



Formation and thermo-physical properties of aerogel ceramic blanket composites synthesized via scalable atmospheric pressure process with methyltrimethoxysilane precursor

Abdullah Tav^{1,2} · Yahya Oz² · Halil I. Akyildiz^{3,4}

Accepted: 16 September 2023 / Published online: 28 September 2023

© The Author(s), under exclusive licence to Springer Science+Business Media, LLC, part of Springer Nature 2023

Abstract

The fragility of silica-based aerogels limits their potential use in various applications. These exceptional materials can be immobilized on more flexible insulation materials to form materials called ‘aerogel blankets’. In situ, sol-gel deposition is one way to synthesize aerogel material directly on the fibrous matrix. This study presents aerogel blankets’ formation and thermophysical performance obtained by in situ deposition of a methyltrimethoxysilane (MTMS) precursor prepared with sodium dodecyl sulphate surfactants. X-ray diffraction analysis indicates increased peak intensities, suggesting a more organized crystalline-like structure. Corresponding Raman spectroscopy and transmission electron microscopy (TEM) observations corroborate the results, highlighting the surfactant’s role in promoting structural order within the aerogel. A distinct silica spherical structure was also observed in the TEM analysis. The process was carried out at ambient pressure conditions; thus, the process is scalable for large-volume production. The MTMS concentration of the sol varied in the range of 5 to 30 wt.%, resulting in different aerogel characteristics deposited onto the ceramic fiber matrix. Samples were characterized mainly for their thermal, mechanical, and morphological properties. Remarkably, the thermal conductivity performance of the obtained composite blanket showed 30% lower heat conductivity than the pristine ceramic blanket.

Keywords Silica aerogels · Ceramic blanket · Sol-gel processes · Thermal decomposition

1 Introduction

Aerogels were first discovered a century ago [1]; however, their commercialization has started in the last two decades [2]. They are used in applications such as aerospace systems [3], building materials [4], catalysts [5], absorption media [6], and composites [7–9]. They are divided into organic and inorganic classes [10], while inorganic silica aerogels

are the most widely studied group. Silica aerogels (SiO_2) are unique materials with ultra-low density and highly cross-linked structures with high porosity, where pore sizes vary by a few nanometers [11, 12]. These structures constitute air more than 90% of their total volume resulting in very low densities ranging from 70 kg/m^3 to 150 kg/m^3 depending on the source materials used in the production [13]. The most common silica precursors for silica-based aerogel are alkoxysilanes; tetramethyl orthosilicate (TMOS), tetraethyl orthosilicate (TEOS), and water glass. They are synthesized with sol-gel chemistry [11], while their processing can be scaled from laboratory to industrial applications. However, many –OH groups are present on surfaces of materials synthesized from alkoxysilane derivatives causing silica aerogels to be highly vulnerable to moisture, thus affecting their thermal and mechanical properties [3, 14, 15].

Recently, there has been a growing interest in methyltrimethoxysilane (MTMS) alkoxysilane precursors. Working with MTMS shortens the process, provides advantages in the drying step, and does not require hydrophobization [16, 17]. The chemical structure of the aerogels formulated with

✉ Halil I. Akyildiz
halilakyildiz@uludag.edu.tr

¹ Department of Polymeric Materials, Bursa Uludag University, 16059 Bursa, Turkey

² Turkish Aerospace, R&D and Technology Directorate, Advanced Composite Materials Technology Center, 06980 Ankara, Turkey

³ Department of Textile Engineering, Bursa Uludag University, 16059 Bursa, Turkey

⁴ UNAM-National Nanotechnology Center, Bilkent University, 06800 Ankara, Turkey

MTMS is resistant to capillary forces that occur during drying due to the ($-\text{CH}_3$) functional groups forming instead of $-\text{OH}$ groups. The obtained material is labeled as methylsilsesquioxane (MSQ) aerogel at the end of the process with the MTMS precursor, according to the literature [12]. The hydrophobicity of MSQ aerogels is important since capillary forces are the reason for the need for supercritical drying which requires high pressure and high temperatures; thus, it is conducted as a batch process. For high throughput production of aerogels, ambient drying condition processing is needed. Remarkably, the MTMS precursor can be a good candidate for the large-scale production of aerogel materials [18–20].

Hydrophobic MSQ-derived aerogels are not ready for large-scale production due to their lack of desired mechanical properties. As a solution to the mechanical shortcomings, aerogels are integrated into fibrous mats to form insulation composites called aerogel blankets [18, 21]. This approach led to the commercialization of aerogels for the commodity market. There are various ways for this integration/immobilization process. One of the approaches synthesizes aerogel pieces in a conventional sol-gel approach. Afterward, powders of aerogel are applied to the blanket material with the help of a binder. The second approach synthesizes the aerogel material within the fiber matrix, labeled as in situ sol-gel deposition. A fiber mat is dipped into a sol of desired precursors while the remaining sol on the fibers undergoes gelation reactions. Hence, gel materials are formed directly on the fiber surfaces. The process is followed by a drying step conducted at supercritical or ambient conditions [22]. An in situ MSQ aerogel blanket synthesis involves directly synthesizing the material within its intended application. Alkoxide groups in MTMS undergo hydrolysis leading to silanol (Si-OH) group generations. These silanol groups subsequently experience polycondensation reactions, where they react with each other to form Si-O-Si bonds. This condensation process progresses, facilitating the formation of a three-dimensional network of MSQ. The resulting MSQ material exhibits a hybrid organic-inorganic structure comprising organic methyl groups and inorganic siloxane (Si-O-Si) bonds [12]. MSQ can be incorporated into composite blankets by in situ synthesis of the MTMS precursor sol within a matrix material, such as a ceramic blanket, through the sol-gel process. Subsequent processing steps are followed by MSQ embedded within the matrix. Remarkably, several scientific studies have extensively investigated and characterized the sol-gel process and integration of MSQ into composite blankets [23–25].

Surfactants (anions or cations) can be added to the sol-gel process to improve dispersion while aerogel was synthesized [26], promoting surface adhesion on fibrous matrices by changing the geometry of the microstructure of silica. Surfactants also affect the shape of silica aggregates, causing an

electrophilic addition mechanism, which leads to branched and cross-linked forms of aggregates. Li et al. used silica aerogel to obtain the silica precursor methyltriethoxysilane (MTES) with the cationic surfactant cetrimonium bromide (CTAB). A two-step sol-gel process and CO_2 supercritical drying were used to prepare flexible hydrophobic aerogels. With the CTAB used at the optimum rate, the gel skeleton structure gradually changed from different spheroids to continuous spheroids structures [26]. Ramaswamy et al. used CTAB to prepare an MTMS silica precursor in an aqueous medium to increase its dispersibility after dip-coating on kapok fibers with MSQ alcogel. The composite material they obtained shows many promising results corresponding to low density (0.053 g/cm^3) and effective thermal conductivity (0.018 W/mK) [27]. The presence of anionic surfactants affects silica species' aggregational behavior, enabling the network's microstructural arrangement [28]. Sodium dodecyl sulphate (SDS) is an anionic surfactant demonstrating the fundamental aggregation phenomenon between MSQ-derived silica particles [28]. However, no report in the literature discusses composites (matrices and aerogels) utilized by the sol-gel method leading to aerogel blankets (about chemical, morphological, thermal, and mechanical properties), where MTMS and SDS are used as precursor and surfactant, respectively.

