

Multiple assembly strategies for silica aerogel-fiber combinations – A review



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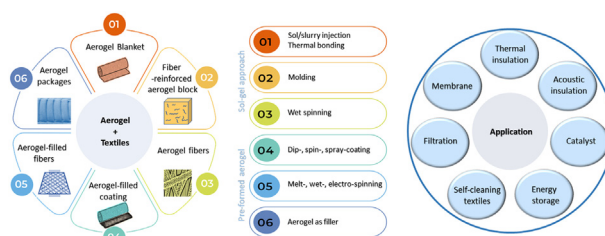
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HIGHLIGHTS

- Comprehensive review of all approaches used to assemble aerogel-fiber materials.
- “In situ sol-gel” and “via pre-formed aerogel” approaches are described and evaluated.
- Enhanced mechanical/physical properties are obtained while keeping low thermal conductivity.
- An overview of the market and future research trends are identified.

GRAPHICAL ABSTRACT



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ABSTRACT

Silica aerogels have a unique structure that makes them promising materials for variable applications. However, they are brittle due to weak inter-particle necks, and also expensive. Combining aerogel with fibers can not only enhance the mechanical/insulation properties, but also reduce dust release, and ease practical application. The majority of review articles in this field have been on the aerogel/textile systems' application or on textile impregnation in silica sol utilizing the sol-gel technique, with a few papers also addressing the use of aerogel as filler. This review for the first time highlights all strategies to assemble silica aerogel with textile materials. For sol-gel approaches, the fibers can be impregnated in a silica precursor sol to form the aerogel in situ between the fibers, but the sol itself can also be spun into aerogel fibers. Other strategies employ pre-formed silica aerogel, mixed in polymer or solvent matrices/slurries, to form aerogel injected blankets, aerogel-filled material coated fibers, and aerogel-filled composite fibers. Aerogel particles-filled textile packages have also been proposed. The emerging activities on simulations of aerogel-fiber combinations are reviewed. The advantages/disadvantages of various approaches are evaluated, and the current market situation and an outlook for the future of the field are summarized.

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1. Introduction

1.1. Silica aerogels

Aerogels are low density, predominantly mesoporous solids with exceptional properties, including low densities, high specific surface areas, low dielectric constants, and ultralow thermal conductivities [1,2]. The first aerogels were produced in the 1930 s by Kistler [3–5] and he already described a variety of materials chemistries, including silica, alumina, cellulose, and egg albumin aerogels. Since then, the range of material compositions from which aerogels can be produced has been extended to include, among others, metal oxides such as silica and alumina aerogels, metal aerogel, carbon allotropes such as graphene or carbon nanotube aerogels, synthetic polymers including resorcinol–formaldehyde, polyurethane and polyimide aerogels, biopolymers such as cellulose, chitosan, and protein aerogels, and their composites and hybrids. Particularly during the last decade, there has been a veritable explosion in the number of scientific papers and patents that describe new aerogel materials, production processes, and applications, including thermal insulation [6,7], drug delivery [8,9], environmental remediation [10], catalysis [11] and acoustics [12].

Despite the growing importance of the aerogel field, or perhaps exactly because of it, the definition of what is an “aerogel” is controversial. Older definitions were often based on the drying technique employed during production: *aerogel* for supercritical drying, *cryogel* for freeze-drying, and *xerogel* for evaporative drying, but more recent aerogel definitions are typically based on the material properties and a high fraction of mesoporosity in particular [13]. Finally, the broadest possible definition of aerogel is that of any material derived from a gel by replacing the pore fluid with air, without any limitations in terms of pore size or other properties. This wider definition includes predominantly macroporous materials that do not display the mesoporosity, high surface area or ultra-low thermal conductivity typically associated with aerogels, e.g. freeze-dried cellulose foams. In this review, we limit the term aerogel to predominantly mesoporous materials.

Among all possible aerogel chemistries, and despite the exciting developments on other materials, silica aerogels remain the only materials that are available in industrial quantities with stable supply chains. Silica aerogels are produced through a sol–gel process and many variations have been proposed to increase the resource- and cost-effectiveness of the production [14]. Nevertheless, most processes follow the same basic steps. A silica sol, i.e. a dispersion of silica nanoparticles derived from waterglass or silicon alkoxides, is destabilized to create a continuous, porous, solid network, i.e. a gel. The gelation trigger almost invariably is the addition of acid or base to reduce the charge stabilization of the nanoparticles [1]. After gelation, the gel can be aged to increase the mechanical stability by reinforcing the inter-particle necks through silica dissolution precipitation reactions [15]. In most production protocols, the silica aerogels are hydrophobized with hydrophobization agents, e.g. hexamethyldisilazane or methyltrimethoxysilane, either by including them directly into the sol or by a separate surface modification step after gelation and aging [16]. Finally, the silica gels are dried by supercritical drying (SCD), from supercritical alcohol at a high temperature [3,17] or more commonly from supercritical CO₂ at more moderate temperatures [18]. Since a few decades, also evaporative drying at ambient pressure (APD) can produce silica aerogels with properties that are virtually indistinguishable from their SCD counterparts [19,20]. Both SCD and APD are applied at an industrial scale: SCD requires more complex equipment but has a wider tolerance on gel properties (mechanical strength and degree of hydrophobiza-

tion), whereas APD is a simpler and arguably more cost-effective process, but with a more narrow window in terms of silica concentration, gel mechanics and hydrophobization [21]. Fig. 1 and Table 1. show a schematic design of an aerogel synthesis and characterization, and properties of common hydrophobic silica aerogels [22], respectively.

The industrial success of silica aerogels is driven almost exclusively by thermal insulation applications. Silica aerogel can have a thermal conductivity as low as 0.012 W/(m.K), as the high porosity and tortuosity of the particle network limits solid heat conduction, and because the small pore sizes, below the mean free path length of gas molecules, reduce gas phase heat conduction due to the Knudsen effect. This ultra-low thermal conductivity, half of that of standing air and conventional insulation materials, has enabled the emergence of a rapidly growing market of a few hundred million dollars. The total thermal conductivity has a strong correlation with the density of materials, as shown in Fig. 2. For conventional insulators, radiative contributions are important [23,24] and in the case of very large pore sizes, also air convection. With increasing density, radiative heat conduction decreases and solid thermal conduction increases. As a result of these competing effects, the thermal conductivity displays a U-shaped dependence on density. For aerogel materials, the aforementioned effects also take place, but there is an additional, strong decrease in the gas phase conduction because the pore sizes within the aerogel are smaller than the mean free path of air, which decreases the collision frequency of air molecules so that the thermal transfer of gas is decreased [25]. This shifts the minimum in the total thermal conductivity to higher densities and (much) lower conductivities.

1.2. Textile materials

Fibers and textiles are present in nearly every part of our daily life. Based on exports, the textile industry is the world's fourth-largest industry sector showing up in clothing, flooring, filtering, automotive technical textiles, composite reinforcement, medical textiles, and other home or technical applications. The main advantage of textiles is their broad distribution of properties that can be adjusted to the technical requirements of a certain application, including mechanical properties such as elongation, strength and modulus, variable porosity, flexibility, and high surface to mass ratio. Importantly, these properties can be adjusted by whether tailoring the fibers' structure itself or optimizing the textile fabrics' structure and properties [28,29].

Fibers are materials with a high aspect ratio (length to diameter ratio) which are divided into natural or man-made (regenerated or synthetic), or organic and inorganic categories based on their sources [30]. Organic or polymeric fibers e.g. cellulose, flax, wool (with natural bases), polyester (PET), polyamide (PA), polyaramid, polyacrylonitrile (PAN), polypropylene (PP), carbon, etc. (with synthetic bases) are more applicable in clothing and high-performance textile products, while inorganic fibers e.g. metallic, ceramic, glass, basalt, mullite, sepiolite, xonotlite, and boehmite fibers (with natural bases) are more suitable for technical applications [30,31]. Natural fibers have their own inherent properties (diameter, length, and so on) while man-made fibers can be manipulated during production by process adaptation. Melt-spinning (Fig. 3A) is the most conventional method for fiber production from thermoplastic polymer bases (and molten inorganic materials), in which an extruder-assisted molten polymer passes through spinneret nozzles with a uniform flow rate adjusted by gear pump speed. Melt-spun fibers can be stretched within drawing godets and collected in a filament yarn form in a winder or cut into staple fibers. Also, thermoset polymers can be dissolved in a solvent and shaped in a fiber form passing through spinning nozzles in solution spin-

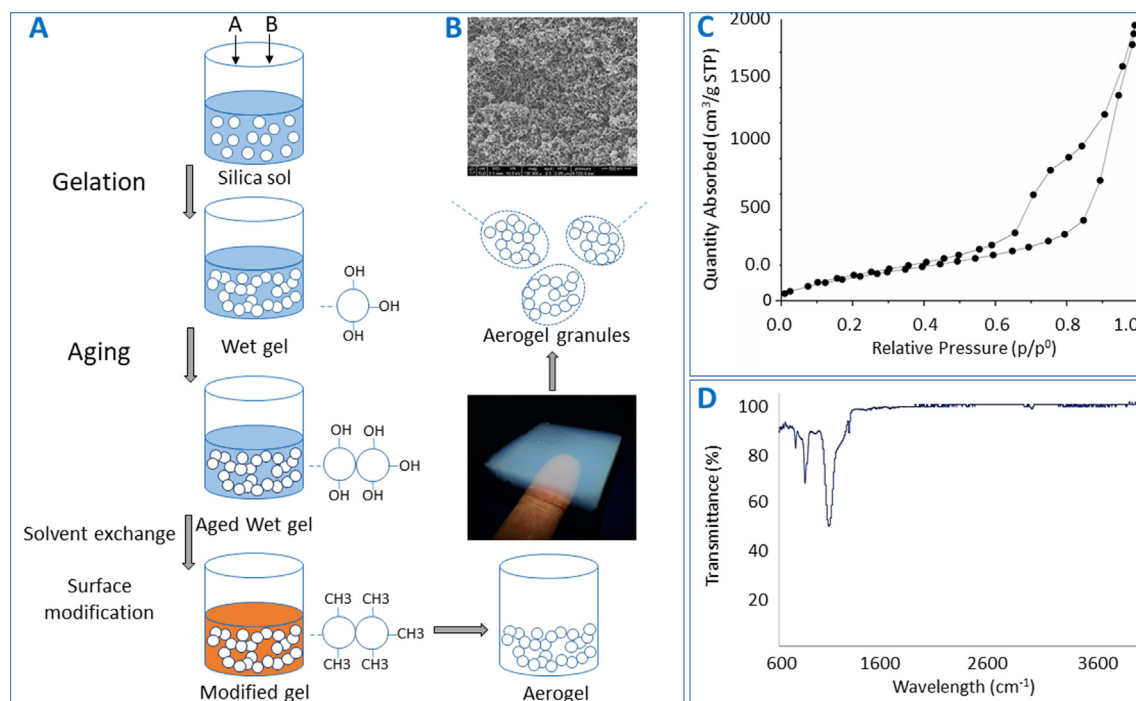


Fig. 1. A) Schematic illustration of sol-gel process, an aerogel B) SEM micrograph [26], C) BET, and D) FTIR graph. Revised/reproduced with permission from Ref [26].

Table 1

Hydrophobic silica aerogel properties Revised/reproduced with permission from Ref [22].

Composition [wt%]	
- SiO ₂	80–88
- Carbon	10–15
- Hydrogen	2–5
Surface chemistry [molecules/nm ²]	
- Trimethylsilyl groups	2–4
- Silanol/(m)ethoxy groups	0.5–2
Structural/physical properties	
Surface area [m ² /g]	500–1000
Primary particle size [nm]	3–5
Secondary particle size [nm]	20–50
Average pore size [nm]	5–60
Density [g/cm ³]	0.08–0.14
Water contact angle [°]	130–160
Sound speed [m/s]	40–1000
Refractive index	1.002–1.050
Thermal conductivity [mW/(m.K)]	12–18

ning (wet-spinning or dry-spinning). The difference is in the desolvation method in which a non-solvent coagulation bath is used in wet-spinning method (Fig. 3B). Fibers can be produced in different numbers (based on the spinning nozzles) with various linear densities. Microfibers (with a linear density of below 1 denier: g mass per 9000 m) provide higher specific surface area and more flexibility. Nanofibers (diameter < 100 nm) are produced through electrospinning by applying a high voltage for accelerated stretching of the spun dissolved polymer fiber through the formed Taylor cone in the spinning needle tip (Fig. 3C). Electrospun fibers from various material bases e.g. cellulose, polyvinylidene difluoride (PVDF), PAN, or even carbon (CNF) provide a high surface to mass ratio which is useful for different purposes [32].

Fibers can be processed into textile products e.g. yarns, woven or knitted fabrics, nonwovens either multiaxial or braided structures. For instance, staple fibers are converted into spun yarns through ring-spinning or the open-end technique. Meanwhile, filament or spun yarns can make a 2D fabric form via processing through weaving (Fig. 3D) or knitting (Fig. 3E). In a weaving pro-

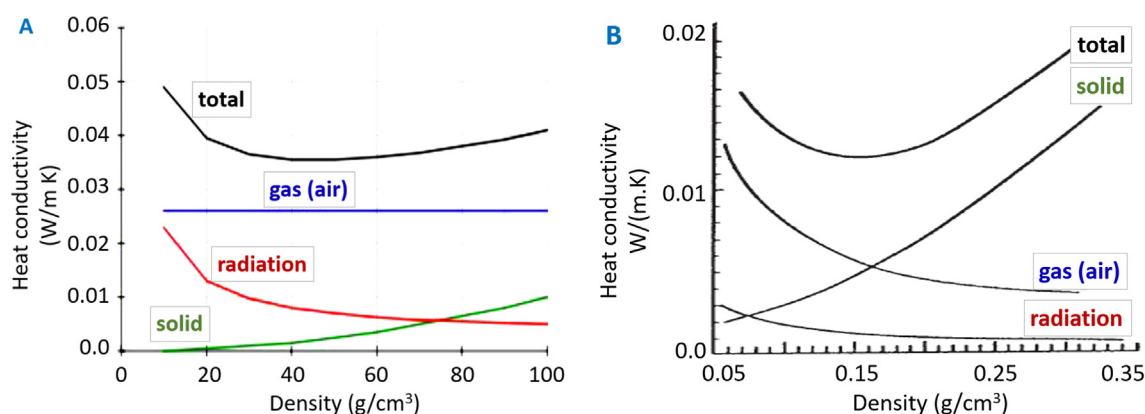


Fig. 2. A) Thermal conductivity of conventional insulation [23], and B) aerogel materials [27]. Revised/reproduced with permission from Ref [23,27].

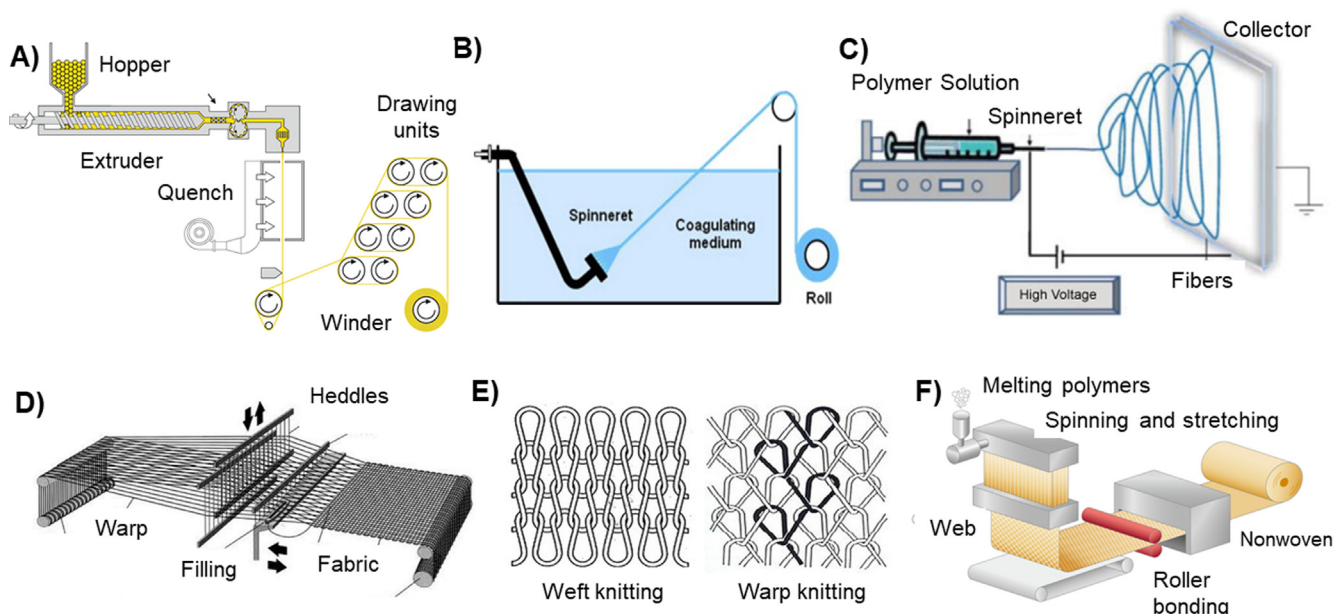


Fig. 3. The schematic view of fiber/textile material processes: A) Melt-spinning [35], B) Solution spinning (wet-spinning) [36], C) Electrospinning [32], D) Weaving [37], E) Knitting [38], F) Nonwoven (spunbond) [33]. Revised/reproduced with permission [32,33,35–38].

cess, weft or filling yarns pass through open sheds, formed from warp yarns, interlacing in a pattern e.g. plain, satin, twill, etc. while in the knitting process yarns are interloped in a weft-knitting (circular knitting) or warp-knitting (flat knitting) machine. More details can be found elsewhere [29,33]. Also, one of the most used fiber-based textile fabrics is nonwovens. There are several definitions to the term “non-woven” e.g. a three-dimensional web-like structure, where the fibers are randomized or orientated in a certain direction (as in wool felt, known as the oldest nonwoven material). In the case of randomly oriented fibers, nonwovens feature isotropic properties. Due to the lower processing costs (skipping the yarn spinning step), and faster production speed, nonwoven products are cheaper than knitted and woven fabrics. In addition to the felting, there are some other methods to form nonwoven structures e.g. thermobonding (thermal heating to bond the randomized fibers), spunbonding (hot rollers for bonding the fibers), needle punching (entangling the fiber webs by using punching needles), meltblowing (stretching and entangling the spun fibers using hot air), spunlacing (using water jets for entangling the fibers). Schematic view of sponbonding process is shown in Fig. 3F for instance. Some methods are based on entangling which can be done on both thermoplastic and thermoset fibers, while some others are heating-based and only suitable for thermoplastic fibers [34]. The bulk density of nonwovens is ranged below 100 g/m^3 (cleaning napkins) or above 1000 g/m^3 (flooring felts) suited for various applications. Nonwoven structures have different performances (fineness, mass per surface, etc.), making them suitable for various applications (filter media, flooring, masks, clothing, etc.).

1.3. Silica aerogel-textile materials

On its own, silica aerogel is very brittle [39], and real-world applications almost invariably rely on fiber reinforcement, impregnation into fiber mats, or the incorporation of silica aerogel granules in a mechanically more robust matrix. Here, the most popular aerogel composite products are fiber-reinforced silica aerogel blankets which first made an impact in industrial and oil-and-gas pipeline insulation and second in building insulation

[40] although silica aerogel renders [41], or translucent elements for daylighting applications [42] have also been introduced. The cost aspect is particularly important for both pipeline and building applications, but even at the current price-point, space-savings through the use of superinsulation can offset the comparatively high material and production cost [43]. Very recently, silica aerogel-filled fiber mats have found widespread use as high-safety separators in lithium-ion battery packs [44]. Combining textiles with aerogels therefore prompted researchers to conduct more research in this area; however, no comprehensive review has been carried out to evaluate all possible strategies to merge textiles and aerogels. Review studies in this area have mainly focused on fiber or textile layer impregnation in silica-based sol using the sol gel approach [45,46], e.g. silica aerogel embedded fibers [45] or silica aerogel-textiles via aggregation of inorganic nanomaterials and polymer nanofibers [46]. The application of aerogel/textile systems [28,46–50] also has been the aim of other review researches, e.g. aerogel/textile applications [47], aerogel/textile materials in high-performance textiles [48], for protection against heat [49,50], or for acoustic applications [12], and recently emerging applications of aerogel/textiles [28]. In contrast, the aim of this paper is a complete overview on all possible production methods for combining aerogel with textile materials, whether through sol-gel synthesis, embedding of pre-formed aerogel on fiber surfaces or incorporating into polymeric matrices, as shown in Fig. 4, along with their advantages and disadvantages, applications and properties, their market readiness and future prospects.

