifested as heat through their interaction with materials. Microwaves initially excite the outer layers of molecules. The inner part of the material is warmed as heat travels from the outer layers inward. Most of the liquid is vaporized before leaving the material. It results in very rapid drying without the need to overheat. Consequences are energy saving by increasing the speed of drying and possible lower drying temperature. Even more, because energy is transferred through the material as electromagnetic waves and not as a heat flux, the rate of heating is not limited, and the uniformity of heat distribution is considerably improved.

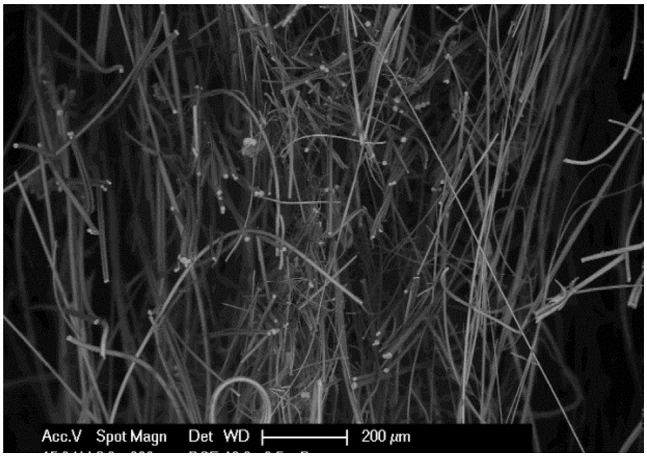

Fig. 1. Glass fiber mat.

2. Materials and methods

2.1. Preparation of composite aerogels

The studied blankets are made by Enersens Company in the framework of the HOMESKIN European project [19]. Initially, a fibrous network (PET or glass) is impregnated by pre-hydrolyzed TEOS [Tetraethoxysilane] precursors. A silica organogel is obtained by the in-situ gelation of the precursor, and the organogel is made hydrophobic by silylation with HMDSO [Hexamethyldisiloxane] in an acid medium. This last step prevents any modifications of the structure in case of capillary condensation, which would lead to the degradation of mechanical and thermal properties. The silylation reaction substitutes hydrolyzable groups such as silanol and ethoxy groups by non-hydrolyzable groups and permits the silica gel to reopen its pore during the last stage of drying due to the repulsion of the graft groups and elasticity of the solid network. Finally, the composite obtained containing the fibrous network and the hydrophobic silica gel is dried by fixed frequency electromagnetic wave. Fibers take the form of an un-woven mat with fiber diameter around 10 μm and a length of few millimeters. The fraction volume fiber is in the range 1%–5% in order to limit the increase of the solid thermal conductivity by the fibers themselves and to strengthen the aerogel. SEM images of PET and glass fiber mat are shown in Figs. 1 and 2. The improvement of the silica gel by the addition of fibers as well as the hydrophobic agent allows getting a dry material, with good mechanical and thermal properties without using super-critical drying. The manufacturing process and the resulting aerogel blankets are shown in Fig. 3. The blankets dimensions are 300 mm × 400 mm and the thickness depends mostly on the fibers mat thickness (15 mm for the PET mat and 30 mm for the glass mat). Comparing to pure silica aerogel, the obtained blankets are more opaque and more flexible but they release a lot of dust.

2.2. Characterization

2.2.1. Textural characterization

Scanning electron microscopy (SEM) provided information on the microstructure of aerogels. All the images were taken in Mines ParisTech with a SEM Philips XL30 for small magnifications (fibers