Studies have shown that silica aerogels could be immobilized with dispersed fibers, nonwoven matrix, and epoxy to obtain highly durable composite materials. Various fibers, such as glass, ceramic, and carbon, are used for high strength and ultra-low thermal conductivity [29]. Major companies such as BASF-Slentex™, Aspen Aerogel-Pyrogel™, and Cabot Corp-Wrap™ produce aerogel blankets commercially for thermal insulation. Superior insulation properties resulting from the nanoporous structure include very low thermal conductivity values measured in solid, gas, and infrared environments [27]. The selection of insulating materials to be used in engine parts in the aerospace industry is critical. Thermal management and the possibility of being used in high-temperature areas of aerospace platforms are appealing [30]. Note that in high-temperature applications, ceramic blankets outperform fiberglass insulation. Ceramic fibers can withstand temperatures up to 1800°C without degrading or losing their insulating properties, making them ideal for aerospace apparatus. Excellent thermal stability is exhibited by ceramic blankets, allowing insulation performance to be maintained despite rapid temperature changes. Furthermore, industries exposed to corrosive substances or harsh chemicals find ceramic blankets well-suited due to their superior resistance to fiberglass [31]. Additionally, higher mechanical strength and durability are possessed by ceramic blankets, making them suitable for applications involving mechanical stress. Unlike fiberglass, ceramic blankets do not suffer sizing-related issues, where binders or coatings on fibers

can degrade or release toxic gases at high temperatures. Moreover, excellent fire resistance is exhibited by ceramic blankets, as they are not readily prone to burning or releasing toxic gases when exposed to flames. Overall, fiberglass insulation is outperformed by ceramic blankets in high-temperature environments and applications involving corrosive substances or mechanical stress while providing advantages such as the absence of sizing-related concerns. Ceramic blankets can effectively maintain their insulating properties even in extreme temperatures without the risk of toxic gas emissions or reduced insulation effectiveness. Note that these advantages are of interest for aerospace applications which combine a number of requirements to be fulfilled. For instance, these standards (EASA CS-23 and CS-29, RTCA DO-160G, MIL-STD-1629 A, E-03-CRI-P) of the varying authorities involve thermal conductivities below 0.15 W/mK at room temperature, thermal stability for at least 1176 °C, resistance against humidity as well as salt fog, waterproofness, absence of any emissions which harmful, toxic or hazardous and a color which allows to detect any leakages.

Multifunctional properties of aerogels with an MTMS precursor were studied for high thermal stability and low simultaneous thermal conductivity, diffusivity, hydrophobicity, and flexibility. An insulation blanket doped with an MTMS precursor (5–30 wt%) via a two-step sol-gel process was prepared. After the aging and solvent exchange, alcogel blankets were ambient pressure dried (APD). Samples were characterized with regard to bulk densities, specific surface, thermal conductivity (hot disk transient plane source (TPS)), and microstructure. Furthermore, mechanical properties were studied using the tensile stress test and three-point bending method. The hydrophobic character was also analyzed using water contact angle tests. Moreover, thermogravimetric analysis (TGA) was used to determine weight loss characteristics. Finally, the air permeability test of samples was carried out with different weight percentages in blankets.

2 Material method

2.1 Materials

Trimethoxymethylsilane (MTMS, 98%, CAS# 1185-55-3), sodium dodecyl sulphate (SDS, 99%, CAS# 151-21-3), hydrochloric acid (HCl, 37%, CAS#7647-01-0), Ammonia solution (NH₄OH 25%, CAS# 1336-21-6), and n-Hexane (CAS# 110-54-3) were purchased from Sigma Aldrich. Absolute ethanol (EtOH, 99.9%) was purchased from Isolab Chemicals. Deionized water (DI) was used throughout the experiments. All chemicals were used as received without further processing. Ceramic insulation blankets were purchased from a local vendor with an average fiber diameter

of around 4.5 µm and without sizing. Before processing and testing, ceramic insulation blankets were conditioned by placing samples in a room at 25 °C and 65% relative humidity for 24 h.

2.2 Synthesis of composite aerogels

MSQ aerogel was synthesized via a two-step acid-base sol-gel synthesis, after which they were aged and ambient pressure dried. First, the anionic surfactant SDS (the final dry concentration was calculated as 0.2 wt%) was dissolved in an ethanol (EtOH)/water mixture where the relevant ratio was kept constant for all samples. The weight ratio of EtOH/water in the sol was applied as 2:1 for all samples. The anionic surfactant was stirred vigorously for 30 min until a clear solution was formed. Subsequently, the silica sol was prepared via MTMS hydrolysis in EtOH and water with weight ratios of 5, 10, 15, 20, 25, and 30 wt%. The hydrolysis was initiated under acidic conditions by the dropwise addition of an acidic catalyst, 0.005 M HCl, to adjust pH 2. After mixing for 4 h, the base catalyst, 0.02 M NH₄OH, was added dropwise into the sol, and the pH was increased to 8. Then, the sol was stirred further for 5 min. Our previous work explained this study's precursor and solvent ratios in detail [32]. Immediately, ceramic insulation blankets (9 × 15 cm², thickness 2 cm) were dipped and immersed into the sol for 10 min to infiltrate MSQ alcogels into the ceramic blanket matrix. The whole gel development took about 6 h. Each sample was processed and repeated separately, and MSQ alcogel blankets were prepared on a ceramic blanket with 5 to 30 wt%. Afterward, alcogel-containing samples were kept in a container of ethanol and replenished with fresh ethanol three times a week. After the aging, ethanol was replaced with n-hexane transferred from a different container. Ethanol in the alcogel was substituted with n-hexane three times within a week. Unreacted precursors, surfactants, water, and ethanol were removed from the environment by continuously immersing hexane exchange. Regarding the final drying stage, two-stage temperature and drying time are conducted. In the first stage, samples were dried at ambient pressure and kept at 75 °C for 8 h. The MSQ aerogel blanket was designed as a multi-layered structure of relatively thick material, which may have the potential for entrapment of by-products corresponding to methanol, ethanol, water, and unpurified materials within its inner layers. The main objective of the aging process is to eliminate these impurities, while its efficiency may vary. Because of the presence of these by-products, polar-type materials can often lead to the deformation of the wet gel during drying due to increased capillary forces. Unfortunately, some by-products can't be effectively discharged via solvent exchange during aging. The oven was kept at 75 °C for 8 h to sustain the aerogel's structural integrity. Subsequently, the next included the

adjustment of the oven to 150 °C for 4 h, eliminating any remaining by-products trapped within the ceramic blanket's inner sections to obtain the samples. The scientific process of synthesizing silica aerogel and its corresponding physical appearance is depicted in a flowchart in Fig. 1. This diagram outlines the sequential steps involved in the synthesis procedure, highlighting critical reactions, parameters, and equipment utilized to produce silica aerogel and aerogel ceramic blankets. Additionally, the flowchart visually presents physical attributes of the resulting aerogel material and aerogel ceramic blankets' nature and distinct structural properties.

2.3 Characterization of composite aerogels

A Quanta 400 F field emission scanning electron microscopy (FESEM) device was used for the morphological analysis of the samples. Before examining the microstructure, the samples were coated with a 15 nm Au/Pd thin film with sputtering. Corresponding micrographs were recorded operating under a high vacuum at 20 kV potential while recording with a secondary electron detector. Elemental analysis was conducted by energy-dispersive X-ray spectroscopy (EDS) attached to the SEM. TEM analyses were performed using the FEI TALOS F200S TEM operating at 200 kV. Samples were prepared using the solution-drop method. A methanol solution containing a small quantity of MSQ and MSQ + SDS aerogel powder was sonicated for 10 min. A droplet of the dispersion was deposited onto a standard holey carbon-coated copper grid. Sorption isotherm analysis was characterized using nitrogen (N₂), while the pore structure of the samples was determined using Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) methods. The Perkin Elmer Spectrum Two spectrometer took Fourier-transform infrared (FTIR) spectra of the MSQ aerogel, pristine ceramic blankets and aerogel insulation blankets. Absorption spectra of the samples in the wavelength range of 4000–500 cm⁻¹ were recorded with an Attenuated total reflectance (ATR) configuration; in addition, aerogels and

aerogel blankets measurements were conducted at room temperature with a spectral resolution of 4 cm⁻¹ and 16 scans for accuracy. The Raman device used in this study was the FRA 106/S, equipped with a 532 nm laser for excitation and designed for single-point analysis at lower frequencies. Raman spectra were collected in the wavenumber range from 3500 to 100 cm⁻¹, providing comprehensive insights into the silica aerogels' molecular structure and vibrational modes. The crystallinity assessment of all samples was carried out utilizing the Rigaku-Ultima IV XRD system, involving scanning of diffraction peaks across the 1–80° degree range with a scan speed of 1°/min. The sample's bulk density (ρ_b) was obtained from the weight measurement (in grams) using a microbalance with an accuracy of 10⁻⁴ g for a known volume. The skeletal density of MSQ aerogel and MSQ aerogel blankets (ρ_s) were determined with the measured porosity using a helium pycnometer (Cryogenic Limited PPMS) and using the formula [27];

$$\text{Porosity (\%)} = \left(1 - \frac{\rho_b}{\rho_s}\right) \cdot 100.$$

Mechanical strength and flexibility were characterized by pristine and composite aerogel blanket samples of 90 mm in length, 15 mm in width, and 20 mm in thickness. Tensile tests of pristine and composite aerogel blankets were carried out according to the ISO 4606 standard in a climatically controlled room (65% relative humidity (RH), 25 °C). A three-point bending test was based on the ASTM D 790–17 testing procedure, while measurements were performed with a loading nose descent rate of 20 mm/min. Both mechanical tests were done using a Shimadzu universal testing machine with a load cell of 1 kN. The thermal stability of samples was determined via TGA from ambient temperature to 850 °C with a heating rate of 10 °C/min, using a Perkin Elmer Pyris 1 under nitrogen flow corresponding to the literature [33, 34]. During the TGA-FTIR analysis, the thermal degradation steps of all samples were examined by TGA from ambient