2. Fiber-silica aerogel blankets/composites: Sol-based approach

Silica aerogel blankets are the most popular aerogel-based industrial product with key manufacturers all around the world e.g. Aspen Aerogels Inc., Nano High-tech Co., Ltd., Alison Hi-Tech Co., Ltd, Enersens, Shenzhen Aerogel Technology Co., Ltd, Xiamen Nameite. Textiles, especially nonwovens, can be impregnated with a silica aerogel sol, to produce fiber-reinforced silica aerogel blankets (Fig. 4A). Alternatively, the embedding of loose fibers in silica aerogels is an effective way to improve their mechanical properties

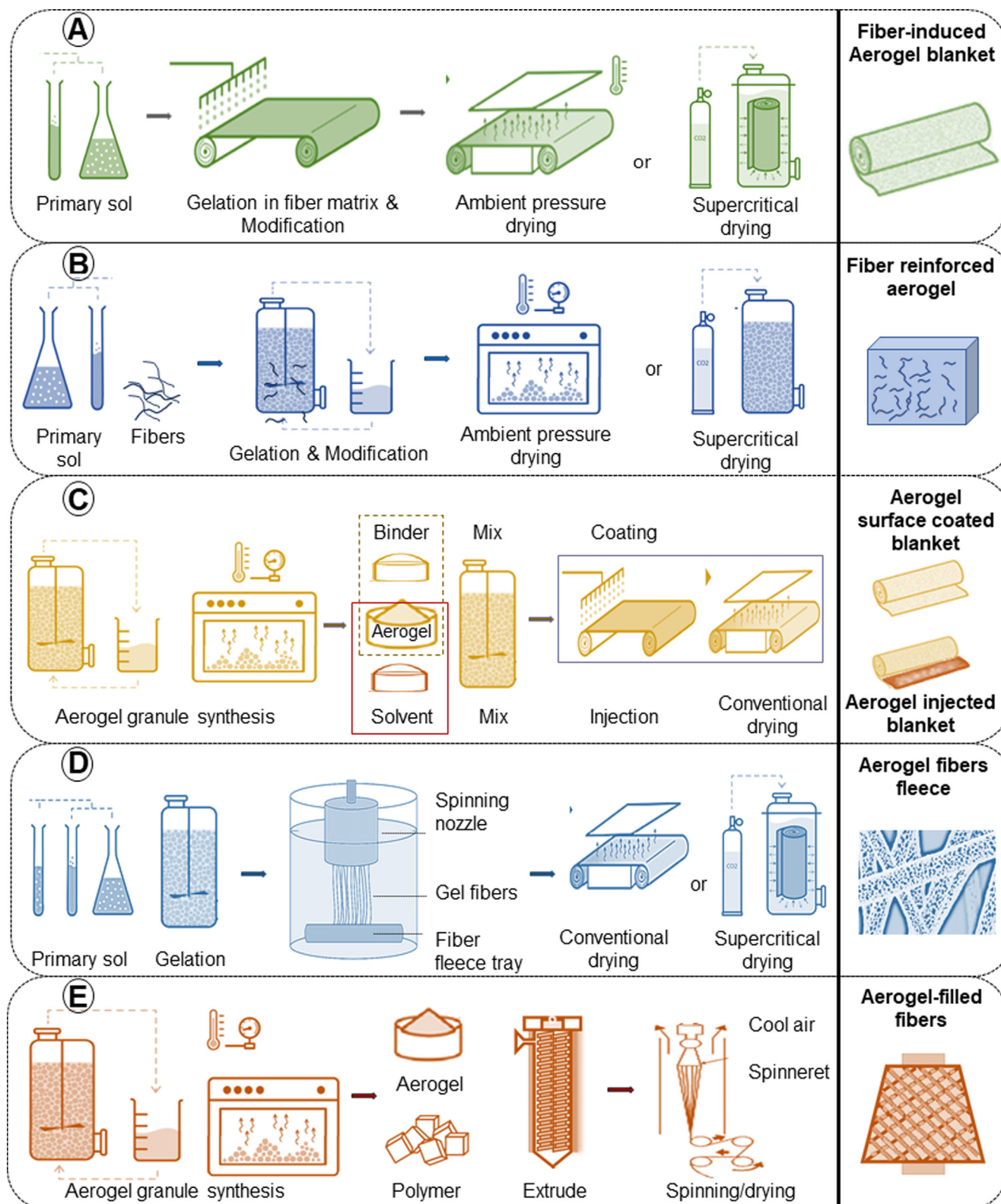


Fig. 4. Main routes to make aerogel-textiles combination: A) Aerogel blankets, B) Fiber-reinforced aerogels, C) Aerogel-filled material coated textiles, D) Aerogel fibers, and E) aerogel-filled fibers.

and provide reinforcement (Fig. 4B). The challenge is to preserve the aerogel's inherent properties e.g., insulation properties, high specific surface area, and low density during the sol-gel processing steps in the presence of the fibers or fiber mats [51]. In general, the

properties of the silica aerogel phase, e.g. porosity, surface area, pore size distribution, and thermal conductivity, and thus also the performance of the composites, are determined primarily by the selection of the precursors and the conditions of the sol-gel

and drying processes, rather than by the type and content of fibers [52]. However, the presence of a fiber matrix and the target to produce cohesive composites can affect the sol-gel processing conditions and production efficiency: for example, the diffusion of reagents during hydrophobization or the evacuation of the solvents during drying are slower for a 20 mm thick fiber reinforced silica aerogel composite compared to a silica aerogel powder and as a result, commercial silica aerogel impregnated fiber blankets are usually limited to 10 mm in thickness.

Fiber immersed silica aerogel composites can be accomplished by fiber impregnation using an aerogel primary sol using a variety of strategies [45]:

- by dispersing the fibers in the sol before the gelation and adding them into a mould [53–55], which request a homogenous structure of the composite before gelation and no sedimentation of the fibers [56],
- by pouring the sol over a fiber mat that was previously placed in a container [57],
- by immersing the fiber layers to the as-prepared silica sol in the mould [58], or by immersing the fibers into the water or solvent in the mould and adding a precursor/other additives whilst stirring [59,60],
- by dipping of fiber layer in the dispersion of silica hydrogel [61], method 2 and 3, but by immersing more than one fiber layer from one or two different types [62],
- method 5, but by immersing the textile layer made of parallel oriented fibers, layer by layer, in the same or the perpendicular direction as compared to the fiber directions in the first layer [63,64].

Because all of these processes deal with the conversion of the sol into a gel and aerogel in situ, between the fibers, the same process of solvent exchange, surface modification, and ambient pressure[52]/supercritical [65]/freeze [66] drying like those applicable for pure silica aerogels have to be applied here as well [52,65,66]. The entire procedure is briefly sketched out in Fig. 4A and B.

2.1. Loading inorganic fibers

Both inorganic or organic fibers can be used to form fiber-reinforced silica aerogel composites mostly to improve mechanical properties [54,65,67–69] while keeping the thermal conductivity as low as that of pure silica aerogel materials [70]. Other applications like sound absorption [61,71,72] or water-oil separation may be used as the second step [60]. In the reported literature, most of the fibers selected to reinforce aerogels are inorganic fibers, including ceramic fibers [65,67,68], glass fibers [64,73], mullite fiber [25], xonotlite [55], sepiolite fibers [53], and boehmite [74], which mainly improve the compressive strength and are applied at high temperatures [67,73,75,76].

- $\text{SiO}_2\text{-Al}_2\text{O}_3$ ceramic fibers

The pioneering investigation to reinforce silica aerogels with fibers in 1995 by Wang et al. [75] consisted of the incorporation of $\text{SiO}_2\text{-Al}_2\text{O}_3$ ceramic fibers to silica aerogels to increase the thermal and mechanical stability. TiO_2 powder was also chosen as an opacifier to reduce the radiative heat transport, which led to improved thermal insulation for high-temperature applications [75]. Later research studies on the $\text{SiO}_2\text{-Al}_2\text{O}_3$ fiber-reinforced silica aerogels were mostly done via immersion of a layer of fibers [65,67] or dispersion of fibers [54] in the TEOS sol [65,67,68,75] or Al-silica sol [54] and then, supercritical drying

[54,65,67,68,75] to achieve high performance in compression test [65], and/or short and long-term creep behavior [67], even at high temperatures up to 900 °C [65,67,68] although the thermal conductivity of $\sim 0.04\text{--}0.05$ W/(m.K) was substantially higher compared to pure silica aerogel.

Based on the slope, the compression curve of the fiber-reinforced aerogel material can be divided into three different steps: (1) The linear step with the constant slope, (2) the yielding step at a fixed rate lower than the first step, and (3) the densification step with an increasing slope, Fig. 5 [65,67]. The first compression step in the fiber-reinforced and pure aerogel materials can be attributed to the open pores of aerogels which performs as the main load-bearing part. The pores' collapse and fibers' failure are probably possible reasons for the stress relaxation and the small inelastic strain of the specimen at room temperature. As the temperature increases to 800 °C, the stress relaxations increase due to the integration of aerogel nanoparticles to form nanoparticle clusters as well as fiber stress relaxation [65]. This fiber-reinforced aerogel has high compression strength, but larger deformability than the pure silica aerogel and is suitable as a load-bearing insulation [67].

Creep behaviors of fiber-reinforced silica aerogels at both low and high pressures, 0.05 and 0.10 MPa, could be also divided into three different steps: (1) First step ($\sim 0\text{--}0.5$ h) with relatively high creep rates, (2) second step ($\sim 0.5\text{--}168$ h) with decreasing creep rates with time and (3) the third step (\sim after 168 h) with constant values of creep strains (Fig. 6). Microstructure modification and thermal softening of silica aerogel nanoparticles with increasing temperature were the key factors to change the mechanical properties such as the compression modulus and composite creep response at low operating stress [67].

$\text{Al}_2\text{O}_3\text{-SiO}_2$ fiber reinforced silica aerogels by Shi et al. in 2013 [68] showed 10 times higher Young's modulus in-plane than that of out-of-plane direction [68]. From compatibility and thermal stability points of view, when working on the $\text{Al}_2\text{O}_3\text{-SiO}_2$ aerogels, Yu, et al. in 2018 [54], reported the use of $\text{Al}_2\text{O}_3\text{-SiO}_2$ fibers to form $\text{Al}_2\text{O}_3\text{-SiO}_2$ fiber-reinforced $\text{Al}_2\text{O}_3\text{-SiO}_2$ aerogels with a high content of fibers at 40–55 wt% and high resistance to temperatures of 1200 °C [54]. In another work a year later, the researchers [77] prepared a quartz fiber-reinforced $\text{Al}_2\text{O}_3\text{-SiO}_2$ aerogel composite with low density (0.36 g/cm³), high specific surface area (580.5 m²/g), and thermal conductivity of 0.049 W/(m.K) at room temperature [77]. To address the inherently weak interfacial bond between fibers and aerogel, mullite whiskers were synthesized in-situ on the surface of the alumina silicate fibers to strengthen the interfacial bond and thereby improve composite's mechanical properties [78].

- Glass fibers

Glass fibers are amongst the most studied inorganic fibers used for the reinforcement of silica-based aerogels. Most of the papers reported the use of TEOS as a precursor, but also water glass or MTMS, and supercritical drying method, but also ambient pressure drying. Furthermore, most of the investigations consider the mechanical and thermal properties as well as improving structural properties. While a low density of ~ 0.012 g/cm³ [57,70] is possible for glass fiber-reinforced silica aerogels including aerogel blankets, they usually benefit from density of $\sim 0.15\text{--}0.2$ g/cm³ [64,66,70,79,80] and thermal conductivity of $\sim 0.013\text{--}0.03$ W/(m.K) [57,58,64,66,70,79–87], but higher mechanical properties in terms of integrity, compared to pure silica aerogels, with a wide range in compressive Young's modulus between 0.1 and 3 MPa [58,64,79,80,86], but mostly between 1 and 2 MPa [85].

In some reports, improvement of hydrophobic properties was also taken into account [66]: the glass fiber-silica aerogel composites benefit from the coverage of the glass fibers with highly

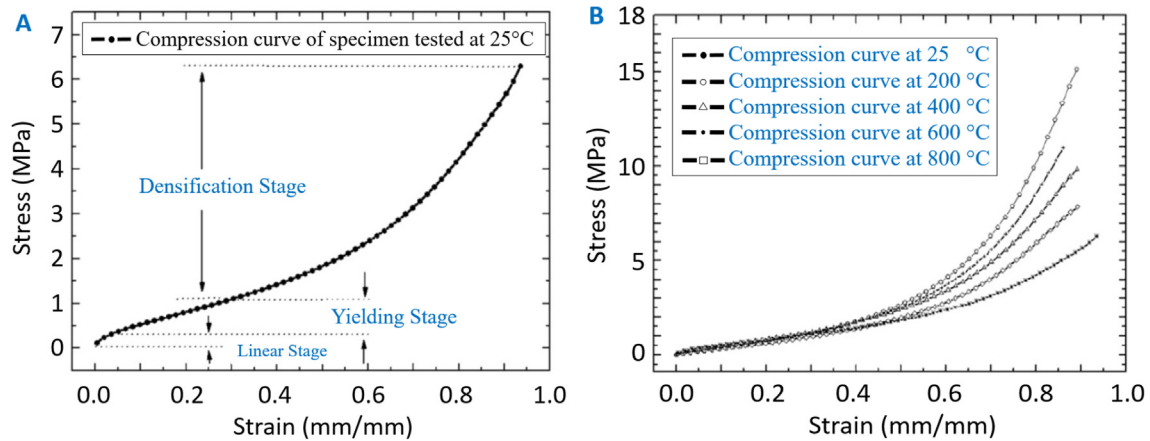


Fig. 5. Example of a compression curve at A) 25 °C, and B) high temperature. Revised/. reproduced with permission from [65]

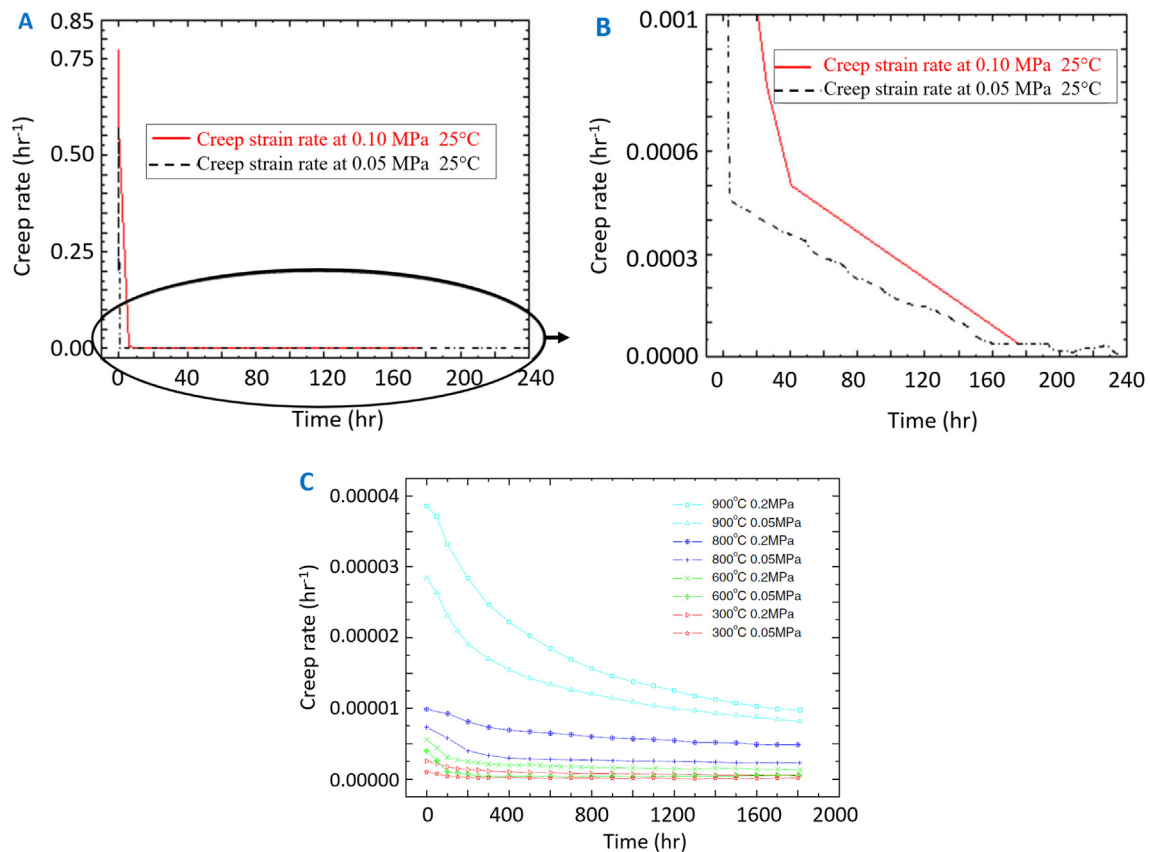


Fig. 6. Example of a creep strain rate history of the fiber-reinforced aerogels. Revised/reproduced with permission from Ref [67].

hydrophobic aerogels and will show good hydrophobic properties in view of their application for construction materials and longtime durability. Including MTMS in the primary sol can lead to a high water contact angle from ~ 130 – 150° , and also remarkable flexibility for the composites [79]. Recently Begum and Horoshenkov in 2021 [88] researched the understanding of the sound absorption characteristics of glass fiber reinforced aerogel composites, but yet more research is required to understand the advancement of their use in acoustic applications [88].

Many parameters are varied to investigate the resulted composites from different aspects, e.g. different types of glass fibers [89,90], the addition of multi-layer glass fibers [63,64], mix of glass

and other fibers [57], different types of primary silica sols including TEOS [58,89], water glass [80,83], MTMS [66], water glass/MTMS [79], TEOS/MTMS [86], added polyimide [82], added fumed silica [84], added silica gel [85,86], various silica content in the sol [66,91], and its ratio to gel modifying agent [81], to water [58,80], and to alcohol [83], adding TiO₂ as an opacifier in the sol [70], and freeze-drying of the composites instead of supercritical or ambient pressure drying methods [79].

A brief abstract of changing parameters and their effect on the properties is reported in the following. Silica precursor type in the sol shows a strong effect on the physical properties of fiber-reinforced silica aerogels, e.g. mixed water glass- and TEOS-based

silica sols showed lower density, higher porosity, and surface area for the final composites in comparison with the pure sols [52]. The same results were achieved by Chandradass et al. in 2008 [91], using two precursors of TEOS and water glass, but with unmodified and modified glass fibers in an alumina sol [91]. Not only the silica sol type but also the silica amount in the sol, meaning the ratio of silica precursor to alcohol or water, has a strong effect on the final properties of composites. For example, thermal conductivity as low as 0.026 W/(m.K) with a flexural elastic modulus of ~ 12 MPa were achieved for glass fiber-reinforced silica aerogel in a 1-day short drying time since both high and low sol's alcohol content leads to high capillary pressure during drying [83] and even lower thermal conductivity of 0.0232 W/(m.K) can be achieved by optimizing the water to silica precursor content in the sol [58]. On the other hand, the fire hazard of the composites can be decreased with an increased ratio of alcohol or water in the primary sol [58,80]. Owing to higher mechanical strength, the glass fiber-reinforced silica aerogel composites can be used in construction industries efficiently [66]. However, the lower silica to glass fiber composition exhibit better insulating properties, but probably lower strength and integrity of the samples. Zhao et al. in 2012 [89,90] reported that the effective thermal conductivity of the fiber-loaded aerogel could be reduced by reducing the fiber length-to-diameter ratio (fiber diameter of 4–8 μm) and the inclination angle and by moderately increasing the fiber volume fraction [90]. Thus, very low thermal conductivity of 0.022 W/(m.K) and high mechanical performance is achievable with including $\sim 9\%$ smaller diameter micro glass fiber mats into the silica sol [81].

To further improve the glass fiber reinforced silica aerogels' mechanical strength Fei et al. in 2018 [82] investigated the effect of incorporating a polyimide (PI) part by dispersing glass fibers in a PI/SiO₂ hybrid sol. The SiO₂ primary particles were combined with PI chains to increase the strength of the composite aerogel, while the glass fibers prevented shrinkage deformation during the drying process. PI-SiO₂ hybrid aerogels normally showed much higher mechanical properties in comparison with the SiO₂ aerogels, but they suffer from high density and then, high thermal conductivity due to higher shrinkage in the drying stage which could be to some extent solved by the incorporation of the glass fibers in the sol [82].

Addition of fumed silica [84] and silica gel [85] to the silica precursor sol can play an important role in structural properties of the final aerogel material. Cluster particles of silica aerogel containing micropores were fused to the fumed silica aggregate particles with macropores, reducing the macropore of the fumed silica into the mesopores and its ability to accumulate dust when bent [84]. Also, the addition of silica gel [85] demonstrated that the impregnated silica gel filled in the gap between the glass fibers and covered the surface of the fibers completely (above 5 %), which not only prevented the heat transfer but also trapped and protected the silica aerogel from collapsing [85]. Inspired by these results, in 2020 [86] a strategy was proposed that the superhydrophobic silica gel-glass fiber composite can be prepared by applying MTMS and silica gel simultaneously to a TEOS precursor, compressed to $\sim 60\%$ of its original length without damaging the structure [86]. As for comparison between glass fibers and carbon nanotube (CNT) reinforced silica aerogel, although CNT showed significantly higher results for mechanical reinforcement, the tensile strength and elastic modulus of glass fiber reinforced aerogels was ~ 3 times higher than the native-SiO₂ in its axial direction [69]. This led to further go into in-depth analysis of glass fiber-reinforced silica aerogels with the non-equilibrium method of Müller-Plathe reverse perturbation [92].

To open a controllable way to improve and engineer the mechanical properties of the aerogel composites with low thermal conductivity, the application of a multi-layer of ordered fibers into

the aerogels was also investigated [63,64]. Applying different layers can either be parallel or perpendicular to the order of fibers in the primary fiber layer, highly affecting the final performance [63]. With the impregnation of the reinforced fibers, the thermostability of the aerogels improves significantly, retarding the severe weight loss of the fiber-reinforced aerogel composites to 370 °C, compared to 300 °C for unreinforced aerogels. The compressive strength of the aerogel composites was increased 1.5–6 times by impregnation with 3 wt% fibers as compared to neat aerogels, while their thermal conductivity was kept low at 0.024–0.027 W/(m.K) [63].

- Other inorganic fibers

Glass alone or in the mix with other fibers, e.g. polyester [57] or carbon [62], is the most fiber studied for reinforcement of the silica aerogels, followed by SiO₂-Al₂O₃, and then other inorganic fibers including mullite [25,76], xonotlite [55], boehmite [74], and sepiolite fibers [53]. Thermal conductivity of these composites is in the range of 0.02–0.05 W/(m.K) at ambient temperature, which is high in comparison with the glass fiber silica aerogel composites, and in some cases close to conventional thermal insulations, e.g. rock wool with thermal conductivity of ~ 0.04 W/(m.K). Therefore, they are usually studied for high-temperature mechanical and thermal properties. For example, the thermal conductivity of mullite reinforced silica aerogels was 0.082–0.182 W/(m.K) between 500 and 1200 °C and even lower, as of ~ 0.05 W/(m.K) at 1000 °C when the fibers were treated with SiC and after pyrolysis process [25,76].

Regarding high temperature application, not only fibers but also the reinforcement of silica base itself, e.g. ZrO₂-SiO₂ aerogels [25] was considered. The incorporation of fibers capable of chemically reacting/interacting with silica sol (silanol groups) during the gelation and aging steps can prevent the aerogel structure from disintegrating throughout drying [74]. As an example, surface pre-coating of the mullite fibers with the polycarbosilane (PCS) [76] or covering of boehmite fibers with polymethylsilsesquioxane (PMSQ) in the sol [74] can be named. Further thermal treatment of xonotlite fiber-silica aerogel composites upon calcination at 800 °C avoided full densification of the composites due to the interaction between the fibers and aerogel [55], leading to better keeping of the porosity.

2.2. loading organic fibers

The majority of the fibers chosen to reinforce aerogels in the reported literature are inorganic fibers [93], which can improve compressive strength but suffer from low flexibility due to their inherent brittleness. This reduced flexibility may make difficulty in meeting the specific requirements of some industrial applications, such as pipe insulation. Since inorganic fibers have a higher density, composites reinforced with inorganic fibers can also have a higher density and a higher thermal conductivity. Therefore, flexible reinforcing fibers with low density and high mechanical strength are required [93]. Studying organic fibers, mostly including polyester, polyolefin, aramid and cellulosic fibers, to reinforce silica aerogels started later than inorganic fibers and in general, can go back to 10 years ago.

- Polyester and polyolefin fibers

It seems that polyester (PET) fibers are very general to be composed with silica aerogel materials. PET as a nonwoven [61,71,94–96] is normally applied in the TEOS-based sol and the synthesized gel on the fiber surfaces is dried under ambient pressure [61,71,94,95,97]. PET can also be mixed with PP fibers for making

aerogel blankets, which are commercially manufactured by Cabot Co. [98]. However, Hoque et al. 2021 [97] recently used PP alone to reinforce silica aerogels [97]. The main characterizations included thermal insulation with thermal conductivity of ~ 0.019 – 0.03 which is lower than that of neat PET nonwoven but slightly higher than silica aerogel [94,95,98], sound absorption as well as hydrophobicity [61,71]. The higher the aerogel content between the pores in the nonwoven, the lower the thermal conductivity or diffusivity and the higher the water contact angle, $\sim 10^\circ$ increase, was analytically and numerically reported by Talebi et al. in 2014–2015 [94,95]. An efficient ambient pressure drying process allowed the up-scaling manufacture of PET-aerogel boards for prototype building thermal insulation [96].

PET-aerogel blankets can also be considered as a good sound absorbing material [61,71,72], particularly in the frequency range of 100–6300 Hz which is normally used for acoustic building materials [99], but more experimental and modeling insights on pure silica aerogel and their composite forms with the textiles are needed to fully understand their sound absorption properties. As silica aerogels normally show a high absorption peak in a narrow range of frequency like sound absorbing resonators, the inclusion of fibers leads to a smoother graph in the whole range of frequency which is characteristic of foam or fibrous sound absorbing materials. Details data on acoustic properties of silica aerogels and their composite with the other materials, including silica aerogel-textile composites, is available in the review paper by Mazrouei Sebdani et al. 2020 [12].

Recent research by Hoque et al. in 2021 [97] looked at preparing spongy, highly porous, and hydrophobic silica aerogel-polypropylene (PP) nonwoven fabric composites with a water contact angle of 129° . TGA analysis found that there was an initial degradation at 445°C and residual ~ 23 wt% loss at 650°C , therefore showing excellent thermal stability for the silica aerogel-PP nonwoven fabric [97]. To further understand the benefits of using organic fibers, other research by Zang et al. [100] used polypropylene fibers that were 12 mm long to achieve higher mechanical resistance than pure silica aerogel and also a much higher absorption capacity when compared to the use of activated carbon fibers.

- Aramid fibers

Aramid fibers are known to possess lower density, lower thermal conductivity, and higher mechanical strength as compared to inorganic fibers. They are also known to have a decomposition temperature in the air of up to $\sim 450^\circ\text{C}$ which makes them highly desirable for use in high-temperature thermal insulation applications [93]. Aramid [93,101,102] and meta aramid [103] fiber reinforced silica aerogel composites (AF/aerogels) were successfully prepared in 2016 and then, Glycidylpropyltrimethoxysilane (GPTMS) grafted aramid fiber [104] as well as polytetrafluoroethylene coated aramid fiber aerogel composites in 2017 [105], mostly with TEOS sol and with ambient pressure drying method like before for PET fibers. Compared to inorganic fibers, the AF/aerogels possess lower density and lower thermal conductivity, similar to that of pure silica aerogels. However, inorganic fiber-reinforced silica aerogel composites have improved compressive and flexural strength higher than that of AF/aerogels. Therefore, AF/aerogels have great application prospects for thermal insulation applications [93,101,102], which was improved with increasing amount of TMCS in the aerogel synthesizing process from 2 to 10% [103]. The good thermal and mechanical properties of the aramid fibers led to further research on the ballistic properties, as fewer fabrics were perforated at a rate of 72 % in all ballistic test samples including aerogel monoliths in comparison to the neat aramid fabric tests [106].

Almeida et al. in 2021 [107] compared the reinforcement of silica aerogels with aramid fibers (AF), a felt (PAF), and Kevlar Pulp (KP). The bulk densities of the composites were lower when using elongated fibers (below 0.15 g/cm^3), compared to those with shorter fibers due to higher shrinkage and easily bending for the shorter fibers. Also among different elongated aramid fibers, the composites reinforced with *meta*-type led to better dispersion and more homogenous composites. However, *meta*-aramid fibers were the less thermally stable fiber, compared to the *para*-aramids. Mechanical tests indicated that composites reinforced with elongated fibers are more flexible when compared to those reinforced with fibers in the pulp form. Thus, these composites are more suitable for applications involving shape adaptation and vibration, e.g. space-related applications [107].

- Other organic fibers

Widely available and inexpensive cellulose fibers can provide strong reinforcement for silica aerogels with a low volume shrinkage and very low thermal conductivities for both supercritical as well as cost-effective and high pressures avoidable ambient pressure drying routes [108]. To compare the supercritical and ambient pressure drying methods of the cellulose fiber reinforced aerogels on their final properties, reinforcement with different sizes and concentrations of man-made cellulose TENCEL fibers [108,109] showed a low thermal conductivity between 0.014 and 0.018 W/(m.K) for both method and surprisingly strong improvement in the mechanical properties of composites, but lower for ambient pressure dried composites in comparison with their supercritical dried counterparts [108]. Sound absorption properties of the cotton nonwoven-aerogel blankets were also studied [110] which is completely reported and compared in another review paper [12].

Shi et al. in 2017 [60] reported new silica aerogel composites for oil adsorption from oil/water mixtures, with successful incorporation of polyacrylonitrile (PAN) fibers into the silica matrix. Hydrophobically modified aerogel with a water contact angle as high as 169° lead to an enhanced adsorption capacity and subsequently oil-water separation effects [60]. As for absorption field, porous materials are the most widely used due to their high efficiency, low cost, and wide adaptability. However, the major drawback of these materials is that water and oil cannot be separately absorbed [60,111]. Aerogels have outstanding access to the inner surface due to their open porous network, making them an appealing candidate for a variety of wastewater treatment applications [60,112]. As well to be used as good water-oil separators [112], for better mechanical properties, fiber-incorporated silica aerogels may also have a great potential for water treatment.

2.3. Loading carbon fibers

Wang et al. in 2018 [113] proposed a novel lightweight carbon fiber reinforced silica-phenolic resin (CF/Si/PR) aerogel nanocomposite with a finer microstructure, higher thermal stability, and better anti-oxidation resistance than PR aerogels. An example of such composites with density of 0.463 g/cm^3 showed a compressive strength of 2.44 MPa and thermal conductivity of 0.116 W/(m.K) . It is strongly obvious that the density and thermal conductivity of the carbon-reinforced silica aerogels are much higher than those of their counterparts with glass or PET or other fibers [57,58,64,66,70,83,87,94,95], but due to very high thermal stability and mechanical properties, carbon-reinforced aerogel composites presents numerous application potentials in the thermal protection and insulation field, particularly in the aerospace industry.

In research carried out by Slosarczyk in 2017 [114], she looked at the physicochemical characterization of carbon microfibers and

nanotubes embedded into silica aerogel nanocomposites. Three types of carbon materials were used: carbon microfibers, grown carbon nanofibers, and polyhedral carbon nanotubes. The results found that when longer fibers of length 700 μm were used, there was a reduction in cracking of the monolith structure of the aerogel at the micro- and nano- levels. This enabled the possibility of larger amounts of carbon fibers to be incorporated into the composite which could enhance the conductivity properties. In another work by Slosarczyk et al. in 2021 [115], the synthesis of carbon fiber reinforced silica aerogel from a much cheaper precursor of water glass than organosilicon compounds were discussed. Regarding the importance of fiber surface modification, before being added to the silica gel, the carbon fibers were surface treated to increase adhesion at the interfacial border. An additional effect of the presence of fibers above 10 % by volume, besides increased mechanical and structural parameters, and thermal conductivity coefficient $\sim 0.032 \text{ W/(m.K)}$, was the ability of the nanocomposite to conduct an electric current [115].

Since glass and carbon fibers are commonly used as reinforcements due to combination of high strength and relatively low density, Hung et al. recently in 2021 [62] attempted to fabricate reinforced aerogel composites with combined glass-carbon fibers in various shapes and functions while maintaining a low thermal conductivity. To achieve a balance between thermal insulation performance and strengthened structure, two layers of glass fibers and one layer of carbon fibers were used as structure reinforcing layers and a thermal insulation layer, respectively [62], which will be beneficial for future industries in producing versatile fiber-reinforced aerogels with great characteristics. Reinforcement of silica-based aerogels with hybrid carbon-quartz fabrics with compressive strength ranging from 5.96 to 7.51 MPa, the low thermal conductivity of 0.112 W/(m.K), and more importantly, good thermal ablative/insulative properties after being subjected to high temperature oxidizing oxy-acetylene flame was reported by Cheng et al. in 2021 [116].

2.4. Loading nanofibers

Another way in which fibers have been used in aerogel textiles is through the use of polymeric nanofibers in aerogels. Electrospinning involves the use of a strong electrostatic field connected to a metallic needle containing a reservoir of the polymer solution. A voltage surpasses a threshold value, and the polymer solution is ejected to form a stable thread of polymer fiber. The electrospun polymer nanofibers provide a reinforcing support network that offers both bendability and flexibility for fragile silica aerogels. Fibers with smaller diameters as well as homogenous diameter size distributions are better preferred to avoid cracks and weaker mechanical properties of the resulting fiber-filled aerogel composites. Therefore, the use of nanofibers can provide a higher interaction with aerogels in comparison to normal fibers but can be much more cost-intensive. Electrospinning as the conventional technique for nanofiber production has attracted researchers' attention due to its ability to improve the integration between the two phases (polymer and aerogel). Generally, nanofibers are dispersed in the silica sol and undergo an aging process to allow the silica particles to strongly adhere to the nanofibers [117]. Various nanofibers can be used to improve the robustness of the aerogel, from carbon [118] to other polymeric types [119].

Meador et al. [118] used a commercially available carbon nanofiber-Pyrograf[®]-III to improve the mechanical properties of silica aerogel composites. The carbon nanofiber had side walls composed of angled graphite sheets which provide a highly reactive edge site along the entire surface of the fiber. This ensures the fiber surface can be easily chemically modified to warrant maximum mechanical reinforcement in the composite. Incorporating

such nanofibers did not compromise other important properties such as density or porosity of the aerogels, but did enhance mechanical properties like modulus and tensile strength. It also introduced a great improvement in the strength of the initial hydrogel before cross-linking occurred [118].

Other research by Boday and Loy et al. in 2012 [120] looked at the introduction of polyaniline nanofibers into aerogels. With the addition of nanofibers by up to 3 wt% and a composite density of 0.074 g/cm³, the bending strength increased, compared to pure aerogel. The strength was 200 times higher than pure aerogel [120]. Silica nanowires are also another use of nanofibers in aerogels. Tang et al. in 2016 [121] found that when the silica nanowires were used in the range of 3.5–14 wt%, the specific surface area declined from 950 to 847 m²/g and the density enhanced from 0.151 to 0.193 g/cm³. While the silica wires in the aerogel improved the compressive strength from 0.3 to 3.5 MPa, the thermal conductivity increased from 0.021 to 0.027 W/(m.K). Both studies here clearly show the importance of using nanofibers to improve mechanical properties whilst maintaining the original characteristics of pure aerogels, such as their advantageous low density [121].

Research by Li et al. [119] prepared polydimethoxysilane hybrid silica aerogel with electrospun fibers. They found that the nanofibers were primarily responsible for allowing the samples to bend without breaking. Cracks form after considerable bending, however, this remains somewhat intact as the nanofibrous layers are anchored tightly together by the aerogel layer, which then prevents disintegration of the whole aerogel [119]. To further develop nanofiber reinforced silica aerogels, Wu et al. in 2013 [51] successfully applied an electrospun PVDF web in the silica sol-gel processing and formed aerogel composites with intact monoliths, improved strength, perfect flexibility, hydrophobicity, and a low thermal conductivity of 0.028 W/(m.K), which provides an increased potential for thermal insulation applications. With applying different PVDF electrospun mats using 18, 23, and 28 % solution with various microstructures of microparticles, combined microparticles-nanofibers, and nanofibers, Fig. 7A-C, it is shown that the fibrous structure of the electrospun mat strongly affects the final properties of the electrospun-reinforced aerogel composites, as with more fibers and less microparticle structure in the electrospun layer, structural properties are enhanced [51]. From left to right in Fig. 7, improved nanofibrous structure and better aerogel properties e.g. more flexibility and fewer cracks occurred [51].

Other candidates for the use of nanofibers in aerogel composites include cellulose. The textile industry is considered to be the next major cause of global environmental harm and pollution after petroleum and gas. Waste cotton fabrics are an ideal source to extract cellulose. Cellulose is abundantly available; it is renewable and environmentally friendly. There have been numerous studies that combine cellulose nanofibrils with silica aerogel [122–124]. For example, [125] proposed to immerse CNF aerogel in a two-step in situ sol-gel silica synthesis process to form a thermally stable and hydrophobic CNF-silica composite aerogel. It was found that the silica content and dispersion in the matrix play an important role in improving the properties of the CNF aerogel, which were better thermal stability (31.7 °C higher pyrolysis temperature) and super-hydrophobicity (water contact angle of 152°) in comparison to pure cellulose aerogel. The presence of silica particles in the cellulose scaffold reduced mechanical properties, but it can still be concluded that a cellulose aerogel with improved thermal properties and water resistance may lead to more functional application areas [125].