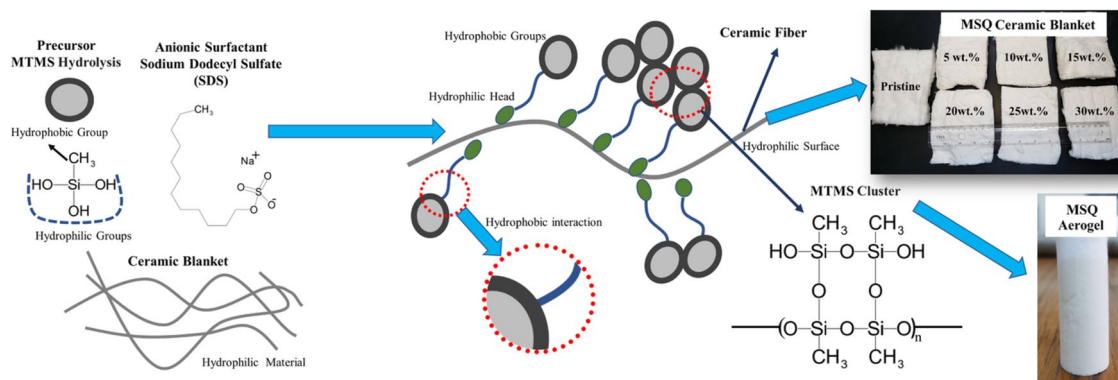


Fig. 1 Process of synthesizing MSQ aerogel and aerogel blankets with their corresponding physical appearance

temperature to 850 °C with a heating rate of 10 °C/min, coupled with FTIR with the Perkin Elmer Pyris STA 600 device. For FTIR detection, evolved gaseous products went through stainless-steel tubing into the gas cell under nitrogen carrier gas. Corresponding spectra were recorded in the range of 4000–500 cm^{-1} . Thermal conductivity measurements were performed employing a TPS thermal constant analyzer via the Hot Disk TPS 2500 S. The TPS device uses ‘hot disks’, i.e., two-sided sensor types and software package modules, to conduct measurements on bulk materials (isotropic and anisotropic), thin films, powders, and liquids. In this work, Hot Disk Kapton 5501™ (polyimide) sensors with a radius of 6.403 mm were used for simultaneous transient heating of the samples. The measurement was performed where at least two identical materials with a thickness of at least 2 cm were placed simultaneously in the device. Before the analysis was conducted in the range of 30–199 °C, the temperature coefficient of the resistivity of the sensor (TCR) was set to the sensor/power supply [35, 36]. It should be noted that both sides of the samples have been measured separately, while the average value has been reported for precision.

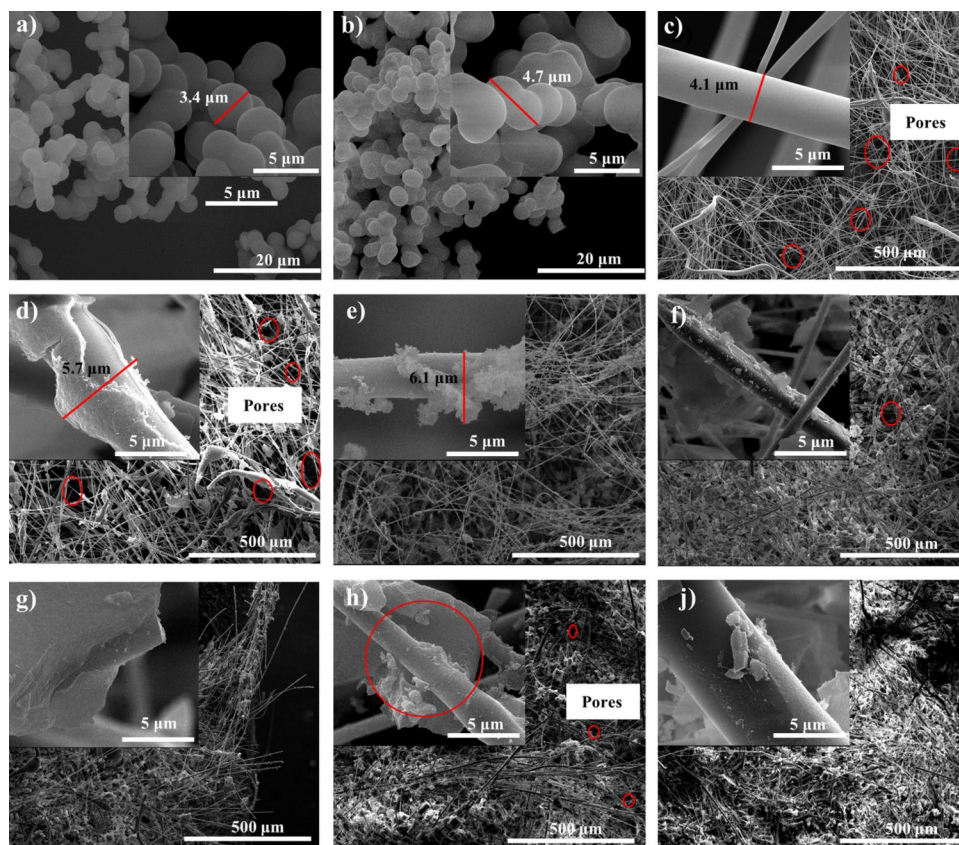
Air permeability of the samples was displayed using Atlas MO21A-SDL according to ISO 9073-15:2007 in a climatically controlled room (65% RH, 25 °C). The test resembles an airflow passing rate ($\text{L}/\text{m}^2/\text{s}$) vertically through the surface of 5 cm^2 by measuring the pressure increase (Pa)

through the samples. Pristine and composite aerogel blankets were used to compare the permeability of the coatings. Water contact angle measurements on samples were carried out using the Attension Theta lite device by placing 5 μL water droplets on the samples’ surfaces.

3 Results and discussion

Figure 2 presents SEM images of MSQ aerogels and blanket composites. The aerogel sample in Fig. 2a was synthesized without using a surfactant in the recipe. It can be observed that the colloidal network of the material is formed with mostly spherical silica particles. Using a surfactant has changed the morphological structure of the material, as shown in Fig. 2b. Spherical silica particles from the sample with a surfactant are intertwined, and the structure becomes much better stacked. This morphological change is attributed to increased electrostatic and hydrophobic interactions with the addition of SDS [37]. SDS has a hydrophobic tail and a hydrophilic head, resulting in lower surface tension and better suitability for composite preparation. The presence of SDS negatively affected capillary stress under APD conditions. Due to these conditions, the electronegative interaction of SDS with aerogel was expected to have much more capillary force [37], but the obtained structure

Fig. 2 SEM images obtained for **a** MSQ and **b** MSQ with SDS aerogels, **c** pristine, **d** 5 wt%, **e** 10 wt%, **f** 15 wt%, **g** 20 wt%, **h** 25 wt%, **j** 30 wt% MSQ aerogel coated ceramic blankets



was much more homogeneous. Additionally, SDS stabilizes the sol by reducing surface tension, influencing the gelation process as it acts as a structure-directing agent, allowing a controlled pore structure and morphology by forming templating structures. Remarkably, the presence of SDS modifies the surface properties of resulting aerogels, with negative charges imparted to the surface, influencing surface charge and interactions with other substances. This alteration affects wettability, hydrophobicity/hydrophilicity, and surface reactivity. In addition, SDS demonstrates compatibility with the MTMS precursor and sol-gel process, enabling its effective incorporation into the precursor solution. Thus, advantages of using SDS in MSQ-based aerogel synthesis can be controlled for achieving improved sol stabilization, controlled gelation and structure as well as enhanced surface properties. However, it is essential to consider the compatibility of SDS with the specific ceramic material and the desired application, as excessive surfactant concentrations or inappropriate surfactant selection may present undesired effects or compromise the performance of aerogel blankets by obscuring the structural integrity.

Finally, this new structure can interact stronger through its adhesion with ceramic fibers. In Fig. 2c, pristine ceramic blankets are formed with randomly oriented fibers as non-woven structures upon integrating the 5 wt.% amount of MSQ aerogel. In Fig. 2d, significant deposition of the aerogel materials can be seen. Clusters of aerogels are evident throughout the fiber matrix. The inset image in Fig. 2d shows aerogels attaching to regions of fibers. As the aerogel MTMS concentration in the aerogel increases to 10 wt.% (Fig. 2e), more deposition becomes evident. As the MTMS concentration increases, space in the fiber matrix is dramatically reduced in Fig. 2f–j. As the amount of the MSQ in the aerogel increases, aerogel accumulates more between fibers resulting in limited deposition on them [23, 27, 38]. Thus, the pristine ceramic blanket voids are closed, and there are almost two phases inside the structure: MSQ aerogel clusters and the ceramic blanket. When the weight of aerogel is amplified, it is expected to affect the composite sample's density, porosity, mechanical properties, and thermal conductivity.

Figure 3 presents the cross-section analysis of the 15 wt% MSQ aerogel blankets center, conducted using SEM to perform its microstructure and coating uniformity. The objective was to demonstrate the uniform immersion of the MTMS sol into the blanket, ensuring its even distribution throughout the entire section. As a result, a well-defined and evenly distributed network of MSQ aerogel clusters integrated within the ceramic blanket is revealed. This homogeneous distribution suggests successful wetting and merging of the aerogel within the material, contributing to the composite's physical properties and promoting its potential for applications requiring mechanical properties. Overall, the

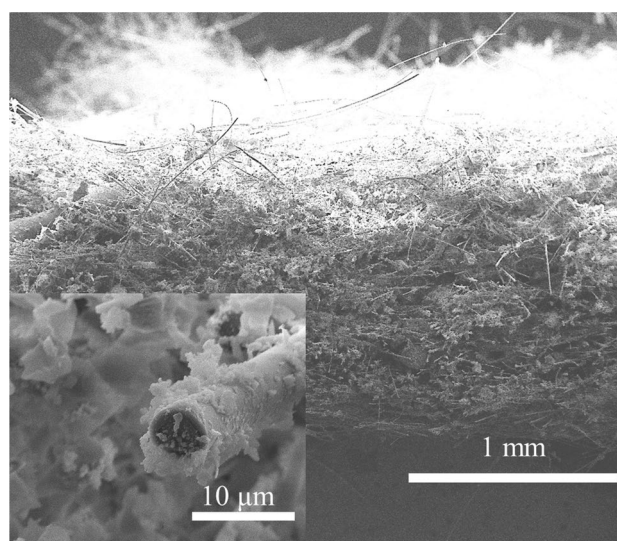


Fig. 3 SEM images obtained from the 15 wt% MSQ aerogel blanket depicting the cross-sectional morphology

Table 1 EDS elemental compositions of the samples

Sample	Elemental composition (%)			
	Si	O	C	Al
MSQ Aerogel	23.25	29.24	47.51	
5 wt% MSQ Ceramic blanket	24.51	39.37	18.5	17.62
10 wt% MSQ Ceramic blanket	27.26	40.44	16.04	16.26
15 wt% MSQ Ceramic blanket	28.47	37.46	22.23	11.85
20 wt% MSQ Ceramic blanket	33.90	33.25	20.54	12.32
25 wt% MSQ Ceramic blanket	31.9	36.86	20.22	11.02
30 wt% MSQ Ceramic blanket	33.16	31.96	29.80	5.08
Pristine ceramic blanket	25.94	42.76	4.64	26.65

cross-sectional analysis demonstrates the effectiveness of MSQ aerogel clusters in the blanket and a well-integrated morphology since the aerogel is evenly dispersed throughout the ceramic blanket.

The elemental composition of MSQ aerogel, pristine ceramic, and MSQ aerogel blanket samples was determined with SEM-EDS analysis, as shown in Table 1. The MSQ aerogel consisted of –23% Si, –29% O, and –48% C, which is in good agreement with the aerogel material that is expected to be formed. The elemental composition of the ceramic blankets consists of –26% Si, –43% O, –4.64% C, and –27% Al. It was observed that the blanket samples showed Al atoms in addition to Si, O, and C elements. The pristine ceramic blanket contains Al, and MSQ aerogel has a C element as a distinguishing material. According to these results, the total element concentration of Al and C in each composite material corresponds to –31 to 36%. With the increasing MTMS in the composite sample, the Al elemental

fraction gradually decreases, which can be attributed to increasing amounts of MSQ aerogel in the composite material. The aerogel synthesized on the surface of the ceramic insulation material can be confirmed with elemental concentrations, and a good correlation was found between C and Al concentrations and the MTMS content.

Nitrogen adsorption-desorption isotherms of MSQ aerogel, pristine, and aerogel-coated blankets are shown in Fig. 4. Figure 4a shows isotherm demonstrations of MSQ aerogel and pristine ceramic blankets. The blanket itself is formed with micro-sized fibers that have large pores. Thus, the isotherm of the pristine blanket shows a rapid saturation. The MSQ aerogel shows a very different response in comparison to blanket samples. Absorption and desorption isotherms start and end at different partial pressure values indicating that the porous structure cannot be filled with the gas phase during absorption, and desorption could not be completed. Absorbed nitrogen remains in the large pores. The larger pore body remains full until a lower pressure is reached when the neck is released [39]. Nitrogen adsorption-desorption isotherms of prepared aerogel insulation blankets are depicted in Fig. 4b. The aerogel blankets have type IV isotherms corresponding to the classical H_2 -type hysteresis loops, affirming the presence of mesoporous structures [40, 41]. Note that the hysteresis loops were even more evident when the MTMS concentration of the aerogels gradually increased.

Calculated surface areas and pore properties of aerogel and aerogel insulation blanket samples are presented in

Table 2. According to these BET results, the pristine ceramic blanket's surface area has grown from 5.52 to 226.1 m^2/g by integrating aerogels. The pore volume varied according to BJH from 0.0248 to 0.3221 cm^3/g with increasing MSQ aerogel amounts. Note that the applicability of the nitrogen adsorption-desorption analysis for the evaluation of nanopore structures is limited in the case of a pristine ceramic blanket with pore sizes in the micron scale. This limitation occurs due to larger pore sizes, which are beyond the sensitivity range of the nitrogen adsorption-desorption technique for accurate characterization. Nonetheless, as the MTMS concentration increases, the pore volume also increases. The

Table 2 Multipoint surface area and BJH method adsorption pore volume values of samples obtained via BET measurements

Material	Multipoint BET Surface area (m^2/g)	BJH Method adsorption pore volume (cm^3/g)
MSQ Aerogel	203.5	0.1055
MSQ+ SDS Aerogel	191.35	0.1188
Pristine ceramic blanket	5.52	0.0248
5 wt% MSQ Ceramic blanket	65.74	0.1429
10 wt% MSQ Ceramic blanket	110.61	0.2473
15 wt% MSQ Ceramic blanket	166.9	0.2971
20 wt% MSQ Ceramic blanket	191.9	0.3095
25 wt% MSQ Ceramic blanket	226.1	0.3205
30 wt% MSQ Ceramic blanket	225.1	0.3221