2.5. Overview of silica aerogel-fiber composites prepared by sol immersion

Fig. 8 summarizes some data available on the different fiber-reinforced aerogel composites and blankets. It can be concluded

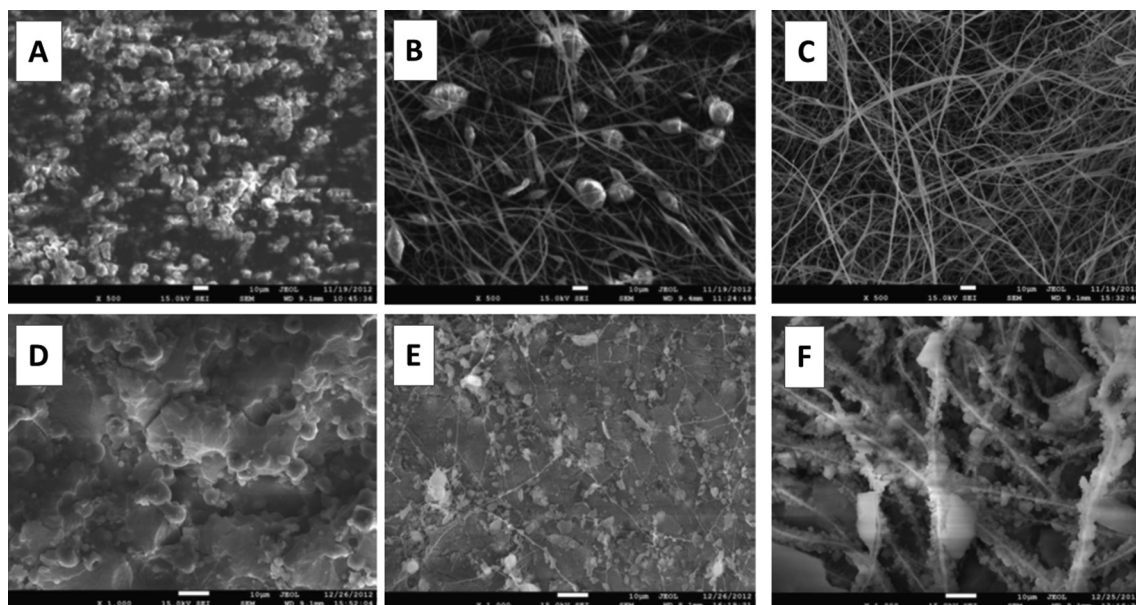


Fig. 7. Morphology and microstructure of three electrospun PVDF webs and their counterpart aerogel composites: A, D) microparticles (18 wt% PVDF), B, E) combined microparticles and nanofibers (23 wt% PVDF), and C, F) nanofibers (28 wt% PVDF). Revised/reproduced with permission from Ref [51].

that carbon fiber-induced silica aerogel materials showed the highest mechanical properties while thermal conductivity is high as well in comparison with neat aerogels and other fiber-induced silica aerogels. A majority of works have been done on glass fiber reinforcement, showing acceptable mechanical properties while keeping the thermal conductivity close to that of pure aerogels, followed by aerogel composites with ceramic fibers, which tend to have somewhat higher thermal conductivity and lower mechanical properties. The mechanical properties of aerogel blankets, such as less brittleness or higher cohesion between the fibers, do not necessarily translate into greater modulus or strength, and strongly depend on aerogels' density [39] and amount as well as fiber type, shape, and orientation in the composite. However, the advantages of combining the aerogel with textiles go far beyond the easily measured compressive properties. By filling the spaces between fibers with aerogels, aerogels behave like textiles, resulting in higher cohesion, ease of handling, and installation, and these blankets have become the most popular aerogel product on the market as a result. Research works on organic fibers have also developed recently. Here, polyester fibers showed enough acceptable properties to be used alone or with glass fibers in commercial aerogel blankets. In one research work, Tencel fibers-aerogel composites showed thermal conductivity in the range of glass fiber composites, but with better mechanical properties. Organic fiber-

loaded aerogels found their way to be used in other applications e.g. fire protective clothing, water/oil separating filters, hydrophobic fabrics, sound absorbing blankets, etc. beside their application for thermal insulation applications, but more research data on their properties and possible reactions between the gelation, exchange and hydrophobization solvents with the polymer are required. A summary of the properties of aerogel-fiber composites and blankets prepared by a sol-based approach is presented in Table 2 and Fig. 8.

3. Fiber-silica aerogel composites: Pre-formed aerogel particles/slurries

3.1. Thermal bonding and/or press forming

As described in section 2, silica aerogel blankets can be produced by soaking a nonwoven layer in the primary sol and synthesizing the aerogel in situ, i.e. uniformly distributed throughout the fiber matrix. Unlike the method of fiber impregnation in the sol (e.g. Aspen blankets [133,134]), aerogel blankets can also be formed by introducing pre-formed aerogel particles into a nonwoven layer or batting (e.g. Cabot blankets [135]). In this method, aerogel granules are spread on a nonwoven layer or web, and the second layer of nonwoven fiber is then placed on top of the aerogel

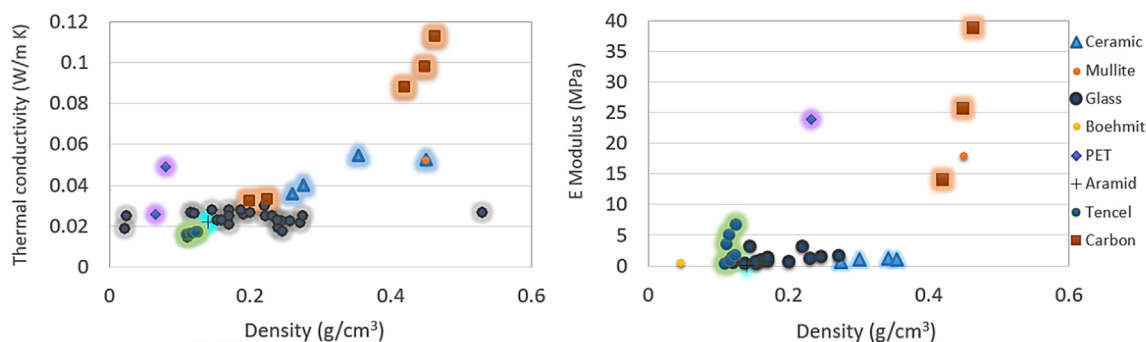


Fig. 8. Summary of research works in terms of thermal conductivity and E Modulus for a variety of fiber-induced silica aerogel composites and blankets.

Table 2

Summary of the research works on silica aerogel-textiles composite through a sol-based approach.

Author Year [Ref.]	Aerogel	Textile	λ (mW/m. K)	Mechanical parameter (MPa)	ρ_{\min} (g/cm ³)	Φ (%) A (m ² / g) d (nm)	Precursor Drying	Method	Variable	Investigated properties
Wang 1995 [75]	SiO ₂ Incl. TiO ₂	Al ₂ O ₃ -SiO ₂ - Fe ₂ O ₃	38 (526 °C)	40	0.26	–	TMOS SCD	Sol to fiber	TiO ₂ (10– 30 wt%)	High-temperature λ , E-modulus, sound velocity
Yang 2011 [65] 2012 [67]	SiO ₂	Al ₂ O ₃ -SiO ₂	–	EM: 7.3	–	–	TEOS SCD	Fiber added to gel	Compression (25–800 °C) Creep test (300–900 °C)	Compressive curve & E- modulus, Short- & long-term creep, modeling
Shi 2013 [68]	SiO ₂	Al ₂ O ₃ -SiO ₂	–	EM: 106 (In-plane)	–	–	TEOS SCD	Sol to fiber under vacuum	Compression (25–900 °C, sample direction)	In-plane and out-of- plane Compressive curve
Y. Yu 2018 [54]	Al ₂ O ₃ - SiO ₂	Al ₂ O ₃ -SiO ₂	40–54	EM: ~0.7– 1.2 FS: ~0.4–0.8	Al/Si: 0.30– 0.34 F: 0.2– 0.4	A:305 d:27.5	Al sol -silica sol SCD	Fibers dispersion in sol	Al/Si molar ratio (2–8) Fiber (40– 55 wt%)	Structural properties, flexural & compressive curve, E-modulus, λ , thermal stability
Yi 2022 [78]	SiO ₂	Mullite whisker-coated alumina-silicate	27.5	CS 50 %: 6.5	0.26	–	TEOS APD	Fiber impregnation under vacuum	Mullite coating via Mo-based or F-based catalyst	Compressive curve, E-modulus, λ ,
L. Xu 2015 [76]	Al ₂ O ₃ - SiO ₂	SiC-coated mullite	49 (1000 °C)	–	–	–	ASB-TEOS SCD	Fiber felt impregnation under vacuum	Pyrolysis & λ time & temp. PCS solution (1–25 wt%)	SiC effect on λ & effective specific extinction
He 2016 [25]	ZrO ₂ - SiO ₂	Mullite	52 82 (500 °C)	CS 7.5 %: 1.05 EM: 18	0.45	Φ :85 d:2–60	ZrO ₂ - SiO ₂ sol SCD	Fiber block to sol under vacuum	With/without aerogel λ test (ambient- 1200 °C)	SA and composites' structural properties, λ , compressive strength
Y. Yu 2019 [77]	Al ₂ O ₃ - SiO ₂	Quartz	49	EM: 0.85	0.36	A:580 d:16.7	TEOS AlCl ₃ , 6H ₂ O APD	Fiber to sol under vacuum	Heat treatment of composites (600–1100 °C)	Structural properties, compressive strength, λ , thermal stability
Kim 2008 [52]	SiO ₂	Glass	–	–	0.12	Φ :90 A:397 d:16	WG-TEOS APD	Fiber to mixed sols	WG/TEOS (0– 100 vol%)	Structural properties
Chandradass 2008 [91]	SiO ₂	Glass & Al- coated glass	–	–	0.12– 0.19	Φ :90– 95	WG-TEOS APD	Fiber wetting with sol	TEOS (0– 50 vol%)	Structural properties
J. Zhao 2012 [89,90]	SiO ₂	Glass	–	–	–	–	TEOS SCD	–	SiO ₂ , silicon, common float, soda-lime silica, borosilicate [89]; Inclina- tion angle (0– 30°), diameter (2–10 μ m), length-to- diameter ratio (1–1000) [90]	Radiative properties and heat transfer using modified anomalous diffraction theory
Wu 2014 [64]	SiO ₂	Multilayer glass	23–28	CS: 1.7–3.7 FM: ~0.2– 0.4	0.16– 0.19 SA: 0.115	– A:867.9 d:5–10	TEOS APD	Fiber to sol, sequentially	Alignment of four fiber Layers	SA & composites' λ , compressive and flexural strength
Yang 2017 [70]	SiO ₂ Incl. TiO ₂	Glass	19–21	–	0.13– 0.19 SA: 0.11	Φ :86– 91 A:898.9 d:11.2	TEOS APD	Fiber dispersion in sol	TiO ₂ (1.5– 9.3 wt%)	SA/composites' structural and thermal properties numerical/analytical analyses
Jiang 2017 [81]	SiO ₂	Microglass	13–22 (200– 650 °C)	FS:1.4	SA: 0.129	–	TEOS APD	Fiber mat to sol under vacuum	Silylating agent/TEOS molar ratio (2–6) Fiber (4.5– 9.1 vol%)	SA & composites' structural properties, λ (200–650 °C), flexural curve
C. Li 2017 [58]	SiO ₂	Glass	23–28	EM: ~0.5– 0.8	0.17– 0.153	A:868– 964 d:8.6–	TEOS APD	Fiber to sol	H2O: TEOS molar ratio (2–6)	Compressive curve and E-modulus, λ , combustion

Table 2 (continued)

Author Year [Ref.]	Aerogel	Textile	λ (mW/m. K)	Mechanical parameter (MPa)	ρ_{\min} (g/cm ³)	Φ (%) A (m ² / g) d (nm)	Precursor Drying	Method	Variable	Investigated properties
Haq 2017 [66]	SiO ₂	Glass	21–26	FS: 7 TS: 4	0.17– 0.19 SA: 0.12	13.9 A:60– 645	MTMS Freeze drying	Fiber to sol	Silica content in primary sol (5–32 %)	Structural properties, λ , flexural/tensile strength, hydrophobicity
Zhou 2018 [79]	SiO ₂	Glass	25–29	EM: 0.6–3.2	0.17– 0.22	A:487– 870 d:7.5– 13.7	MTMS-WG Freeze dry	Fiber to sol	MTMS/water glass molar ration (0–1.8)	SA & composites' structural properties, λ , compressive & bending curve
Z. Fei 2018 [82]	SiO ₂ - polyimide	Glass	27–28	CS at 8–9 %: ~0.1–0.3	0.116– 0.15	A:844– 963 d:14.8– 17	ODA, PMDA, TEOS SCD	Fibers dispersion in sol	Polyimide (2– 10 wt%) Glass fiber (1– 3 wt%)	Structural properties, λ , compressive curve
Nocentini 2018 [57]	SiO ₂	Glass PET	15–20	CS at 10 %:~58 TS: 0.8	0.11 SA: 0.11	A:90 d:2–50	TEOS Microwave- APD	Sol to the fiber layer	SiO ₂ -Glass SiO ₂ -PET	Fiber type (glass & PET)
Huang 2018 [80]	SiO ₂	Glass	23–25	EM: ~0.1– 1.0 FM: ~0.3– 0.7	0.157– 0.18	Φ :~85– 87 A:512– 1091 d:9.1– 15.7	WG APD	Fiber web to sol	Water/Si molar ratio (12–24)	Microwave drying Structural properties, λ and thermal stability, flexural & compressive curve, flammability, hydrophobicity
Iswar 2019 [15]	SiO ₂	Glass PET	16.3 SA: 15.4	–	SA: 0.1	–	TEOS SCD, APD	Fiber blanket in sol	Aging and drying process EtOH/Sol (38.7–72.5 vol %)	Structural properties via X-ray microtomography, λ
Huang 2019 [83]	SiO ₂	Glass	24–30	FM: 2–12	0.15– 0.20	Φ :83– 88 A:233– 377	WG APD	Sol to fiber		Structural properties, λ , flexural curve & modulus
S. Shafi 2019 [84]	SiO ₂ - Fumed silica	Glass	19–25	FS: 0.3–0.6	0.22– 0.244 SA: 0.217	– 805– 1149 9.6– 13.2	TEOS Fumed silica SCD	Sol to the fiber slice	Fumed silica (1–12 wt%)	SA & composites' structural properties, λ , flexural curve
S. Shafi 2019 [85]	SiO ₂ - Silica gel	Glass	18–28	EM: ~1.1– 1.7	0.226– 0.27	Φ :87– 92 A:762– 1135 d:7.9– 9.6	TEOS, Silica gel SCD	Silica gel- mixed sol to fiber slice	Silica gel (1– 9 wt%)	Structural properties, λ , compressive curve
S. Shafi 2020 [86]	SiO ₂ - Silica gel	Glass	18–26	EM: ~0.7– 2.3	0.236– 0.28	Φ :86.5– 91 A:604– 1077 d:8.1– 15.2	TEOS- MTMS Silica gel SCD	Fiber to sol	MTMS/ TEOS molar ratio (0–1) Silica gel in sol	Structural properties, λ , compressive curve
Patil 2020 [69]	SiO ₂	Glass, CNT Graphene	–		SA: 0.418	–	Simulation	–	Filler type: Glass fiber, Graphene sheet, CNT	Tensile/compressive curve, E modulus, toughness
X. Li 2013 [53]	SiO ₂	Sepiolite	20–25	CS at 10 %: 0.5–1.1	0.19– 0.21 SA: 0.12	A:795– 950 d:2–60	TEOS SCD	Fibers dispersion in the sol	Fibers (0.5– 1.5 vol%)	SA & Composites' structural properties, compressive strength, λ
M. Li 2018 [55]	SiO ₂	Xonotlite	28–33	–	0.126– 0.15 SA: 0.14	A:633– 705 d:12– 14	TEOS APD	Fibers dispersion in the sol	Fiber (2.1– 16.8 wt%) Composites calcination	SA & composites' structural properties, λ , hydrophobicity, chemical reactions
Hayase 2016 [74]	PMSQ	Boehmite		EM: ~0.5– 24	0.046– 0.23	Φ :84.2– 98 A:159– 405	MTMS SCD	Silica sol to dispersed fiber sol	Fiber (0.9– 6.4 wt%) MTMS/12 ml sol (2.5– 10 ml)	SA & composites' structural properties, λ , compressive curve and modulus
Talebi 2014 [94,95]	SiO ₂	PET nonwoven	21–32	–	SA: 0.284	Φ :80 A:660 d:11	TEOS APD	Nonwoven felt into sol	Sol/PET vol. ratio (1.9, 2.4)	Temperature distributions curves, numerical thermal diffusivity and λ
Oh 2009 [61]	SiO ₂	PET nonwoven	SAC	–	0.053– 0.18	–	TEOS APD	Fiber to sol and gel	Impregnation in sol or gel Gelation pH	Structural properties, λ , sound absorption, hydrophobicity

(continued on next page)

Table 2 (continued)

Author Year [Ref.]	Aerogel	Textile	λ (mW/m. K)	Mechanical parameter (MPa)	ρ_{\min} (g/cm ³)	Φ (%) A (m ² / g) d (nm)	Precursor Drying	Method	Variable	Investigated properties
Ramamoorthy 2018 [71]	SiO ₂	PET nonwoven	SAC	–	0.101– 0.16	–	TEOS APD	Fiber layer to sol	(7–10) MeOH:TEOS molar ratio (28–110)	Structural properties, sound absorption, water contact angle, TGA
Hoque 2021 [97]	SiO ₂	PP	–	–	–	–	PEDS APD	Fiber layer in sol.	Aging times, drying conditions	Structural properties, Heat conducting performance
Z. Li 2016 [93,101]	SiO ₂	Aramid	22	EM: 0.14 FM:1.4	0.14 SA: 0.15	Φ :84 % SA: 866 SA: 12.4	TEOS APD	Fibers layer by layer to alcosol	Fiber (1.6– 11.4 %)	Structural properties, compressive curve, E-modulus, λ , TG- DSC
Chakraborty 2016 [103]	SiO ₂	Meta aramid	23–38	–	0.09– 0.12	–	TEOS APD	Sol to fiber batting	TMCS (1– 20 vol%)	SEM, FTIR, λ and thermal stability, hydrophobicity
He 2017 [104]	SiO ₂	Aramid	32	–		Φ :96 d:3.5	TEOS APD	Fiber dispersion in sol	Fiber grafting with GPTMS and SA	Aerogel covered fibers' λ , chemical/ structural properties
Huang 2017 [105]	SiO ₂	PTFE coated aramid			SA: 0.003	–	–	Silica aerogel felt embedded under fire conditions	Dynamic degradation (20–1000 °C)	Thermal decomposition and TGA
An 2021 [102]	SiO ₂	Aramid	34	EM: 1.1	0.08	–	WG CTAB APD	Fiber to sol (vacuum filtration)	Aerogel (20– 90 wt%)	Structural properties, λ and thermal stability, flexural & compressive curve & modulus, flammability
Almeida 2021 [107]	SiO ₂	Aramid	28–62	EM: ~0.2–1	0.13– 0.22	Φ :81– 90 A:311– 780 d:24– 49	TEOS-VTMS APD	Fiber to sol	Fiber type: Twaron, Technora, Teijinconex, PAF, KP	Structural properties, λ & stability, compressive curve & modulus
Jaxel & Fiber length (2–12 mm) Fiber (0.5– 2 vol%)	SA & SiO ₂	Markevicius 2017 [108,109] composites' structural properties, λ ,	SiO ₂	Man-made cellulose (TENCEL)	15–18	SCD EM: 3.5–7 APD EM: 0.3–1.8	0.1–0.13 SA: 0.103	A:600–700	PEDS SCD APD	Sol to fiber flexural/compressive stress, E-modulus
Shi 2017 [60]	SiO ₂	Polyacrylonitrile	–	EM: 0.26	SA: 0.086	Φ :95 A:230	MTMS- DMDMS APD	Fiber dipped in methanol/ ethanol	Molar ratio: Methanol/ MTMS (2–18) DMF/MTMS (0.1–1) DMDMS/ MTMS (0.3–1)	SA & composites' structural properties, oil and solvents' absorption, hydrophobicity, E- modulus
Wang 2018 [113]	Si-PR	Carbon (rayon- based)	89–116	CS at 4–5 %: ~0.3–2.4 EM: 10–40	0.402– 0.46	Φ :64– 71 A:53– 143 d:30– 60	PR-HMTA- APTES- MTMS-EG APD	Fiber to sol	MTMS/PR weight ratio (0.14–0.54)	Structural properties, λ , compressive curve & E-modulus, ablation
Slosarczyk, 2017 [114]	SiO ₂	Carbon	25–36	TS: 750	1.56 – 0.2	Φ :90– 92 A:503– 590 d:9.9– 11.4	TEOS APD	Fibers dispersion during the gelation.	Carbon fiber 10–15 vol%	Structural properties e.g. SEM, BET, TGA
Slosarczyk 2021 [115]	SiO ₂	Carbon	32–33	CS: ~0.04–0.05	0.199– 0.34 0.209	Φ :467– 571 d:12– 14.6	WG APD	Fiber to sol	Carbon fiber (1–15 vol%)	SA & composites' structural properties, λ , compressive stress & E-modulus
Hung 2021 [62]	SiO ₂	Carbon/Glass	31–51	FS: 1.9–2.9	0.13– 0.15	Φ :81.1 A:861.7 d:3.09	TEOS APD	Sol to fiber	Glass fiber (3.5–50 %) Carbon fiber (3.5–15 %)	SA & composites' structural properties, λ , flexural curve

Table 2 (continued)

Author Year [Ref.]	Aerogel	Textile	λ (mW/m. K)	Mechanical parameter (MPa)	ρ_{\min} (g/cm ³)	Φ (%) A (m ² / g) d (nm)	Precursor Drying	Method	Variable	Investigated properties
Cheng 2021 [116]	Si-PR	Carbon/Quartz	112–133	CS: 5.9–17	0.47– 0.52 SA: 0.16– 0.2	Φ : 64.6– 68 d: 20– 80	PR-HMTA- APTES-EG APD	Sol to fabric under vacuum	APTES: PR weight ratio (0.25–1.0)	Structural properties, λ , compressive curve, E-modulus, NMR

CS: Compressive strength, EM: E-modulus or Young's modulus, FS: Flexural or Bending Strength, FM: Flexural or Bending modulus, Temp.: Temperature, SAC: Sound absorption coefficient.