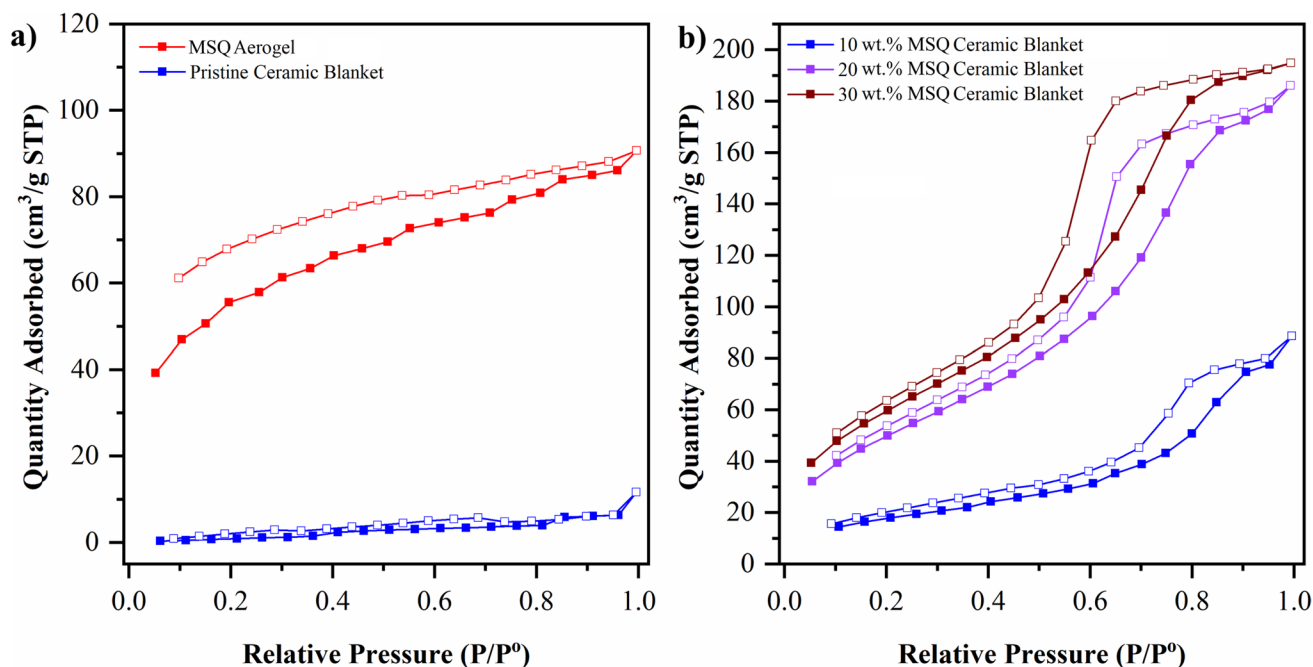


Fig. 4 N_2 adsorption-desorption isotherms of **a** MSQ aerogel and pristine ceramic blanket and **b** MSQ ceramic blankets (10–20–30 wt%)

change in the pore size distribution was associated with the strength of the formed silica network. Increasing the silica source increased the density and, thus, the power of the aerogel structure. The strengthening of the structure increased the resistance to capillary forces formed during drying. For instance, since the silica network structure of the insulation blanket was observed at 5 wt%, the MTMS concentration was weak, and the tiny pores in its structure collapsed during drying and formed larger pores.

Figure 5 demonstrates FTIR spectra of samples containing MSQ aerogel, the pristine ceramic blanket, and blankets with varying amounts of incorporated MSQ silica aerogels. When the results of the composite samples are grouped, peaks display no dissimilarity; however, they differ in intensity. Nevertheless, significant peak differences exist between MSQ aerogel and pristine ceramic insulation material. In Fig. 5, the pristine ceramic blanket has only two distinct peaks at 828 and 1063 cm^{-1} , assigned to the symmetrical stretching and asymmetrical absorption of Si–O–Si [42, 43]. However, unlike the pristine ceramic, the MSQ composite insulation material shows various peaks. These absorption peaks are stretched Si–C bonds at 770 and 1260 cm^{-1} [19, 44–47]. Asymmetrical stretching of –CH₃ bonds appear at 2976 and 1416 cm^{-1} [19, 47] and are intensified due to the addition of MSQ aerogel. Due to this observation, it can be expected that MSQ aerogel and coated ceramic blankets are hydrophobic.

Raman spectra of MSQ and MSQ + SDS aerogels are shown in Fig. 6, revealing characteristic peaks that offer significant insights into the molecular structure and

composition of the materials. In Fig. 6a, symmetric and asymmetric stretching vibrations of –CH₃ groups (methyl groups) at 2918 and 2980 cm^{-1} , respectively, affirm the presence of methyl groups in aerogels conducted with MSQ [48, 49]. The bending vibration of Si–CH₃ bonds, represented by the peak at 1420 cm^{-1} [48, 50], provides further evidence of the existence of methyl groups. Additionally, the bending vibration of Si–O–Si bonds, as defined by the peak at 798 cm^{-1} [51, 52], characterizes the silica network formation in MSQ aerogels. The peak at 480 cm^{-1} corresponds to the bending vibration of Si–O–CH₃ bonds, substantiating the presence of methoxy groups. Notably, in Fig. 6b, the symmetric stretching vibration of Si–O–Si bonds was identified at 475 cm^{-1} [53], while the asymmetric stretching vibration of –CH₃ groups (methyl groups) appeared at 1413 cm^{-1} [48]. Additionally, the symmetric stretching vibration of Si–C bonds was observed at 1270 cm^{-1} . Moreover, Raman spectra displayed bending vibrations of Si–CH₃ bonds at 798 cm^{-1} , Si–O–CH₃ bonds at 771 cm^{-1} [50], and Si–CH₃ groups at 684 cm^{-1} [49]. These peaks provide valuable distinct fingerprints to comprehend the molecular characteristics of MSQ aerogels. Vibrational modes and scattering properties of MSQ aerogels have been altered by SDS, resulting in a stronger disordering within the silica framework, an increase in Raman vibrational peaks, and the induction of gelation leading to a more organized silica framework. Also, indirectly, SDS increases the crystallinity in MSQ aerogels, resulting in intensified Raman scattering and peak intensities. In Fig. 6c, characteristic peaks of 10 and 30 wt% MSQ aerogel blankets are revealed, which offer significant insights into the molecular structure and composition of the materials. As the content of MSQ aerogels increases in the composite material, significant changes are observed in the Raman spectra. Specifically in Fig. 6d, peaks at 745, 1069, 1299 and 1441 cm^{-1} are no longer detected while remaining peaks are weakened. The loss of these specific peaks can be attributed to several factors, such as the increase in MSQ aerogel content within the composite while maintaining the ceramic blanket's constant weight leading to the double peaks weakening in Raman spectra. Moreover, the higher MSQ content results in a greater and thicker coating on the ceramic blanket, potentially causing the peaks to become disguised, thereby reducing their exposure to the laser. Consequently, changes in the Raman spectra suggest alterations in the composite's crystallinity and interactions between MSQ aerogels and the ceramic blanket.

XRD analysis of MSQ and MSQ + SDS aerogels are shown in Fig. 7a, revealing two broad peaks. the corresponding functional groups, Si–CH₃ and Si–O–Si, are characterized by peaks at 9 and 22°. The presence of Si–CH₃ groups contributes to the peak at 9° suggesting specific silica attached by alkyl groups. In addition, the peak at 22° relates to the Si–O–Si group's presence of alignment within