SA: silica aerogel, TEOS: Tetraethoxysilane, TMOS: Tetramethoxysilane, MTMS: Methyltrimethoxysilane, WG: Water glass.

ASB: Aluminum sec-butoxide, ODA: 4,4'-oxydianiline, PMDA: Pyromellitic dianhydride, PEDS: Polyethoxydisiloxane, PR: Phenolic resin, HMTA: Hexamethylenetetramine, APTES: (3-aminopropyl) triethoxysilane, EG: Ethylene glycol, PTFE: Polytetrafluoroethylene, CTAB: Cetrimonium bromide, PCS: Polycarbosilane, GPTMS: Glycidyoxypropyl trimethoxysilane, PMSQ: Polymethylsilsesquioxane.

layer. This sandwich structure is then fused together at a high temperature, e.g. 160 °C, and/or under pressure to entrap the particles in the nonwoven structure and bond the nonwoven fibers together to achieve the desired thickness and low detachment of the aerogel particles. This technique can take advantage of using bi-component fibers in the nonwoven layer with a higher and lower melting polymer and the heating process can be done above the melting point of the lower melting polymer [131]. Good examples of fibers are polyester and/or polypropylene or polyethylene [72,136], while recently the use of a nonwoven layer of viscose fibers, due to their excellent hydrophilicity and flexibility, was also reported by Shaid et al. in 2021 [131]. A fusible interlining was also embedded between the two layers of fabric to enhance layer integrity. Fig. 9 illustrates an example of an aerogel blanket.

A similar method for embedding aerogel particles in PET nonwoven fabrics was reported by Xiong et al. in 2016 [136] in which silica aerogel particles were added to the layer with spraying a low melting thermal adhesive powder, followed by placing a PAN nanofiber web on the top and applying a heat treatment of 110 °C to form a layered aerogel embedded PET blanket with thermal conductivity values of 0.036–0.037 W/(m.K), based on the area density of the embedded aerogels. In a series of works with Venkataramana et al. in 2014–2017 [126–130] on this aerogel thermally bonded- nonwoven blankets, the experimental and numerical modeling of thermal properties including conductivity, convection, and diffusivity showed a much higher performance at ambient and subzero temperatures for the aerogel treated samples. While the main application developed for these blankets is thermal insulation, sound absorption coefficient of the aerogel-(polyethylene/polyester) nonwoven bonded blankets was investigated by

Yang et al. in 2019 [72], in which the sound absorption coefficient linearly increased with increasing frequency [72].

While the general studies were on forming an aerogel blanket, Tian et al. in 2020 [87], presented a facile heating-compressing approach to prepare glass fiber-fumed silica-reinforced silica aerogel composite using ball milling of the silica aerogel and fumed silica according to the desired weight ratio, then mixing it with glass fiber, followed by pressing into a mould. Optimum results of low thermal conductivity of ~ 0.023 W/(m.K), density of ~ 0.26 g/cm³, and flexural modulus of 2.56 MPa was achieved with the silica aerogel: fumed silica ratio of 1:1 under a pressure of 1 MPa since the structure was not too loose (leading to heat convection) and not too compact (leading to heat conduction) [87]. Press forming of mixed silica aerogel powders and glass fibers while using TiO₂ as an opacifier, was also investigated by Yual et al. [73]. They investigated how to use forming pressures at 0.5–2 MPa and found a pressure of 1.5 MPa to be optimal for heat insulation. By adding the glass fibers, the strength of the composites improved but the heat insulation properties deteriorated. The thermal conductivities of the composites with 20 wt% glass fibers at 300 and 600 °C were 0.025 and 0.030 W/(m.K), respectively. As an opacifier, the addition of TiO₂ decreased the radiative thermal conductivity abruptly, particularly at high temperatures e.g. from 0.041 to 0.030 W/(m.K), at 700 °C, with the addition of 20 wt% TiO₂ [73].

3.2. Aerogel particle slurries injection/permeation in between the fibers

This method is similar to the method above, but here a particle slurry, prepared by mixing pre-made silica aerogel powders (e.g. 20 μ m particle size) with a solvent (e.g. an organic solvent, alcohol

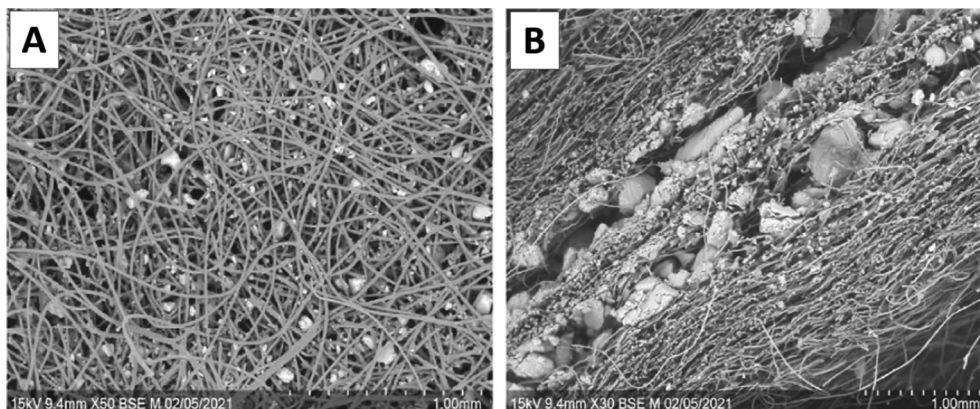


Fig. 9. SEM images of fabric surface and cross-sectional view of an aerogel-nonwoven blanket by thermal bonding method. Revised/reproduced with permission from Ref [131].

or non-polar solvent), is injected into a supplying fabric (e.g. a glass fiber mat, PET or PP nonwoven fabric), followed by removing the solvent from fabric, e.g. with hot air blowing, to form an aerogel blanket. Injecting the mixture with a needle directly into the fabrics solves the problem with a long-time permeation process and separation of the wetting agent from the outer surface in a system using a blade. However, an example of a velocity of injection can be 20 cm/min for a 10 mm thick glass fiber mat with a density of 0.11 g/cm³ [132]. Such an approach, proposed by Armacell-JIOS Aerogels Ltd. [132], simplifies the sol-gel and drying processes because it reverts simply to the production of a silica aerogel powder, which can be produced more cost effectively than silica aerogel blanket through a primary sol approach. However, maximizing the filling factor of aerogel in the pore space between the fibers sufficiently large gaps between the fibers (relative to the aerogel powder particle size), which makes the retention of the powder by the fiber matrix more difficult. Typically, a compromise has to be found leading to somewhat higher thermal conductivities and/or stronger dust release of the final products. A summary of the mentioned studies on aerogel incorporated fibers with pre-formed silica aerogel particles is presented in Table 3.

3.3. Overview of aerogel-fiber composites with pre-formed aerogel

The finished products from this method aim to be comparable in performance to sol-impregnated aerogel blankets, and some products are also commercially available. However, there is a greater likelihood of dusting with less binding between aerogels and fibers; after all, the fiber network had to be open enough to get the aerogel grains in. There is also a probability of higher thermal conductivity because the space filling with aerogel is expected to be lower after powder slurry impregnation. In terms of production complexity, the production process consists of two steps: synthesizing aerogels and loading them onto fibers and during the second step, rewetting of aerogels can degrade their porous structures unless careful wetting/drying cycles are implemented. On the

other hand, the synthesis of aerogel powder is more straightforward and affordable, compared to a sol-based aerogel blanket, because adding fibers in the sol impregnation method complicates the aerogel-production process, which is more similar to that of monolithic aerogels with similar limitations (issues with heat and mass transport and diffusion, handling of large rolls of blankets, sizing of supercritical drying autoclaves).

4. Silica aerogel surface coated textiles: Pre-formed aerogel

Since demands for advanced aerogel-based materials with functional performance have increasingly been extended, attempts have been made to introduce coating methods in which the main problem of the aerogel-textiles composites, dust-releasing from the fiber assemblies, is resolved. Rather than filling up the entire volume, silica aerogel particles are used in coatings in different technical textile applications. The good temperature resistance and low thermal conductivity of the aerogel coatings are the predominant reasons for their use as coating material. Other applications focus on the hydrophobic characteristics of the coatings to obtain beneficial moisture handling or self-cleaning textile characteristics.

The textile substrates can be coated by silica sols or silica aerogel particles-filled materials via different techniques, e.g. sol-gel coating, dip-coating, spin-coating, and spray-coating including electro spray-coating. When the sol is coated on a substrate, the dispersing medium constituting the sol is evaporated and the gel forms a layer on the surface of the substrate. The dip-coating technique is based on three general steps. The textile is first dipped in a coating slurry, then dried and finally cured. The coating slurry is composed of nano and micro-scale particles, which are dispersed in an organic solvent. A polymer in the slurry works as a binder, improving the bonding strength of the coating layer [137].

The spray-coating technique was also applied using polymers with low surface energy, like aerogel-fluorocarbon electro-spraying on a polyester woven fabric, which was introduced by

Table 3

Summary of the research works on silica aerogel-textiles blankets/composite with pre-formed silica aerogel.

Author Year [Ref.]	Aerogel	Textile	λ (mW/m.K)	Mechanical parameter (MPa)	ρ_{min} (g/cm ³)	Φ (%) A (m ² /g) d (nm)	Method	Variable	Investigated properties
Yuan 2012 [73]	SiO ₂	Glass	30 (700 °C)	CS: 0.3–1.2	0.35–0.53	–	Press: fibers, aerogel, TiO ₂	Fiber and TiO ₂ (0–20 wt %) Compressing pressure	Compressive strength and high temp. λ
Venkataraman 2014–2017 [126–130]	SiO ₂	PET/PE nonwoven	26–49	–	0.066–0.08 SA: 0.12–0.14	Φ :98 d:20	Pre-formed aerogel-fiber layer thermal bonding	Fabric density (0.066–0.08 g/cm ³), thickness (3.5–6.6 mm), aerogels (1–2 %)	Thermal properties e.g. λ , diffusivity, convection, air permeability and sound absorption
Yang 2019 [72]	SiO ₂	PET/PE nonwoven	Sound absorption coefficient	–	0.066–0.08 SA: 0.135	d:20	Pre-formed aerogel-fiber layer thermal bonding	Fabrics' density (0.066–0.080 g/cm ³), thickness (3.5–6.6 mm), lamination	Air permeability and sound absorption
Tian 2020 [87]	SiO ₂	Glass	23–25	FM: ~1.8–3 FS: ~0.3–0.7	0.238–0.28	A:668 d:6.58	Press of fibers, aerogel, fumed silica	Fumed silica/aerogel (0–9 wt%) Compressing (0.5–2 MPa)	SA & composites' structural properties, λ , flexural curve
Mishra 2021 [98]	SiO ₂	PET/PP	19–27	–	SA: 0.055	–	Market blanket (Cabot)	Fiber/aerogel (10/90–30/70 vol%)	λ , FE simulation using COMSOL and ANSYS
Shaid 2021 [131]	SiO ₂	Viscose	–	–	SA: 0.12–0.15	Φ – SA:700 d– SA:20	Pre-formed aerogel-fiber layer thermal bonding	Aerogel (1–4 g/900 cm ²)	Thermal comfort, Fabrics' strength and flexibility, thermal resistance
Joung 2021 [132]	SiO ₂	e.g. Glass, PET, PP	–	–	–	–	Pre-formed aerogel-solvent injection into supporting fabric	e.g. SA/solvent weight ratio (5–15)	System for injecting

Shams-Ghahfarokhi et al. [138], Fig. 10. The coating system is sprayed on the substrate before it is cured under certain conditions. After 24 h, heat transfer decreased from $\sim 71\%$ for the unsprayed fabric to $\sim 31\%$ for the electrosprayed fabrics with a contact angle of 152.2° . In addition, the weight losses of the sprayed samples after the abrasion test were slightly $<5\%$, indicating low dust release behavior of aerogels from fiber surface [138]. The spin-coating technique is comparable to sol-gel-coating. A coating layer is applied on the surface of the textile before the dispersing medium is evaporated. The described techniques are useful to create a homogeneous coating on the textile substrate, but they are linked with some major challenges. The fragility of the aerogel films and particles often leads to a defragmentation of the aerogels during the coating and handling of the coated materials. Moreover, a major challenge is the loss of insulating properties due to pore wetting, since an organic solvent is often needed for homogeneous dispersion of the particles. As a result, the durability of the inorganic aerogel coatings is usually limited. This major challenge of the aerogel coatings is tried to be overcome by constructing hybrid organic-inorganic aerogel composite coatings [139,140]. Some examples of organic-inorganic aerogel coatings and their effect on the textile properties are described in the following.

4.1. Examples of coating systems

In some instances, Shaïd et al. in 2015 [141] for the coating of wool/aramid blend fabrics, which could be used for firefighter clothing. Impregnated fabrics with a hybrid coating system containing 2–4 % of aerogel nanoparticles on the surface showed a significant increase of thermal resistance by up to $\sim 68\%$. The wearing comfort was also investigated, showing that the superhydrophobic aerogel particles can contribute to good moisture management when they are used on the next-to-skin layer. A further advantage is the reduction of the air permeability by more than 45 %. This coating system can be used for other textile applications as well to provide superior insulation in extreme hot or cold conditions [141]. In further investigations, Shaïd et al. in 2016–2018 [139,142,143] tested the incorporation of silica aerogel particles in phase change materials (PCM), also for firefighter clothes. PCM materials offer advanced thermal regulation properties due to their high heat storage capabilities. Unfortunately, most of these materials are inflammable. The mean ignition time of these PCM-containing coatings could be increased from 3.3 sec. to 5.5 sec. by incorporating silica aerogel particles in the coating. Moreover, the spreading of the flame could be reduced as well as the weight of the textiles, which are mainly suited for the inner liner of the

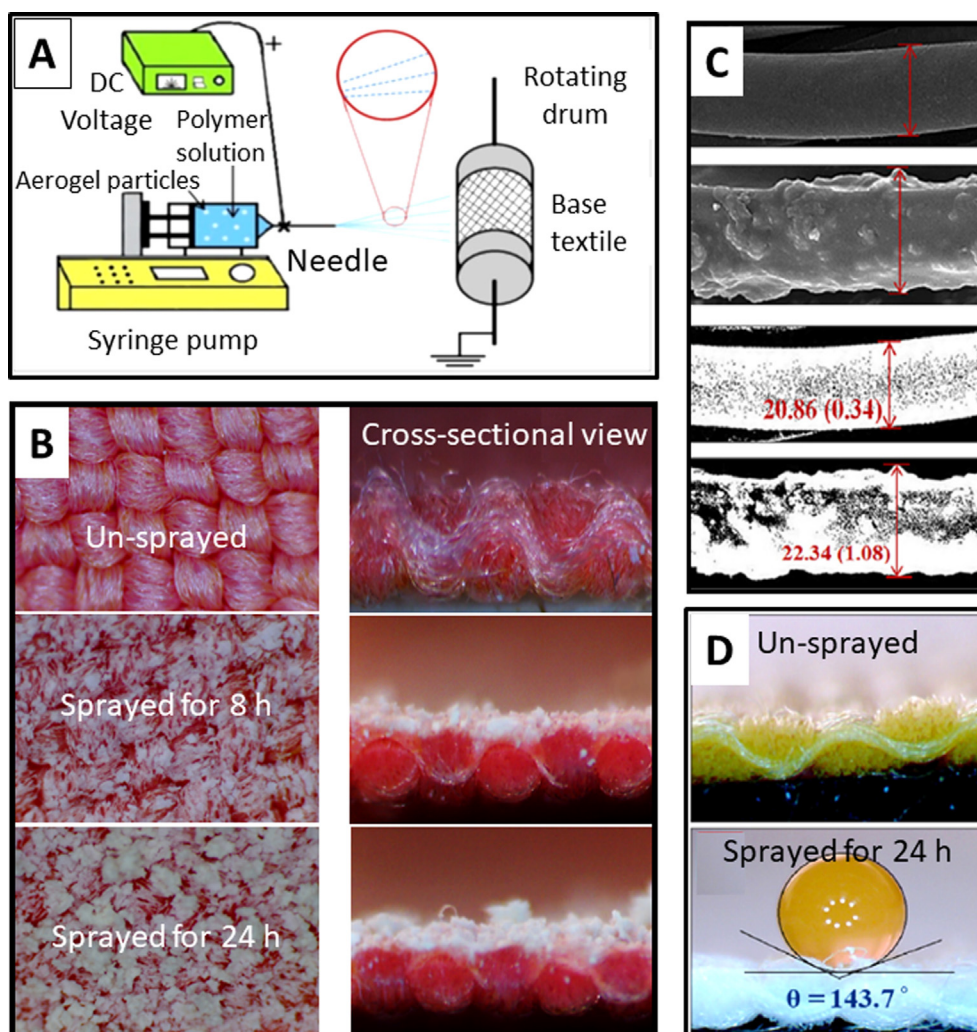


Fig. 10. A) Electro-spraying device for aerogel coating, B) digital microscopic photographs C) SEM micrographs and binary images, D) Water droplets on the fabrics coated with and without aerogel. Revised/. reproduced with permission from [138]

jackets, as described above [139,142,143]. A promising coating was composed of Aerogel/eicosane microparticles [139,144].

Jabbari et al. in 2015 [145] investigated silica aerogel coatings on PET-woven fabrics. A coating of aerogel-doped poly(vinyl chloride) (PVC) was used. The thermal insulation and moisture handling were improved. In contrast to the results of Krzemińska et al. [146], no negative effect on the degradation behavior could be observed with this polymer and coating system [145]. In Krzemińska's study [146], dispersion of polychloroprene latex and synthetic resins is compared with an acrylic-styrene dispersion with 6–14 % aerogel on aramid fibers, in regard to their resistance against thermal radiation and thermal degradation behavior [146].

Yan et al. in 2018 [147] by a simple method of dipping covered PET and cotton as well as hybrid fabrics with silica aerogel for thermal protective garments. Thermal insulation and hydrophobic properties of the silica aerogel-polyurethane (PU) coated cotton fabrics were also investigated by Wang in 2018 [148] and Bhuiyan et al. in 2019 [59]. Contact angles formed on PU aerogel-coated fabrics were similar to PU-coated fabrics. However, instead of liquid repellency resembling a PU coating, most of the chemicals were adsorbed in the aerogel coating [59].

The coating with silica aerogel particles was also tested for industrial products in 2021 [149]. The coating is used on polyester textiles for outdoor clothing with a beneficial effect on moisture handling and thermal insulation and a low soil release during repeated laundering. The clothes were impregnated with silica aerogel paste and binder at lower temperatures and time than that applied for the conventional coating method. Thus the coating with silica aerogel systems can also contribute to energy saving [149]. Further developments in coating technologies and textile substrates are summarized by Mekonnen et al. in 2021 [140].

4.2. Overview of aerogel surface coated fibers

Maintaining the aerogel's porosity, i.e. avoiding infiltration with polymers during mixing, remains the biggest challenge in this method and remains an open question with limited published data and a strong need for more systematic experiments. When these issues are solved, this method can be used for technical textiles and clothing as well. For the aerogel surface coated textiles described in this section, the aerogel amount in the structure is much lower than the amount in the aerogel blankets/composites formed in sections 2 and 3. The viscosity of polymer matrix, its chemical base, and order of mixing materials are the most important parameters while mixing with aerogels. Hydrophobic silica aerogels, the most popular aerogels on the market, are easily penetrated by organic solvents. Alternatively, silica aerogels float on water-based materials due to their low density and hydrophobicity. Hence, high-viscosity water-based materials could protect hydrophobic aerogels' porosity until coating or curing on textiles. Even though the thermal conductivity of the fabrics is not as low as that of aerogel blankets, the main goal of developing these materials would be to improve textile thermal insulation while simultaneously providing a hydrophobic coating or self-cleaning properties.