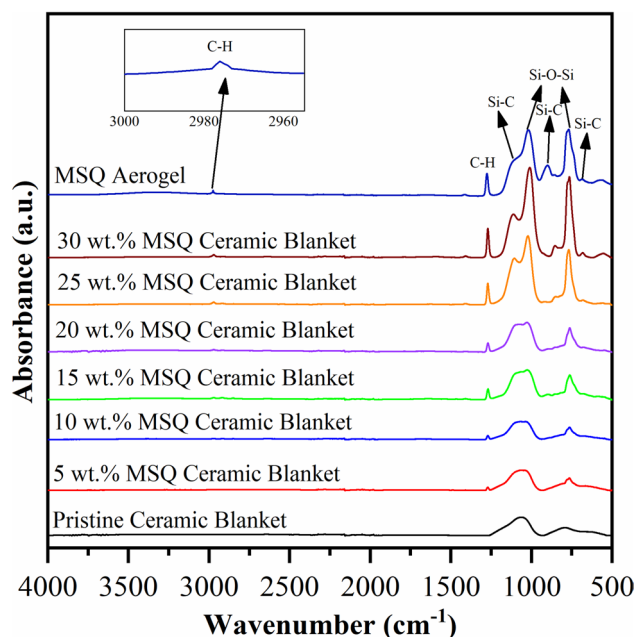


Fig. 5 ATR-FTIR spectra of MSQ aerogel and blankets samples

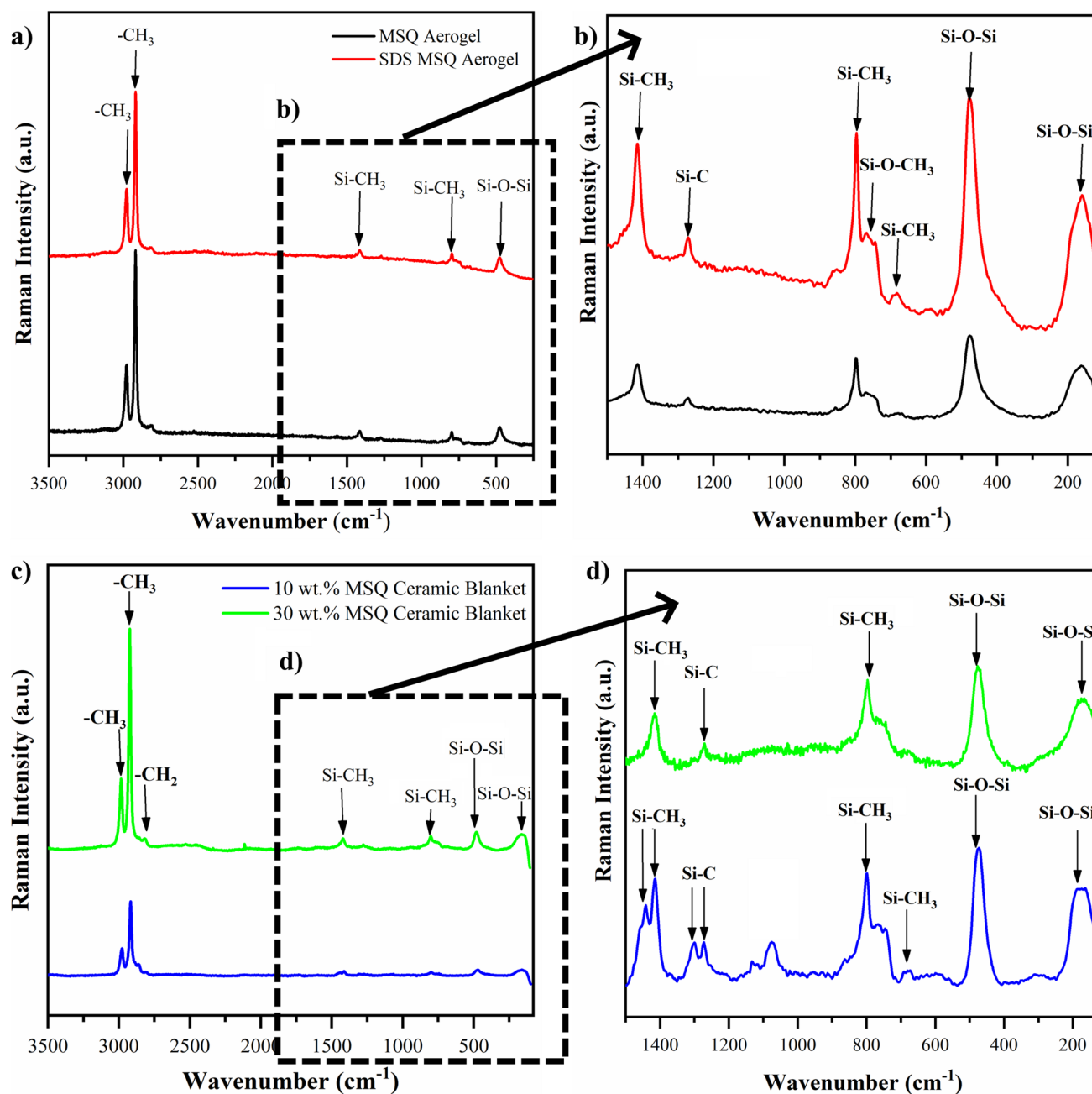


Fig. 6 Raman Spectroscopy of the **a, b** MSQ+SDS aerogel and **c, d** MSQ aerogel blanket

the MSQ aerogel. Moreover, SDS addition has led to an intensification of these peaks, indicating a higher degree of order due to the interaction between the anionic surfactant and the MSQ aerogels functional groups, advancing a more organized configuration. SDS likely altered the nucleation and growth of silica particles during the synthesis, potentially promoting the formation of more structured segments. The XRD pattern highlights significant peaks at 9 and 22° of diffraction angle, results consistent with those reported in the literature [54–57]. Note that comprehensive analyses

by complementary characterization techniques like Raman spectroscopy and SEM are needed to fully understand the specific implications of the observed XRD peaks and the effects of SDS addition on the crystallinity of the aerogels. In this study, all three approaches are in good agreement. In Fig. 7b, one containing 10 and 30 wt% MSQ aerogel ceramic blankets, XRD analysis was performed. The material with lower MSQ content exhibits a lower peak intensity at 9° and higher peak intensity at 25°. These alterations suggest that the composite ceramic blanket's composition has altered the

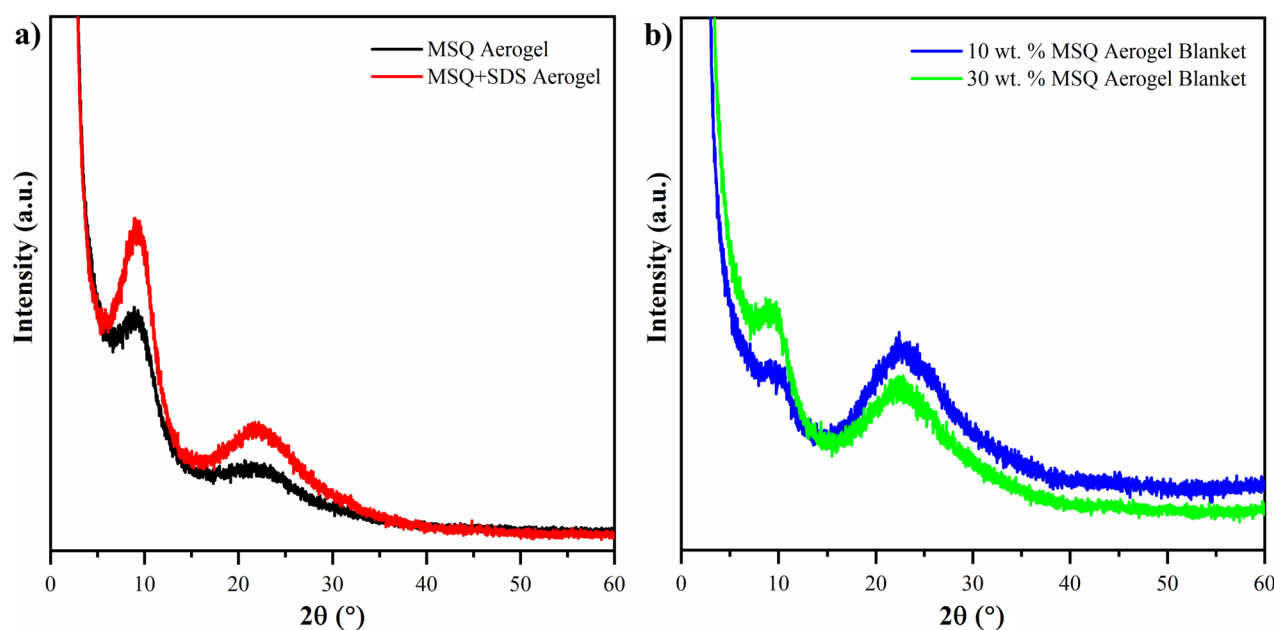


Fig. 7 XRD patterns of **a, b** MSQ and MSQ + SDS aerogels and **c, d** 10, 30 wt% MSQ aerogel blankets

materials' crystalline properties. The material with higher MSQ content has a higher crystalline content, which could lead to more well-defined crystal regions, resulting in higher peak intensities at 9 and 25° [48]. Conversely, the material with lower MSQ content may have a preferred crystal orientation induced by the composite, aligning more favorably with the incident X-rays at 25° , thus resulting in a higher peak intensity at that angle. Also, the 9° peak in

the XRD analysis establishes a contextual correlation with the increasing MSQ content, implying the increase of the Si-CH₃ group. This association is further confirmed by the increased prominence of this peak in the XRD analysis, which in turn is further validated by the increased presence of MSQ aerogel.