5. Silica aerogel-filled fibers: Pre-formed aerogel

Although most aerogel/fiber compositions are made in fiber-reinforced aerogels through immersion of fibers in the sol-gel state (as mentioned in section 2.1), the aerogels' lack of bonding into the fibers and the dusting problem are the main challenges in the mentioned products. However, there are some efforts in an inverse model to make aerogel-filled fibers, motivating investigations on the use of aerogel particles as a porous filler inside the polymer

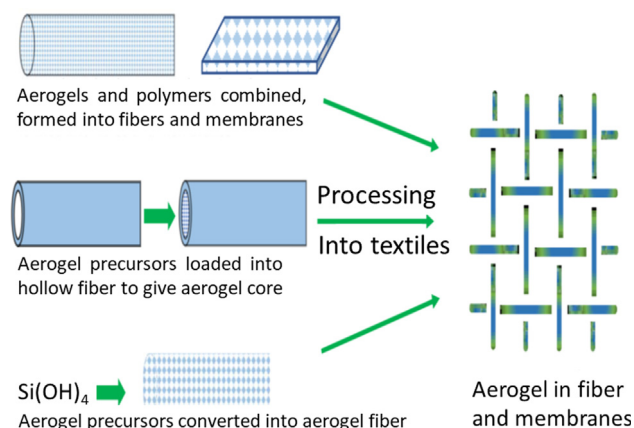


Fig. 11. Different methods for incorporation of aerogels in fibers. Revised/reproduced with permission from Ref [28].

phase, where it is completely protected [150]. This idea can be a solution for dusting problem of the commercial aerogel blankets. Fig. 11 shows the possibilities for incorporating/forming aerogels in a fibrous shape.

As shown in Fig. 11, aerogels can be combined or filled in a polymer matrix for making a fiber. Due to the porous and fragile structure of aerogels, the compounding parameters and the porosity of composite fiber are questionable. Also, aerogels can be incorporated into a hollow fiber after spinning. This idea gives higher porosity due to the higher aerogel and air content. The last method for making aerogel fibers is more expensive and time-consuming (this method is described in the next section).

5.1. Melt spun aerogel-filled fibers

Additionally, aerogels have been used as reinforcement fillers in thermoplastic nanocomposite compounds. In a research work by Krishnaswamy et al. in 2018 [151], a low-speed (5 RPM) compounding process through a twin-screw extruder resulted in polyamide 6/silica aerogel nanocomposite with 0.04 wt% filler content, while the higher speed process (65 RPM) collapsed the aerogels. They showed that the aerogels were not largely damaged during the experiments, while their further experiments indicated lowering the thermal conductivity by 0.39 W/(m.K). In contrast, compression molding (for 5 min under 2.76 MPa pressure), caused the aerogels to be damaged in the pelletized nanocomposites. These challenges highlight the novelty of the concept for the processing of aerogel-nanocomposite compounds. Either Yoda et al. in 2021 [152] used silica aerogels to reinforce polypropylene foam to make a light-weight insulating composite blanket.

Furthermore, melt-spinning is the most conventional, and the fastest method for fiber production from thermoplastic polymers [32]. The proposed procedure is illustrated in Fig. 12. Also, Wu et al. in 2021 [153] prepared a ternary blend to fabricate composite textiles. This material consists of recycled polyester from plastic bottles, (rPET), oyster shell powder (OSP), and silica aerogels (SAG). In addition to the high hydrophilicity and poor thermal conductivity of rPET textiles, OSP is included for antibacterial effect. With the addition of SAG, the hydrophilicity was improved and the thermal conductivity was reduced [153]. A recent literature review on nanometer materials in textiles states that fabrics are not only limited to keeping warm, but also require waterproofing and antibacterial properties [154–156]. Adding SAG or ASAG (antibacterial SAG) to rPET improved mechanical and thermal properties by up to 0.6 % for rPET/SAG and rPET/ASAG filaments compared to rPET filaments. However, after 0.6 %, it was observed that the

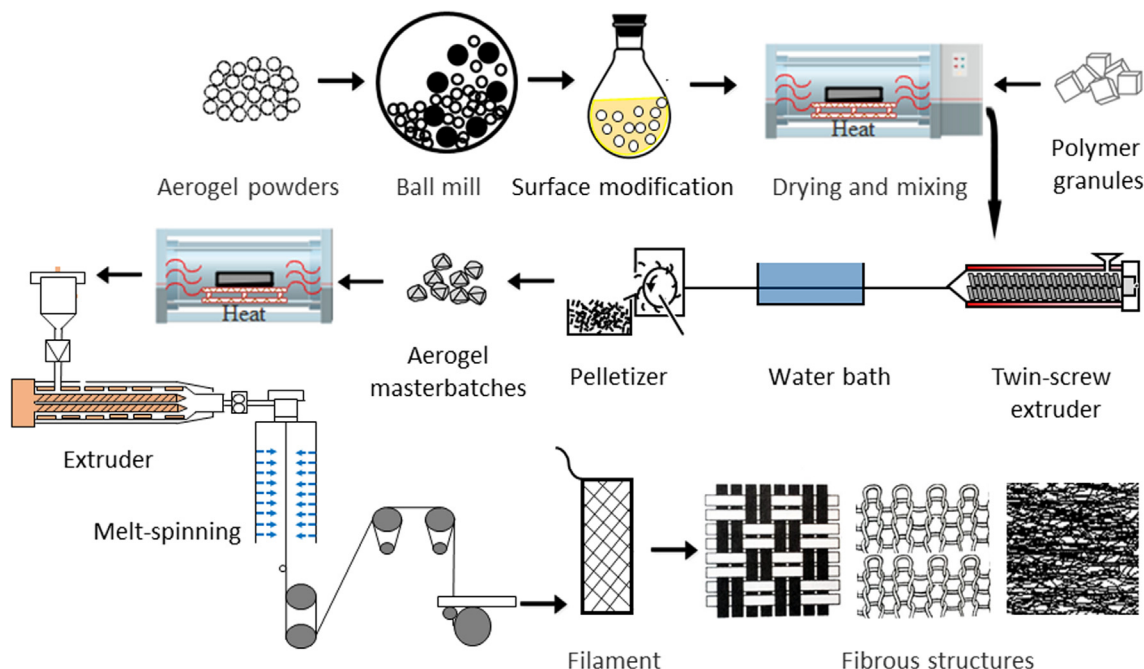


Fig. 12. Schematic diagram for the melt-spinning of aerogel-filled fibers from surface treatment, and compounding to the fiber and fabric production.

mechanical properties decreased rapidly due to agglomeration. Materials obtained as a result of the work of Wu et al. [153] exhibited enhanced temperature regulation properties as demonstrated by thermal imaging [10]. Continuing the work on the aerogel-filled melt spun PET fibers, Sedighi et al. [157] recently investigated the successful production of fibers up to 2 % aerogel fillers, with a roughening effect, due to the fillers, on the fiber's surface, leading to more hydrophobic properties [157].

5.2. Wet spun aerogel-filled fibers

Besides melt-spinning, wet-spinning and electrospinning are the other techniques for fiber formation from polymers. Other fillers such as aerogels can be added into polymer solutions as well. For instance, Jahid et al. in 2020 [158] incorporated silica aerogels in polyurethane solution to make a composite membrane for thermal regulation of the human body through a cooling mechanism. Mazrouei-Sebdani et al. in 2015 [150,159] added aerogel micro granules to PET nanofibers in the electrospinning process and could reduce excessive dusting of aerogels significantly in this way. Also, super-fine aerogel-filled polyester fibers showed high hydrophobicity, high disperse dye absorption, and high insulation properties [150,159]. Furthermore, Shaid et al. [160] prepared a 1.5 wt% silica aerogel and polyvinyl alcohol solution for making a nano-web through needleless electrospinning and electrospinning via a curved wire inside the syringe tip [160]. In the same line, Venkataraman et al. in 2018 [161] used an electrospinning process to produce flexible polyurethane (PU) and polyvinylidene fluoride (PVDF) nanofibers embedded with silica aerogel. Electrospun PVDF nanofibers are preferred because they not only improve mechanical properties but also maintain low thermal conductivity. They aimed to produce materials with very low thermal conductivity, suitable for extreme conditions. As a result, thermal insulation improvement was achieved by increasing the number and weight per unit area of nanofiber membranes in the produced materials. In addition, by increasing the electrospinning time, materials with higher web thickness are obtained [161]. This resulted in a significant reduction in air permeability.

5.3. Overview of aerogel-filled fibers

Research shows that for up to a few weight percent loading silica aerogel inside the polymer, spinning is still possible, but there is a lack of literature on this topic. For a higher load of aerogel particles into the polymer matrix, for example 4 wt%, it is hard to form fiber. Similar to section 4, there is a lack of comprehensive information on how to assess the final porosity of the aerogel. If the fibers are less dense, and aerogel mesoporosity is retained, the heat conductivity would be lower. Hydrophobic aerogels with low surface energy and their roughening effect on the fiber surface lead to greater hydrophobicity for the final fibers. The size of the aerogel particles is crucial in this situation because they need to be small enough to be mixed and pass through spinnerets, but too small of a powder also makes mixing difficult because the aerogel particles tend to aggregate, are poorly distributed in the fibers, and have a higher likelihood of being completely filled with polymer in their pores. A summary of the mentioned studies in aerogel-incorporated fibers is presented in Table 4.

6. Silica aerogel-filled packages: Pre-formed aerogel

The other possible method to incorporate the aerogels into textiles is to produce aerogel-enhanced packages, of which only a few studies have been published to date. Krzemińska et al. in 2020 [162] studied thermal properties of aerogel enhanced packages, prepared in the form of aramid fabrics, which were coated with a polyurethane layer, Teflon membrane, or an acrylic-styrene dispersion, filled with aerogel particles, differing in terms of pouches' number and width. All packages were made by sewing pouches onto a bottom layer as one example shown in Fig. 13.

Package design was found to play a significant role in achieving high levels of performance in terms of radiant, convective, and contact heat protection, allowing its use as an insert in stronger protective clothing. The variant with the most pouches gave the best results, but only in the contact heat experiments did the type of textile fabric matter. It was found that the radiant and convective heat resistance of the packaging approximately doubled after

Table 4

Summary of studies on aerogel-filled fibers.

First Author Year [Ref.]	Aerogel	Polymer	Fiber production method	Properties
Mazrouei-Sebdani 2015 [150]	Sodium Silicate	PET	Electrospinning	Low dust-releasing behavior, high hydrophobicity, dyeability, and insulation
Mazrouei-Sebdani 2016 [159]	Silica	PET	Electrospinning	Water resistant, water vapor/air permeation
Shaid 2018 [160]	Silica	PVA	Needleless Electrospinning and Electrospinning	Super-fine fibers
Venkataraman 2018 [161]	Silica	PU, PVDF	Electrospinning	Super-low thermal conductivity
Jahid 2020 [158]	Silica	PU	Melt-blowing	Cooling effect through evaporation of respiration
Wu 2021 [153]	Silica	rPET	Compounding & melt-spinning	High hydrophilicity, poor thermal conductivity and antibacterial properties

**Fig. 13.** A photograph of prepared aerogel-enhanced textile packages. Revised/reproduced with permission from Ref [162].

incorporating aerogel, with an approximately eightfold improvement in contact heat at the highest test temperature of 250 °C. Threshold time increased from 17.7 to 139.9 s for the best packaging variant improved with aerogel with most bags meeting the highest performance level criteria. However, further studies should be carried out to determine the best package shape and size for individual user requirements [162].

7. Spun silica aerogel fibers: Sol-based approach

Aerogel particles inside impregnated textiles can be released as dust when the textiles are subjected to bending stress and during general handling. Therefore besides using aerogels in combination with fibrous structures (which were described in previous sections) to maintain their flexibility for different applications, the aerogels themselves can be formed in a fiber shape through different methods [163]. This form of thin product can utilize higher flexibility and better performance, compared to aerogels in monolithic or granule form. Thus in fiber form, the brittleness of silica aerogels can be significantly reduced, providing them with higher flexibility, absorbing bending stress and vibrations without damaging the aerogel structure. This phenomenon can be observed in the case of glass and ceramic fibers and textiles.

7.1. Fiber chemistry

In the 1960 s, the structure formation of wet spun polyacrylonitrile fibers was studied by freeze-drying the spun gel fibers to

investigate the porosity and capillary formation of the filaments [164,165]. Thus cryogel fibers could be produced and later industrially developed for the purpose of moisture absorption under the trade name Dunova by Bayer AG, Leverkusen [166]. Highly porous fibers made of polyacrylonitrile (PAN), polyvinylidene fluoride (PVDF), polystyrene (PS), and poly-ε-caprolactone (PCL) were prepared in liquid nitrogen from Dimethylformamide (DMF) electrospun and then directly freeze-dried [167]. Porous polyester fibers were obtained in 2010 by foaming in supercritical CO₂ [168]. Moreover, some methods for the development of foamed fibers have been published in patents [169,170]. In recent years, other organic materials could be converted into aerogel fibers, e.g. kevlar [171], silk fibroin/graphene oxide [172,173]. Cellulose-based aerogel fibers [174,175]. Further development of these fibers was derived in Germany in 2015 to improve the resulting fiber characteristics [176–181], and recently, polyimide aerogel fibers [182].

Although organic aerogel fibers provide good results in thermal insulation, with thermal stability up to ~ 200 °C, inorganic aerogel fibers can withstand temperatures up to 600 °C or even higher and show good resistance to acids at the same time. Thus, inorganic aerogel fibers are interesting for moderate conditions as well as in the high-temperature range or in acidic atmospheres. In comparison to aerogel-loaded textiles or aerogel-filled fibers, the developed inorganic aerogel fibers show some major advantages. The developed fibers are flexible and can withstand vibrations and bending forces without damage or dust formation. Moreover, the recycling or disposal of the developed silica aerogel fibers could be easier, since it is not a hybrid material like other aerogel textiles such as fiber-reinforced silica aerogel mats. It can easily be molten and reused as glass fiber material. Since now it is only produced on a lab scale.

Silica-based aerogel fibers are developed in Germany since 2015 [183,184]. Tetraethylorthosilicate (TEOS) is used as starting material, which is synthesized and converted into fiber form in the wet spinning process. The manufacturing process was patented in 2015 (EP3250508A1). A similar development is published by a research group in China [185]. A different approach to producing silica aerogel fibers is published in 2019 [186], where the fiber is obtained from water glass and sulfuric acid in wet-spinning process. Besides, an inorganic aerogel fiber material from TiO₂ could be obtained by electrospinning process combined with supercritical drying [187].

7.2. Manufacturing process

The sol-gel process is based on the eponymous process of the transition of the reaction mixture from sol to gel. In addition to its economy, effectiveness, and mild reaction conditions, the sol-gel process offers the advantage of simplified control of the poros-

ity and the surface properties. This is achieved by adding suiting additives to the reaction system during the ongoing synthesis [188]. The sol–gel process is generally used for the synthesis of amorphous materials with a metal oxide backbone. It is divided into the steps of sol formation, gelation, aging, and drying [189]. To use the gels from the sol–gel process for fiber production, the gel composition and the aging have to be adjusted to obtain a spinnable gel. The process is based on the hydrolysis and gelation of tetraethyl orthosilicate (TEOS). The physical mechanisms and microstructural effects are described in more detail elsewhere [185]. The gels have to be optimized regarding their extrusion capability (mono and multifilament extrusion) and concerning their fiber formation properties. Some key findings on gel synthesis and aerogel fiber production have been made during a public-funded project (FKZ 03VP01502) [184]. The development of a reproducible process for the production of spinnable gels is the central challenge in the production of silica aerogel fibers. During synthesis, the energy input, the air exchange as well as the stirring speed are key parameters to control the progress of gelation. The use of suitable, corrosion-resistant vessels and materials is crucial for the reproducibility of the synthesis results. A spinnable solution can only be obtained when the synthesis conditions and time courses have precisely adhered since the cross-linking of the solution and thus the increase in viscosity are critical for the coagulation and fiber formation properties of the solution. The chemical reaction mechanisms involved are described in more detail in [188].

Fiber production is carried out in wet-spinning process. The wet spinning process is used to give shape to wet gels by controlled extrusion into an appropriate spinning bath [190]. The spinning of a gel takes place before it dries. For this purpose, the gel is extruded from a storage container through a nozzle with a large number of fine spinning holes into a coagulation bath. On a laboratory scale spinning plant, the number of holes per nozzle is usually between 10 and 500 holes and the hole diameter is in the range of 50 to 100 μm (Fig. 14), and the flow rate can be regulated by a gear pump. A basic chemical is used to coagulate the spinning solution in the spinning bath. This initiates further cross-linking of the gel-based on accelerated cross-linking condensation reactions. These mechanisms are called coagulation, which stabilizes the shape of the gel.

When silica sols are extruded into a spinning bath, the cross-linking of the silica network is completed, resulting in a silica gel in fiber form. These gel fibers can be washed and dried in subsequent steps. An important factor for a stable spinning process is the coagulation speed, which is mainly determined by the spinning bath composition, gel characteristics, and bath temperature. Coagulation must take place within a certain time frame after extrusion. If the coagulation time is too long, the extruded gel will not form filaments but the gel will distribute in the spinning bath. Moreover, the gel could form filaments that are sticking together and cannot be pulled off the bath for the subsequent washing and drying steps. On the other hand, a too short coagulation time leads to a blocking

of the spinning holes, as the gel solidifies immediately and sticks on the metal extrusion plate. Thus, the gel composition and viscosity and the composition of the spinning bath are influencing the spinning behavior and the resulting fiber properties. The fibers can be dried by evaporative or supercritical drying. For supercritical drying, the fibers are first subjected to a solvent exchange process. In this step, the water content in the fibers is reduced and the impurities from the coagulation bath are washed out. The fibers are transferred several times into ethanol. If the water content is too high, the fibers cannot be dried sufficiently because the solubility of water in supercritical CO_2 is limited. For subsequent drying, the fibers are loaded into a high-pressure container. At elevated temperature and high pressure (supercritical state of the solvent mixture), the container is flushed with supercritical CO_2 in several cycles, e.g. 1–2 cycles, because of the fibers' small dimensions. The small dimension and high surface area allow very short drying times, leading to reduce the production time of the fiber materials compared to monolithic samples. By using the supercritical drying method, the pore structure is preserved from capillary forces, resulting in higher porosity and typical aerogel nanoporous structure (Fig. 15), compared to an air-dried fiber. The mesoporosity of the fibers can be determined via physisorption measurements. The inner surface area is normally 500–1000 m^2/g for supercritical dried fibers and 50–100 m^2/g for air-dried fibers.

7.3. Overview of aerogel fibers

Aerogel fibers are completely different from the commercially available aerogel impregnated textiles. They have been produced from different organic and inorganic materials. Silica aerogel fibers were successfully produced by extrusion of a sol into a coagulation fluid, which controls the gelation and structure formation. Aside from the materials chemistry, key parameters to determine the final fiber properties are the spinning process and the drying of the fiber materials. The silica aerogel fibers are flexible and display the typical mesoporous microstructure with promising application characteristics, e.g. for lightweight temperature and noise insulation or catalysis technology. Although the development is still ongoing, the industrial application will be tested in the nearer future.

8. Modeling of aerogel-fiber composites

Modeling and numerical simulations of aerogel-fiber composites are extremely challenging due to the wide range in relevant length scales. The thermal conductivity in aerogels is dominated by the gas phase conduction (molecular scale). The aerogel itself is composed of nanoparticles/nanofibers (1–10 nm diameter) enclosing mesopores (2–50 nm), but is most often present as larger grains (2–50 μm diameter) between the fibers (0.1–50 μm diameter). No single simulation technique can resolve all of these length scales simultaneously. Due to this and the fact that many of the developments in the field have been very recent, modeling work is not abundant. However there still are a couple of interesting works to report on both mechanical and thermal transport properties.

8.1. Thermal conductivity

The bulk of theoretical works on silica-fiber and silica-textile composites use analytical models to get a better idea of the influence of the composite structure on the thermal transport properties [64,70,89,191], sometimes within the context of a study of material degradation and aging under different conditions [192–195]. One of the only full-scale simulations has been done by Petru

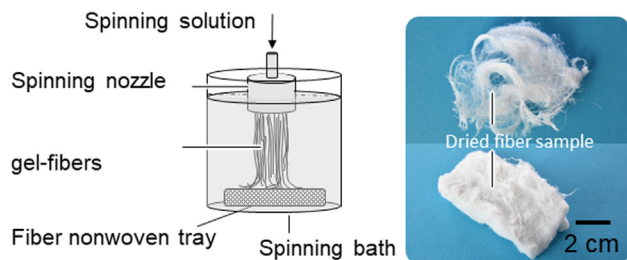


Fig. 14. Lab-scale manufacturing process schematic and dried silica aerogel fibers.

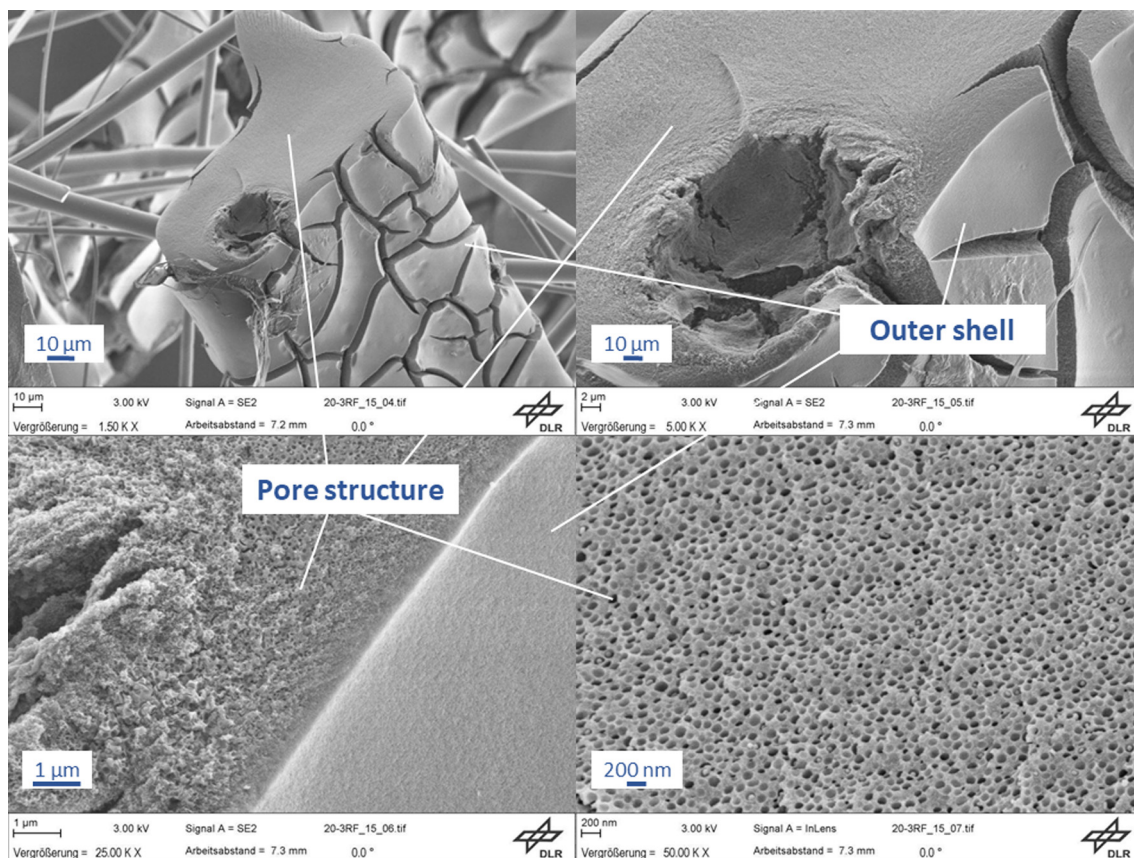


Fig. 15. Scanning electron microscopy image of a silica aerogel fiber sample, which was developed at the Institute for Textile Technology at RWTH Aachen University in cooperation with the German Aerospace Center; nanoporous structure below. Revised/reproduced with permission from Ref [184].

et al. [196] who performed finite element simulations to investigate the heat flow and thermal conductivity of aerogel embedded nonwoven thermal insulation materials. In computational predictions of actual thermal performance of aerogel-treated nonwoven fabrics, the ANSYS predicted conductivity values under laminar and turbulent flow were much closer to the experimental values at the selected sub-zero temperatures. Errors in experimental measurements arise due to structural and geometrical unevenness of the nonwoven samples [98]. There are also some larger scale simulations [197,198], but here the aerogel-textile composites are treated as a homogeneous material with no detail on the composite structure. In addition, some atomistic work has been done by Patil et al. [199]. Atomistic simulations are not well suited to give direct insight into the macroscopic properties as the maximum simulation size ($74 \times 74 \times 75 \text{ nm}^3$), but can give valuable contributions in the form of insights e.g. on the thermal conductivity of the bulk phase of silica and the phonon mean path within as well as the phonon transport / thermal resistance of the aerogel-fiber contacts.

8.2. Mechanical properties

Even less work has been done on modeling the mechanical properties of aerogel-textile composites. Yang et al. [67] used finite elements to simulate the creep in fiber reinforced silica aerogel using a power law to describe the creep deformation with coefficients determined from experiments. Although there was a good agreement, very few details are provided on the actual simulation.

Finally, some atomistic simulations have been performed by Patil et al. [69,200] representing silica-carbon and silica-silica nanocomposites (both graphite sheets and carbon nanotubes, even

silica glass nanorods). In follow-up work, Rege et al. [201] linked the properties from the atomistic scale to a continuum model for the elastic properties. While results were globally consistent between the atomistic and the continuum scale, validation with the experiment has not yet been achieved. This is all the more important due to the high strain rates (in the order of $1\text{e}9\text{--}1\text{e}8 \text{ s}^{-1}$) and small system sizes considered ($\leq 50 \times 50 \times 50 \text{ nm}^3$). Additionally, as mentioned above, the typical length scale of the nanocomposites studied is orders of magnitude below typical textile composites with fiber diameters of $0.1\text{--}50 \text{ }\mu\text{m}$. However atomistic methods can be useful to get insights into factors such as interface strength and fracture mechanisms.

9. Silica aerogel-textile composites in the market

9.1. History and future prediction

The invention of aerogels dates back to the late 1920 s early 1930 s [3] and the first commercialization activities started in 1939 by Monsanto [202], but this fascinating material showed little commercial uptake until the twenty-first century. Here, we could summarize the industrial history of aerogel materials in four R&D activity waves, shown in Fig. 16. The first wave started just after the appearance of aerogel in 1931, the only activity was launched by Monsanto with silica aerogel powder product with a brand name 'Santocel', and it was applied as additives to cosmetics, silicone rubber, and Napalm. The production process inherited Kistler's development of using sodium silicate and supercritical alcohol drying, which at the time was too cumbersome to reduce the material cost. Hence, this material production was stopped in the

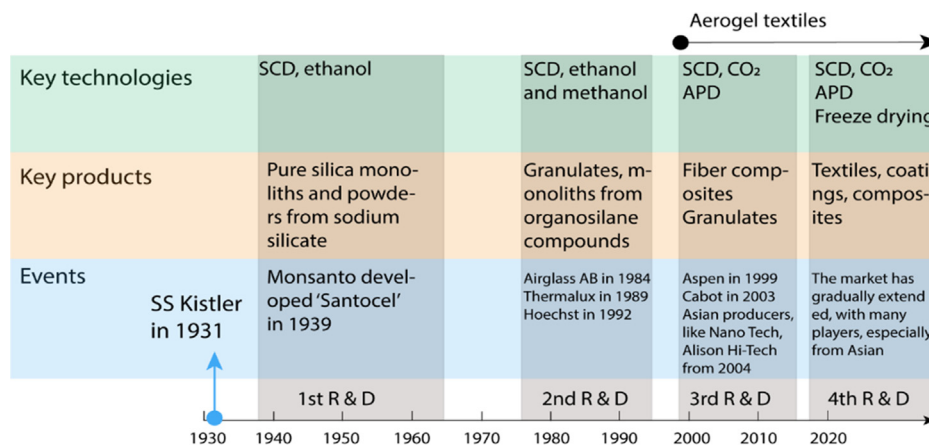


Fig. 16. The industrial development of silica aerogel materials [3,202,210–214].

1960 s. After the decay of stagnation, the second wave of R&D started in the late 1970 s, due to progress in CO₂ supercritical drying technique (Thermalux) in 1989, and ambient pressure drying (Hoechst) attempts in 1995 [19,203,204], even those techniques were not applied to industrial production of aerogels immediately, they did promote the take-off of the aerogel production from this period onwards. During this period, one main product was the monolithic silica aerogel developed by Airglass A.B., and it was used for Cherenkov detector.

The first textile-aerogel composites appeared in 1999, which it could be recognized as the start of the third R&D wave. Aspen Aerogels Inc. applied for several patents on fibrous batting aerogel composites (US patent 7,078,359 [205], 7,399,439 [206], and 7,780,890 [207]), while the textiles, mostly glass fiber, but also including polyester and silica fibers, were fully soaked into an aerogel precursor solution of organosilanes, and the gels were dried by supercritical CO₂. Another pioneer patent in the field of flexible aerogel nonwoven was filed by Hoechst AG [30], which Cabot Co. is the current assignee for it. The Cabot product was prepared by thermally bonding pre-formed aerogel particles between two layers of nonwoven fabric [131].

Since 2001, Aspen Aerogels Inc. blankets had been successfully applied to aerospace exploration, military, and petrochemical fields [52,91]. Following these successes, similar technology was also applied by NanoTech and Alison Hi-tech in China, and Enersens in France, but they propose that the aerogel composite could also be prepared by ambient pressure drying technique. In 2016, Aspen Aerogels Inc. filed legal complaints in the US and Germany about infringing its patents by Chinese producers [208]. Hence, the Chinese producers after this event, focus mostly on the domestic market.

Entering 2020, many new techniques and products have been developed in the field, and the market has also gradually extended with increasing players on the court, especially the growing market and manufacturers from Asia. They are looking for the development of new manufacturing approaches to secure the freedom to operate in the future. And this period, we could define it as the taking off of the fourth R&D boom in the aerogel field. The production and market entry of aerogel-fiber mats produced from pre-formed silica aerogel powder slurries by JIOS and Armacell are part of this wave.

An accurate evaluation of the aerogel market size is difficult because exact numbers are not available for many producers. From the IDTechEx prediction published in 2018, we could summarize the total market size for aerogel manufacturers in 2019 to be

\$223 million [209], and the current commercial aerogel industry is comprised of over 98 % silica-based materials by revenue, with over 85 % of the market for aerogel composite blankets in which a fibrous or textile reinforcement is applied during the gel preparation.

Aerogel composite blankets have found their greatest commercial successes in thermal insulation applications in the energy and industrial sectors, with a gradual increase in the other application fields, such as buildings, transportation, apparel, electronics, etc. Fig. 17 shows the current and forecasted breakdown of total aerogel market revenue (only silica was counted) by applications for 2019, 2024, and 2029.

9.2. Fiber types

The aerogel textile products on the current market include glass fiber, polyester fiber, polyethylene fiber, silica fiber, mineral fiber, and carbon fiber. The main products are listed below:

Glass fiber batting is the most used technical development by Aspen Aerogels Inc. It is normally used for compounding silica aerogel for high-temperature applications (greater than 400 °C) and with high fire resistance. A typical product is Aspen Aerogels Pyrogel XTE®, reinforced with a glass-fiber batting with an upper-use temperature of 650 °C.

Glass and silica fibers: The products are also from Aspen Aerogels Inc., the Pyrogel XTF® is claimed to include also silica fibers, the performance is close to Pyrogel XTE with a similar application temperature range.

Glass and polyester fibers: Spaceloft Subsea® is also a product from Aspen Aerogels Inc., it includes glass fiber and PET fiber, which leads to a lower thermal conductivity of 0.014 W m⁻¹K⁻¹, and lower use temperature.

Glass fiber batting reinforced silica aerogels are still the main products used for superinsulation, and the products are dominantly from Aspen Aerogels Inc., and supercritical CO₂ drying is the main technique (Fig. 18A). Similar products were also found in many aerogel producers, such as BASF Slentex®, NanoTech, Alison High-tech, and Enersens, but Asian manufacturers claimed that the textile aerogel bats could also be produced by ambient pressure drying (Fig. 18B).

Glass fiber reinforced aerogel mats could also be prepared by aerogel powder impregnation, a joint venture between JIOS and Armacell was established in 2016 [215]. Their product 'ArmaGel' comprises silica aerogel powder incorporated into an E-glass fiber mat. This textile aerogel composite was described with a similar

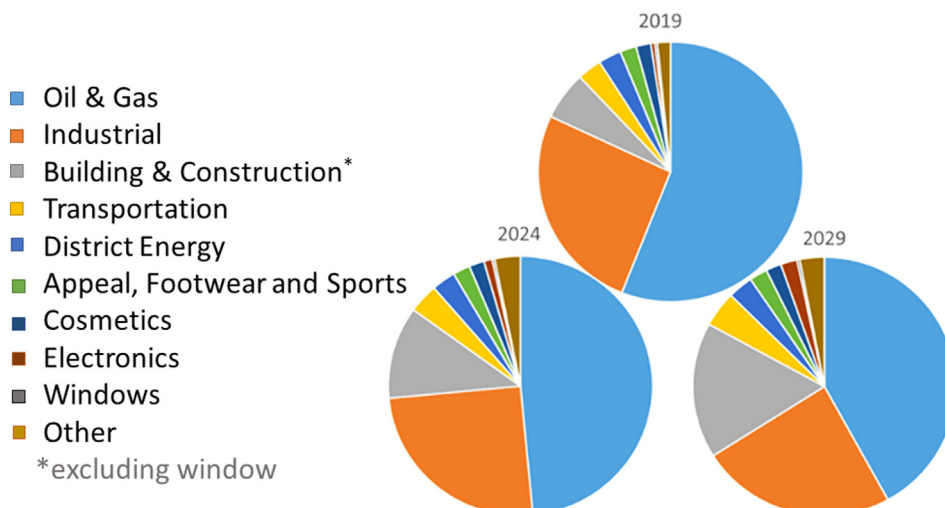


Fig. 17. Breakdown of the silica aerogel industry by end-user application (modified from IDTechEx research report). Revised/reproduced with permission from Ref [209].

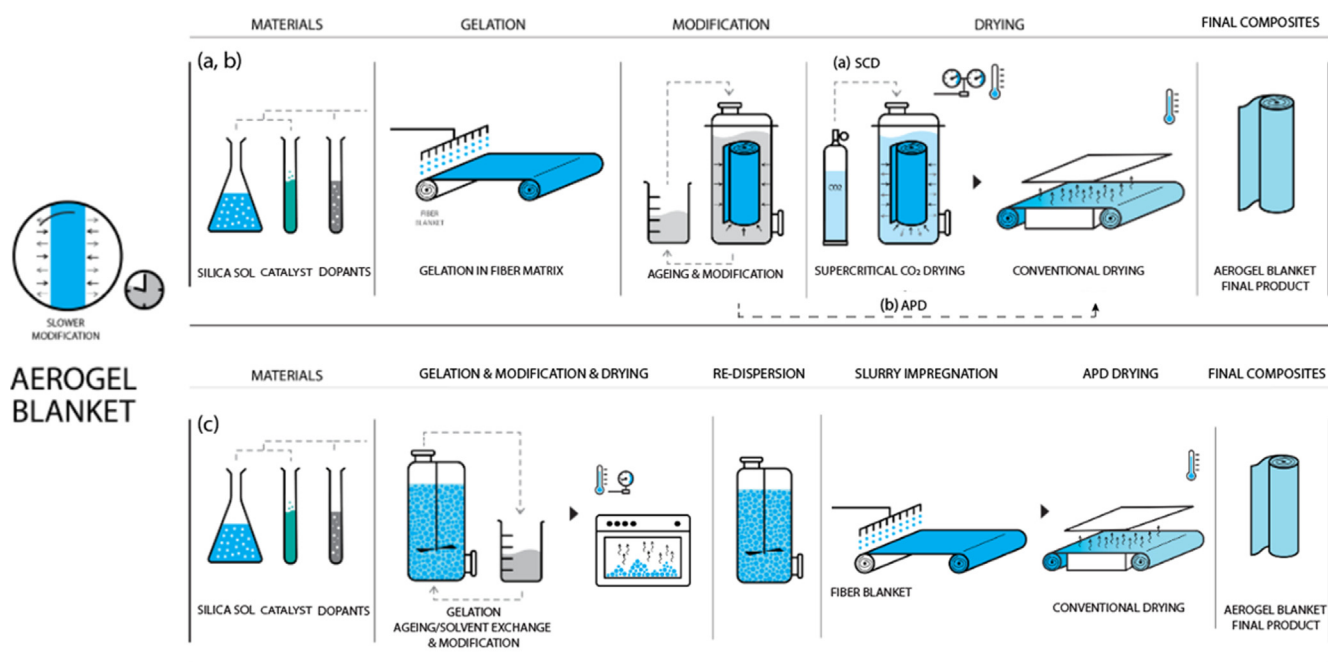


Fig. 18. The production processes for silica aerogel blankets. Revised/reproduced with permission from Ref [216].

operating temperature range as Aspen glass fiber batting (-200 to ~ 650 °C). But the production is under 2 times of ambient pressure drying processes.

Polyester/polyethylene fibers: One main product with polyester fiber is the Cabot Co. Thermal Wrap[®]. The production process of Thermal Wrap[®] is different from many inorganic fiber-reinforced silica aerogel blankets, which are produced by infiltrating silica sol into fibrous mats and drying the composite after the gelation of the composites. Thermal Wrap is produced by taking already dried silica aerogel particles and combining them with polymer fibers, the polymer glues the aerogel particles by heating or needle punching, resulting in a flexible composite blanket. Because of its polymer component, it is typically limited to lower application temperatures of -200 to 125 °C. But Thermal Wrap is easy to cut and handle and shows less dust during machining. Similar products could be found in Enersens Skogar[®], it was used recy-

cled PET fiber, but claimed with lower thermal conductivity of 0.016 W/(m.K) than Thermal Wrap[®].

Polyester and mineral fibers: Panasonic Corporation also has a thin aerogel blanket NASBIS [217,218], which is made by taking a non-woven sheet comprising two types of interwoven fibers, one is polymer fiber, and one is mineral fiber [219]. Since this product is very thin, NASBIS could be used for the thermal management of electronic devices.

Carbon fiber: Niagara LLC from Russian Federation specializes in the manufacturing of woven and non-woven substrates for composite aerogel materials, the products could be made carbon-fiber for high-temperature thermal insulation.

Except for silica-based textile composites, other solutions are also coming on the market. For example, the use of open-celled polymer foams, e.g. PU or melamine resins, instead of fiber matting can lead to composites with reduced dust release, e.g. PUREFLEX

from AGITEC AG. In addition, organic-based aerogel composites are also coming to the market. Aerogel Technologies LLC launched several polyimide aerogel composites with halogen-free fiber-reinforcement: Airloy HR116[®], and Airloy XR116[®] with an operating temperature of 300 °C. These new products are flexible, machinable, and mostly dust-free. The fire properties of these approaches using polymer foam matrices and/or polymer aerogels have to be considered.