Alterations in aerogels particle size resulting from SDS are visually represented in Fig. 8, demonstrating how the

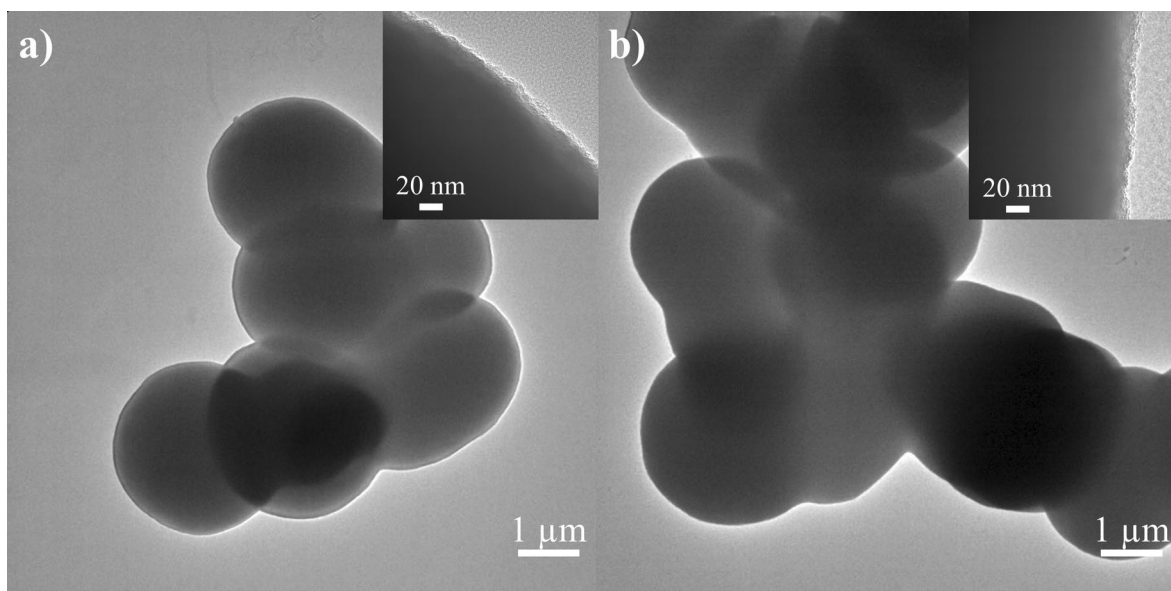


Fig. 8 TEM images of **a** MSQ and **b** MSQ + SDS aerogel

material's microstructure has been impacted. This study observed distinct silica spherical structure characteristics of MSQ aerogels [58]. TEM images revealed an enhanced interconnected network and reduced void spaces between structural entities observed for MSQ-SDS aerogel. In contrast to Fig. 8a, i.e., the MSQ aerogel, the presence of the surfactant slightly increased the particle size in Fig. 8b, i.e., the MSQ + SDS aerogel. In Fig. 8b, SDS increased aggregation as a result of higher particle size. This result can be confirmed with BET analysis. In the literature, larger particles have lower surface areas due to less nitrogen (N_2) exposure, decreasing the surface area while aggregating and reducing the overall accessible surface area [59]. MSQ aerogels' BET results are shown in Table 2 and coupled with TEM images.

The thermal conductivity of insulation blankets is affected by differences in bulk densities. These insulating materials between fibers consist of air pockets, affected by external pressure and chemical interactions, resulting in volume reduction causing undesired bulk density increases. Depending on the manufacturing process of ceramic insulation blankets, various by-products (small particles) might be present between blanket fibers. These by-products can be removed from the structure by dusting when external pressure is applied and solvent exchange with the sol-gel technique used to obtain aerogel blankets. In the solvent exchange process, solvents cause fiber reorientation with a slight increase in volume [36]. In this study, the 5 and 10 wt% MSQ aerogel composite blankets bulk density is lower than the pristine ceramic blanket. This fact is related to the lower bulk density of MSQ aerogels in comparison to the pristine blanket and lower MSQ aerogel content in the composite. Note that these results are also confirmed in the literature [41]. After all, 15 wt% or more of MSQ silica aerogel in the composite blanket, followed by a volume shrinkage, leads to a bulk density increase. Figure 9 displays the bulk densities of the MSQ aerogel substance in the ceramic blanket. However, density values are essential for the determination of thermal conductivity values. The resulting volume shrinkage tends to affect the porosity and thermal conductivity performance. As the MSQ aerogel structures grow and fibers come together, the resulting volume shrinkage can also be confirmed in the inset in Fig. 9.

The thermal properties of the samples were measured using the Hot Disk plane source device. The device has a plane heat source. Thermal properties of a material (such as the heat transmission coefficient, thermal conductivity, and heat capacity) can be measured quickly using the time-dependent (transient) method. Consistent results were obtained by taking measurements twice, both from the top and bottom sides of the composite samples, shown in Table 3. MSQ aerogels' thermal conductivity ranges from 0.018 [27] to 0.164 $Wm^{-1} K^{-1}$ [60]. MSQ aerogels

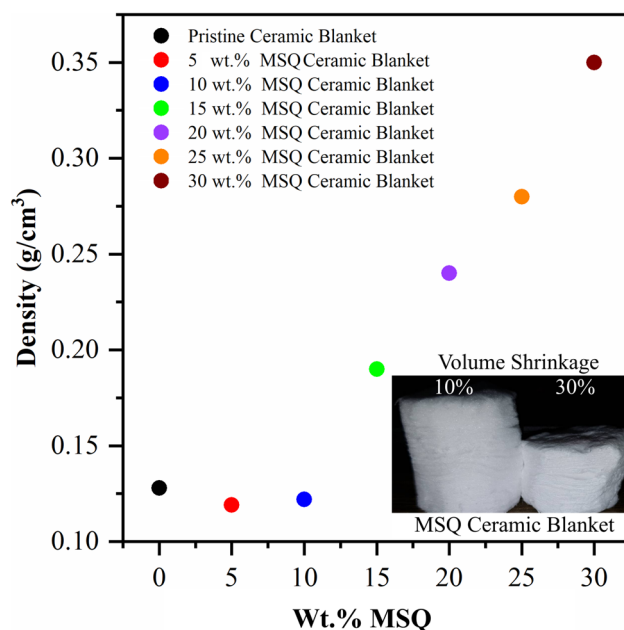


Fig. 9 A volume shrinkage followed the bulk density of the MSQ aerogel composite blanket

Table 3 The thermal performance of studied aerogel and aerogel blanket samples

Sample	Thermal Conductivity $\frac{W}{m.K}$	Experimental Density (g/cm³)	Porosity (%)
Pristine ceramic blanket	0.120	0.128	92.39
5 wt% MSQ ceramic blanket	0.085	0.119	92.47
10 wt% MSQ ceramic blanket	0.1185	0.122	95.20
15 wt% MSQ ceramic blanket	0.114	0.190	92.08
20 wt% MSQ ceramic blanket	0.1155	0.240	90.60
25 wt% MSQ ceramic blanket	0.1425	0.281	87.12
30 wt% MSQ ceramic blanket	0.096	0.350	81.64
MSQ Aerogel	0.099	0.092	93.84

obtained in this study show a thermal conductivity of 0.099 $Wm^{-1} K^{-1}$, consistent with results in the literature.

Note that the pristine blanket in this study exhibits a relatively high thermal conductivity in contrast to many other blankets used in the literature [7, 61–63]. However, aerospace applications typically include a number of requirements. For insulation materials developed for engine and avionic regions of aircrafts, standards for these properties exist. Both standards and required properties were provided in Sect. 1. For the purpose of fulfilling all requirements

ceramic fibers were chosen in contrast to fiberglass which excels primarily in the thermal conductivity performance.

In general, adding aerogels decreases the thermal conductivity in comparison to the pristine blanket due to the porous structure. The 5 wt% sample has the lowest density (0.119 g/cm^3) and the lowest thermal conductivity among the composite materials. The increase in aerogel content up to 30 wt% results in a matrix where almost every region is covered with aerogel. Hence, the thermal conductivity has decreased significantly in samples with 30 wt% fillers. The structure changed from a fibrous matrix to an almost dense aerogel matrix [60].

In thermal conductivity measurements, the specific temperature used with these devices can vary depending on the material being studied and research objectives. Each method has its temperature range suitable for obtaining accurate measurements. For example, the Steady Hot Plate Apparatus, Transient Hot Plane Method, Laser Flash Apparatus, and Heat Flow Meter can be operated within specific temperature ranges based on their design and capabilities. The temperature range selection depends on factors such as the thermal properties of the material, expected temperature conditions in research, and sensitivity and accuracy requirements of measurements. Also, the choice of conducting thermal conductivity measurements at room temperature instead of higher temperatures can be prompted by various factors. Firstly, aerogel blankets are often studied at room temperature because it represents a common and standardized reference point for comparison across different studies. Using room temperature for researchers can establish consistent and comparable results, making it easier to evaluate and

validate the thermal properties of aerogel blankets. However, it is important to note that higher temperature measurements are not entirely disregarded in aerogel blankets. In some cases, researchers perform measurements at elevated temperatures to investigate the temperature-dependent behavior of aerogels [64, 65]. These studies aim to understand how the thermal conductivity of aerogels changes as temperature increases, which can be helpful in specific applications or thermal management scenarios. In summary, the focus on room temperature measurements for aerogel blankets is often determined by the need for standardized comparison and consistency in research and industry. However, higher temperature measurements are also conducted in specific studies to explore temperature-dependent behavior. In addition, within the framework of this study, aerospace applications are focused on. For insulation of avionics 71°C is the upper boundary of the allowed operational temperature (RTCA DO-160G), which also contributed to the decision to perform measurements at lower temperatures.