9.3. Monitoring aerogel-textile composites after market introduction

Aerogel blankets in the market still suffer from a high dust release [57] or little resistance to compression [220], demanding more research works to be solved. For example, Bardy et al. [220] compared and analyzed samples of a prototype and a normal product-line aerogel insulating blanket from Aspen Aerogels Inc. for thermal conductivity and compressive strain at incremental pressure stops up to 1.2 MPa and showed that the compressive strain of the prototype aerogel blanket reached a level of 25 % whereas the product-line aerogel blanket compressed to 48 % at 1.2 MPa, leading to higher thermal conductivity and lower thermal resistance for the latter after compression [220]. In another study by Hoseini et al. in 2016 [191], “Cryogel Z” and “Thermal Wrap TM” aerogel blankets produced by two manufacturers, Aspen Aerogels Inc. and Cabot Co., were investigated for their thermal conductivity theoretically and experimentally, Fig. 19. Cryogel_Z contains PET/fibrous glass, while Thermal Wrap TM contains a PET core covered by a copolyolefin sheath. According to the modeling results, the highly porous structure, micropore sizes, and large surface

areas are the key features that make the aerogel blankets, including different fiber-aerogel combinations, an effective and promising insulating material with very low thermal conductivity for all types of enclosures, compared to many other traditional types of insulation materials over a wide temperature range. There are no fiber-to-fiber connections inside the aerogel-fiber composite matrix, according to the manufacturers, which would allow solid thermal conduction through aerogels [133]. This aerogel blanket structure mitigates the handling problems (mechanical strength) of aerogel powders while reducing the rate of heat transfer through the fibrous matrix. In addition, densely packed aerogel particles on the fibers suppress gas conduction heat transfer by reducing the voids between the fibers, resulting in higher thermal resistance achieved in both evacuated and non-evacuated systems [133,191]. When handled or cut, aerogel blankets may emit dust which is a recurring issue for almost all aerogel blanket manufacturers and can be problematic in the manufacturing process due to contamination of workspaces and surrounding equipment. Because the dust on the micrometer scale contains very few individual nanoparticles, the risk of exposure to nanoparticles is quite low from a health standpoint. On the other hand, silica is amorphous and therefore harmless to health, but the hydrophobic agent can cause skin dryness and inhalation problems [57].

One way to improve the non-dusting behavior of fiber-reinforced aerogels is to laminate the aerogel blankets, which can lead to good mechanical/thermal insulation properties, Fig. 20. In this context, the silica aerogel blanket Spaceloft[®] 3251 with dark gray color, hydrophobic/oleophilic properties, and thermal conductivity of 0.013 W/(m.K) from Aspen Aerogels Inc. laminated on both sides with a breathable membrane made of polyether block amide

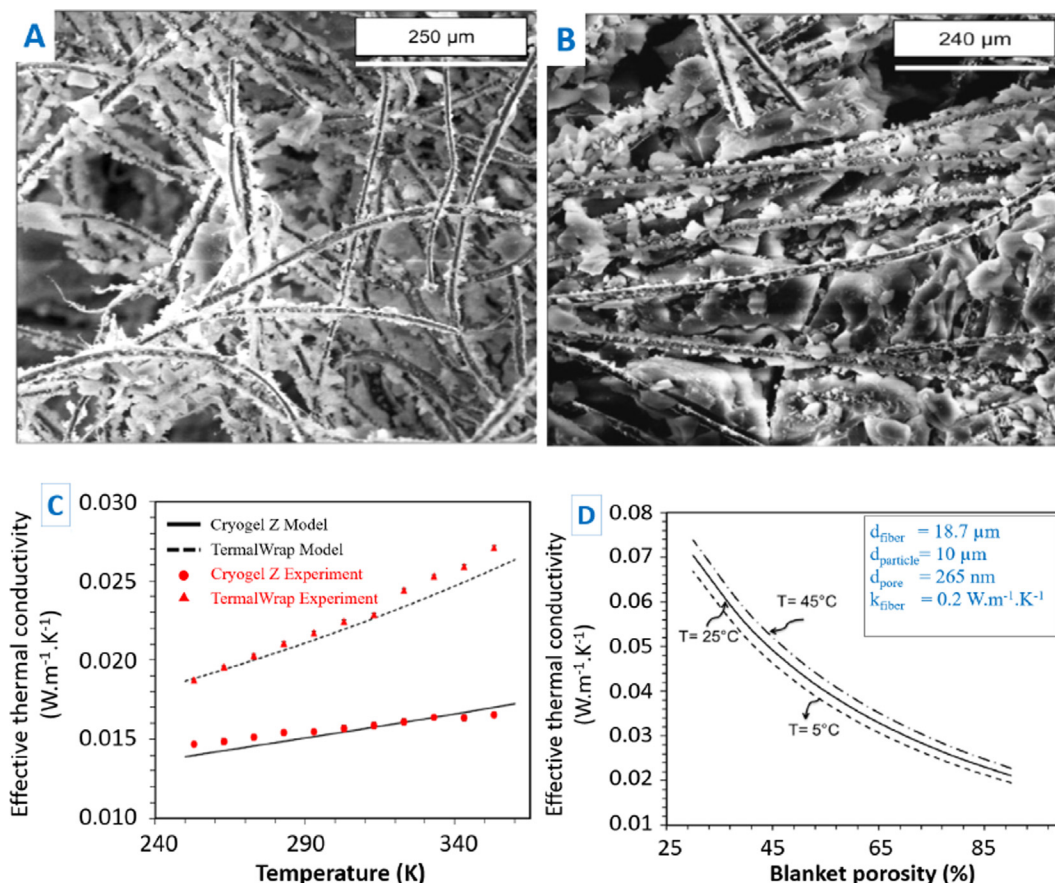


Fig. 19. SEM images of A) ThermalWrapTM aerogel blankets, and B) Cryogel Z, and thermal conductivity based on C) Temperature, and D) blanket porosity. Revised/reproduced with permission from Ref [191].

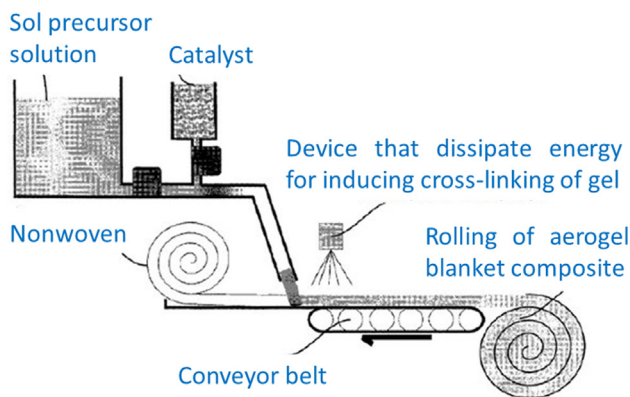


Fig. 20. Scheme of preparing a silica aerogel blanket. Revised/reproduced with permission from Ref [221].

and a fine knitted polyester fabric industrially by hot melt gravure bonding with a reactive polyurethane. The analyzed laminate is suitable for sleeping bags, protective covers, and seat covers of wheelchairs for winter conditions, etc.

To see if an aerogel blanket dried at ambient conditions can replace those dried with supercritical methods for building thermal insulation, Nocentini et al. in 2018 [57] investigated silica aerogel blankets reinforced with glass or PET nonwovens from Enersense company with a new method of ambient microwave drying. With a thermal conductivity of 0.015–0.020 W/(m.K), the studied blankets are in the range of industrial blankets e.g. Space loft from Aspen Aerogels Inc. and Aerorock ID from Rockwool, but not dried with cost-consuming supercritical method, which is a step forward in the aerogel blanket industrialization. Glass fiber aerogel blankets absorb more water (0.034 kg/kg) than PET fiber aerogel blankets (0.019 kg/kg) due to the presence of an organic binder in the layer, but high-water contact angle of 135–150° regardless of the type of fibers. There is also less cohesion between the silica aerogel matrix and glass fibers than with PET fibers resulting in more cracks during the drying process. Capillary condensation occurs for glass fiber aerogel blankets with 90 % humidity but not for PET fiber aerogel blankets. However, a significant improvement of up to twentyfold in compression properties happens with the incorporation of the glass fiber than native silica aerogel [57]. Finite element simulation of aerogel blankets including 70–90 vol% aerogel particles from Cabot Co. was investigated with Mishra et al. in 2021 [98] via COMSOL and ANSYS with <10 % error in the prediction analysis.

9.4. Service life

Although aerogel-enhanced materials have shown successful performance even in the market, the durability data under the effect of the various climatic aging processes is still unknown. This may become a barrier to the implementation of state-of-the-art materials such as aerogel granules [222]. Therefore, researchers started working on the laboratory accelerated aging of the aerogel materials to predict mostly the long-term consistency of aerogel materials' superior thermal performance, as their most probable application so far, as well as mechanical properties. Generally, it is not possible to simulate all the possible weathering conditions in a laboratory setup, and often, this might not be even necessary. In fact, laboratory tests considering only temperature, light, and moisture-related effects are often adopted for assessing long-term performance [223].

To address the durability of the silica aerogel granules themselves in a sealed system, simulating glazing windows, with 100–

300 aging cycles of warming up and cooling down, 10–20 years-natural outdoor condition performance based on environmental heat and moisture transfer was predicted. Aerogel granules can have ~10 % higher thermal conductivity after a moisture-aging test in worse conditions of seal deterioration [222]. Alvey et al. in 2017 evaluated mass and thermal conductivity change of market aerogel blankets by Aspen Aerogels Inc., Cabot Co., and Thermablock at 32.2–65.6 °C and 30–90 % and compared them with polystyrene and polyurethane foams [224].

In 2018, Berardi et al. [223] checked not only strong constant aging conditions of 70 °C and also, 70 % humidity, but also –30 to 40 °C freeze–thaw cycles for 170 days for aerogel blankets (Aspen Aerogels Inc.), aerogel plaster loaded with Cabot Co. aerogel granules, and aerogel boards (Aderma Locatelli Co). The results showed that high levels of relative humidity had the greatest impact on the thermal performance because of the residual moisture in the material exposed to such a laboratory condition. Also, a combination of different weathering effects, i.e. strong UV radiation alternated with high moisture and temperature levels, resulted in a greater increase in thermal conductivity than high humidity alone. The effect of other factors alone, e.g. high temperature or UV, was by far less significant. Finally, among all the assessed aerogel-enhanced products, blankets, and fiberboards showed superior performance both in nonaged (prestine) and long-term conditions [223].

In the other work with Chal et al. in 2018, Isogel and Isogel + SiC aerogel granules (Enersense, Cabot Co.), Spaceloft aerogel blankets (Aspen Aerogels Inc.), Lumira aerogel granules (Cabot Co.) in accelerated aging condition at 70 °C and humidity of 90 % for 96–384 days, the structural properties as well as thermal conductivity and water absorption were experimented. It can be concluded that commercial products show a wide variety of behaviors which is based on hydrophobicity and structural properties of silica aerogels as well as fiber types in case of blanket [225].

Besides climate conditions of use e.g. moisture, temperature, solar radiation, microorganism, etc., aerogel materials including completely OH group modified and hydrophobized silica aerogels show less influence by environmental moisture, while organic fibers e.g. PET fiber, compared to glass fibers, can suffer from earlier effect by moisture due to hydrolysis of ester groups. Also, in an extensive study by Lakatos [194,195], it was shown that the thermal conductivity of the aerogel blankets can be increased significantly (approximately 20–40 %) after wetting them. Heat absorption is increasing in function of the wetting time (moisture content), while the thermal inertia is almost constant.

10. Challenges and future trends

In the current aerogel market, silica aerogel-fiber composites is the most popular product (85 % of the current aerogel revenue). The main, and practically the only application is for thermal insulation. The current producers and distributors are widely distributed North America, Asia and Europe. Silica aerogel textile composites are considered to be the most promising consensus solution for future thermal superinsulation. Yet, compared with traditional insulation materials, such as mineral wool, expanded polystyrene (EPS) and polyurethane (PUR) foam, and inorganic porous materials such as perlite, etc., silica aerogel are still a niche in the market, with only 1.1–2.6 % of the global market share of thermal insulation materials from 2016 to 2020 [226]. In the existing fields, such as building and industrial insulations, there is no definite trend that silica aerogel products will completely take over the market shares from any existing insulation materials in a short period. To the authors' knowledge, the main challenges are listed as follows.

- (1) Over-advertised properties. Once the silica aerogel was developed by Kistler, this material has been dubbed the lightest material in the world, the reality is the current world record lightest solid material is graphene aerogel. Practically relevant silica aerogel has never been the lightest solid, most of the large pore foams, such as cellulose, PU, and even cotton are much lighter than the silica aerogel applied for thermal insulation. Not to mention that in the silica aerogel fiber composites, the reinforced fibers are normally dense, which increases the overall density of the composite aerogels. However, for insulation applications, too light is also not good, the large pore size could increase the gas conduction. And the thermal conductivity of the materials follows the U shape curves, the lowest thermal conductivity value is always at the medium density [39]. This value for silica aerogel is around 0.10–0.15 g/cm³. At this density, the thermal conductivity is indeed extraordinarily low, but for many investors that are attracted by exaggerated claims of record-breaking low densities, such inaccurate propaganda leads to disappointments and negative effects on future commercialization.
- (2) High production costs. The price of the current silica aerogel products is 400–5000 USD/m³. In comparison, the prices of EPS and mineral wool with a low λ of around 35–45 mW/(m·K) are only 25–180 USD/m³ and 40–350 USD/m³ respectively. The price difference is at least 10 times and remains to a large extent, even when the higher performance (hence lower material consumption) is taken into account. Hence, the current low market volume is to be expected. In the current aerogel industry, cost reduction is definitely the first challenge. New blankets produced from pre-formed silica aerogel powder have been introduced in the market: whilst this approach has the potential to strongly reduce cost, it remains to be seen how close such products can get to those prepared by sol impregnation in terms of performance (thermal conductivity and dust release).
- (3) Poor mechanical strength caused by the fragile structure. Due to the highly porous structure and necklace-like particle-to-particle connections, the pristine silica aerogel is particularly fragile with almost no tensile elongation (tensile stress < 16 kPa), low compressive stress of < 3 kPa, low bending strength of < 20 kPa, and Young's modulus of 1–10 MPa [57]. Although the incorporation of fibers in the silica aerogel improves its integrity and mechanical properties, the fragile nature of aerogel has not been changed. The aerogel incorporated into the fiber matrix is easily broken into fragments if an external force is applied to squeeze, bend or cut the composites. Hence, in most cases, the aerogel textile products are silica aerogel particle-filled fiber matrices that are prone to loss of aerogel particles. As a result, the performance of the silica aerogel products could be severely influenced and the released small aerogel particles also bring health-related concerns. Polymer cross-linking is an efficient strategy to reinforce silica aerogels by forming a conformal polymer coating on the silica structure [227]. However, it is still challenging to fully retain the outstanding properties of the silica aerogels, such as flammability, thermal insulation, transparency, and density [228,229]. Thus, the integrity of silica aerogel products should be further improved through more advanced and more mature techniques.
- (4) Health-related issues. As the silica aerogels have poor mechanical strength, the small silica particles can be very easily released during the handling or machining of the aerogel composites. The increased concerns about the parti-

cles released from aerogel products have been raised in several countries. The released particles from the aerogel products can range from nanometer to centimeter-scale [230]. In the past, the toxicity risk assessment of silica aerogel was reported as nontoxic and did not represent health risks as its main component, i.e., amorphous silica, has long been regarded as much less hazardous than crystalline silica [231]. However, breathing amorphous silica can still cause respiratory diseases (but no silicosis), lung inflammation, and injury [232]. Skin itch may occur upon touching many aerogel products. A recent study from Pavan C. [233] shows that the occurrence of nearly free silanols (NFS) on the surface of both fractured quartz and amorphous silica particles may activate the toxicity of silica, thus revisiting the established paradigm whereby crystallinity is assumed to be critical for silica toxicity. Therefore, regardless of the source and preparation methods, silica particles with a high concentration of nearly free silanols may damage cellular membranes and initiate inflammatory reactions. Hence, it is required to find the appropriate strategy to reduce the dustiness and probably proper inspection (size, toxicity) on the released dust particles from the aerogel products of concern [231].

Even though there are several challenges, silica aerogel fiber composites remain the go-to solution when extreme thermal insulation performance is required. Its ultra-low thermal conductivity is still attractive in many application scenarios, for instance, due to the fast development of electric vehicles, battery safety starts to be critical, and the silica aerogel fiber composite thermal barriers seem to be the first product considered. And other new emerging markets, such as 5G communications, unmanned aerial vehicles, and new designs of aviation, could bring great business opportunities for this material. With the increase of the market size, the completion of the corresponding product/application standards, and the participation of more market players and continuous development of the products, the above-mentioned challenges will be only a period of labor pains like all mature products and technologies currently in the market.

Compared to the now standard fiber-reinforced silica aerogel blankets, the field of aerogel fibers is still at its infancy. Many academic groups are targeting the production of aerogel or aerogel-filled fibers by wet-spinning, melt-spinning and other approaches. Based on the strong preliminary results in the field, we foresee that high quality aerogel and aerogel-filled fibers will become available, also commercially, provided that a suitable application can be found that valorizes the unique selling points of such fibers.

11. Concluding remarks and outlook

Silica aerogels as unique porous materials with very large surface area and high mesoporosity have found increased attention in the various fields of applications e.g. thermal and sound insulation, but the silica aerogels generally are highly brittle. The mechanical properties can be improved effectively with the introduction of fibrous materials into the silica aerogels making aerogel-textiles composite materials. The silica aerogel composites reinforced with fibers normally benefit from higher mechanical properties, but it is imperative to keep their thermal conductivity as low, and their porosity as high as possible. The addition of the textile to aerogel does not significantly increase the overall thermal conductivity of composite since many textiles have low solid conduction, especially low-density, aligned nonwovens. Regarding mechanical properties, the density of the aerogel, its composition, the type of fiber used, and the orientation of the fibers within the

layer are all effective on the bending and compression properties with a wide range of data on moduli and strengths. However, the reason why blankets are the most popular aerogel product on the market is not because of their better compressive properties, but because they are simpler to handle than monoliths or granular products, less dusty, and less fragile due to the aerogels protection within the matrix of fibers. Aerogel-fiber composites are thus preferred to aerogels alone from this perspective. Possible effects on hydrophobicity and fire properties also need to be considered. Hence, the materials selection and the method used for the integration of the aerogel with textiles, including solvents, binders, and other additives, needs to be considered carefully to address the above properties.

Review studies in this area have mainly focused on the application of aerogel/textile systems [28,46–50] or on textile impregnation in silica-based sol using the sol gel approach [45,46]. Here, we reviewed the current state-of-the-art of literature to look at all the different ways in which silica aerogels are combined with fibers and textiles, whether through sol-gel synthesis, embedding on fiber surfaces, or incorporating into polymeric matrices, as shown in Fig. 4, along with their advantages and disadvantages, applications, their market readiness, and future prospects.

We described the important possible ways to have hybrid composite materials of aerogel and textiles, including forming aerogel blankets by introducing silica aerogel to the fibers via sol-gel process (section 2), thermal bonding and pressing, or mixing the aerogel particles with a solvent and injecting into the fiber structures (section 3), aerogel-filled material coated textiles (section 4), aerogel-filled fibers (section 5), silica aerogel packages (section 6), and aerogel fibers (section 7). We concluded this review with a detailed look into the modeling (section 8), and current and future market developments of silica aerogel-fiber composites (section 9).

A majority of works on the aerogel/textiles addressed impregnation of fiber material into the aerogel sol and synthesizing the aerogels on the fiber surface, as the main route of the aerogel textiles composites in terms of academic research and industrial points of view, by which the aerogels keep their properties mostly, but suffering from releasing from the fiber surfaces as dust. The fibers are mostly inorganic, mostly glass and ceramic fibers, and the composites improve mechanical properties while keeping low thermal conductivity. In terms of organic fibers, e.g. aramid, carbon, polyester, and polyolefin, were used in studies that concentrate on thermal/mechanical properties, hydrophobicity, fire resistivity, and absorption and filtration properties. While high aerogel contents lead to low thermal conductivity, in fiber immersed aerogel compositions (aerogel content 50–95 wt%) and aerogel packages, dusting and selected membrane fabric/method to sew package are the most problematic issues with the former and later methods, respectively. Although limited data are available, aerogel-filled material coated fibers extremely reduce dusting problems, but aerogel content mostly is below 10 wt%, leading to very different properties. Here, systematic efforts are needed to fully understand and reduce the penetration of polymer into aerogel pores which vanish aerogel porous structure as remaining or collapse the porosity by removing the polymer or solvents from the pores. Research where the focus is on aerogel with the fiber itself, as a filler or as the only fiber component, rather than in between the fibers, is at an early stage and most work focuses on organic, rather than inorganic aerogels. Using aerogel particles as a filler in fiber production can lead to a surface roughness for aerogel-filled fibers in comparison with the smooth surface of aerogel-free fibers, and is effective for tailoring hydrophobic/hydrophilic properties.

Data availability

Regarding a review paper, the data were gathered mostly from previously published papers.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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