Note that thermal conductivity results were obtained by the TPS method (derivative). Compared with studies in the literature, the results are 30–140% higher than steady-state (absolute) measurements [65] since higher temperatures are taken into account due to the experimental regime of $30\text{--}199^\circ\text{C}$. Hence, direct comparison is quite difficult since measurements are often taken at room temperature [8].

TGA curves of silica aerogel and ceramic aerogel blankets synthesized utilizing an MTMS precursor were used to analyze the thermal stability and are shown in Fig. 10. It was observed that the thermal degradation processes of MSQ aerogel and its composites consisted of three steps.

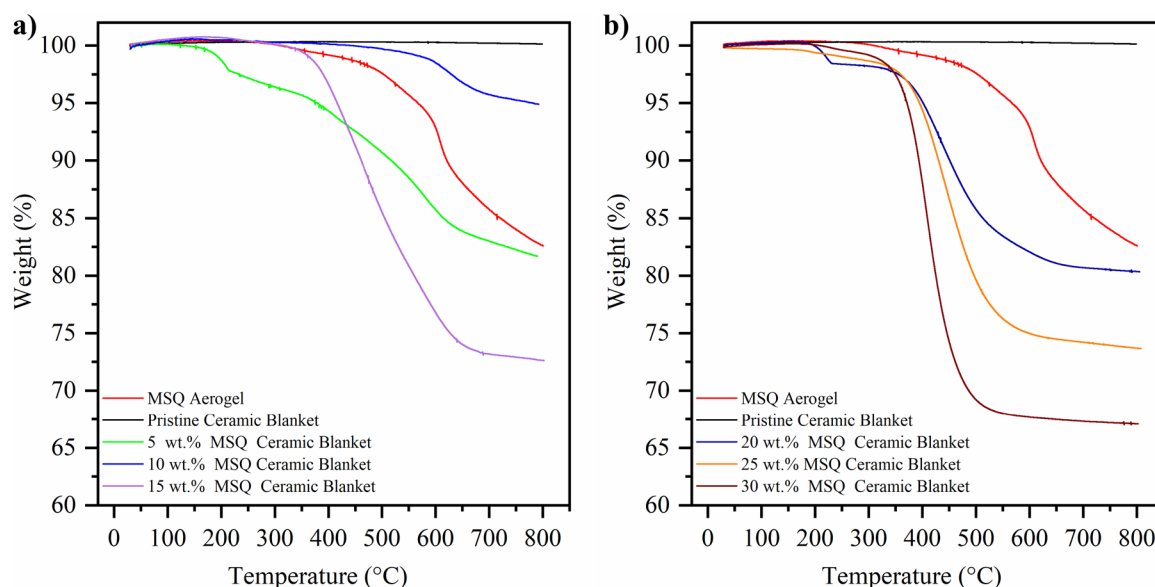


Fig. 10 TGA of **a** MSQ aerogel, ceramic blanket, and MSQ ceramic blanket (5–15 wt%), **b** MSQ aerogel, ceramic blanket, and MSQ ceramic blanket (20–30 wt%)

Removing the water, solvent residues, and precursor molecules on the material surface caused a slight mass loss of up to 150 °C. Almost no significant degradation behavior is observed up to 400 °C. The second mass loss starts at 400 °C and reaches up to 600 °C due to the decomposition of organic groups in the MSQ aerogel structure [45]. It can be interpreted as an indication of loss of the hydroxyl (–OH) and aliphatic (–CH₃) silica end groups. The third degradation step lies above 500 °C, where organic structures are entirely decomposed [61]. Eventually, 82.5 wt% of the MSQ aerogels with remained intact at 800 °C, showing that the obtained material had high thermal stability.

The $T_{d5\%}$ (temperature of 5% weight loss) and residual weight data obtained from Fig. 10a, b are given in Table 4. These two critical parameters are often analyzed in TGA, $T_{d5\%}$, and total residual weight. The $T_{d5\%}$ value provides information about the material's thermal stability. Generally, a higher $T_{d5\%}$ value suggests better resistance to thermal degradation since the material can withstand higher temperatures. Residual weight implies the remaining weight % of the material after complete thermal degradation. It represents the remaining material that does not undergo significant weight loss during heating [66]. The $T_{d5\%}$ temperatures optimum results are 566.97 °C for the MSQ aerogel, 702.19 °C for the 10 wt% MSQ ceramic blanket and 415.99 °C for the 15 wt% MSQ ceramic blanket which suggests excellent thermal stability, while the pristine ceramic blanket does not show any deterioration. The residual weight typically consists of inorganic components after the organic components have been volatilized or decomposed. Hence, the ash formation amount increased as the aerogel's MTMS concentration increased due to the additional silica presence. The presence of aerogel acts as a heat barrier within the insulating material. In addition, aerogel has a maximum service temperature, and its thermal performance and stability decrease above this temperature. Based on this, a careful interpretation of Fig. 10; Table 4 reveals a correlation between aerogel amount and increased ash content. The thermal

conductivity of the materials can yield a plausible mechanism. The ceramic blanket has a higher thermal conductivity than the other materials, as presented in Table 3. Moreover, the 10 wt% MSQ composition has reached an optimum point regarding the aerogel composite's overall properties, improved stability, and reduced mass loss. This composition may balance the ceramic content and the network structure, leading to enhanced thermal stability since the presence of CH₃ groups in the aerogel structure introduces weaker van der Waals forces compared to stronger covalent bonds in Si–C, Si–O, and Si–O–Si. Thus, this weaker bonding in CH₃ groups can increase molecular mobility and thermal degradation vulnerability. Consequently, aerogel blankets with higher concentrations of CH₃ groups may exhibit higher mass loss during TGA analysis, as these bonds are more prone to breaking under thermal stress.

Furthermore, higher concentrations (15, 20, 25, and 30 wt%) may lead to excessive aerogel, negatively impacting the overall structural integrity and increasing mass loss during thermal degradation. Thus, increasing temperatures accelerate the deterioration of insulation blankets and thermal stability by applying heat to aerogel structures in fibers and transmitting the heat from additional points. Hence, it can be assumed that the presence of –CH₃ organic structures in the aerogel deteriorates at increasing temperatures. Thus, resulting gases accelerate the combustion reaction by contributing and accelerating the combustion reaction and increasing it cumulatively. These results are consistent with data reported in the literature [67].

A simultaneous TGA-FTIR analysis presented in Fig. 11 was conducted to investigate the thermal decomposition mechanism of the MSQ aerogel with composite materials. Figure 11a shows FTIR spectra of MSQ aerogel gas samples at temperatures of 334, 411, 596, and 723 °C corresponding to 32.4, 41.1, 54.7, and 70.4 min during the degradation. Water, methane, silane, and carbon dioxide gases were formed through pyrolysis of the samples by reacting with volatile gases during thermal degradation [46]. At 32.4 min at a temperature of 334 °C, moisture and organic solvent peaks were observed in the spectrum, yielding characteristic peaks at 1293 and 2000 cm^{–1} [68]. Similarly, at 41.1 min, continuous heating at 411 °C showed a weak peak at 3200 cm^{–1} because of C–H, indicating aliphatic group deterioration. Moreover, at 596–723 °C the most distinctive peak is observed at 750 cm^{–1} and is related to CO₂ generated by combustion reactions. Possible deteriorated groups are due to C–H and Si–CH groups. In total, MSQ aerogels weight loss is approximately 17.5 wt%. FTIR spectra of gas samples obtained during the 10 wt% MSQ aerogel-blanket composite degradation is presented in Fig. 11b. The scan at 11.4 min and 128 °C showed peaks that might occur due to small unreacted oligomers remaining from the synthesis. Many various peaks were observed because of the release

Table 4 Thermal performance of the samples in TGA given with the $T_{d5\%}$ and residual weight

Samples	$T_{d5\%}$ (°C)	Residual Weight
MSQ Aerogel	566.97	82.588
Pristine ceramic blanket		
5 wt% MSQ ceramic blanket	371.49	81.695
10 wt% MSQ ceramic blanket	702.19	94.902
15 wt% MSQ ceramic blanket	415.99	72.615
20 wt% MSQ ceramic blanket	401.15	80.339
25 wt% MSQ ceramic blanket	394.56	73.663
30 wt% MSQ ceramic blanket	371.09	67.087

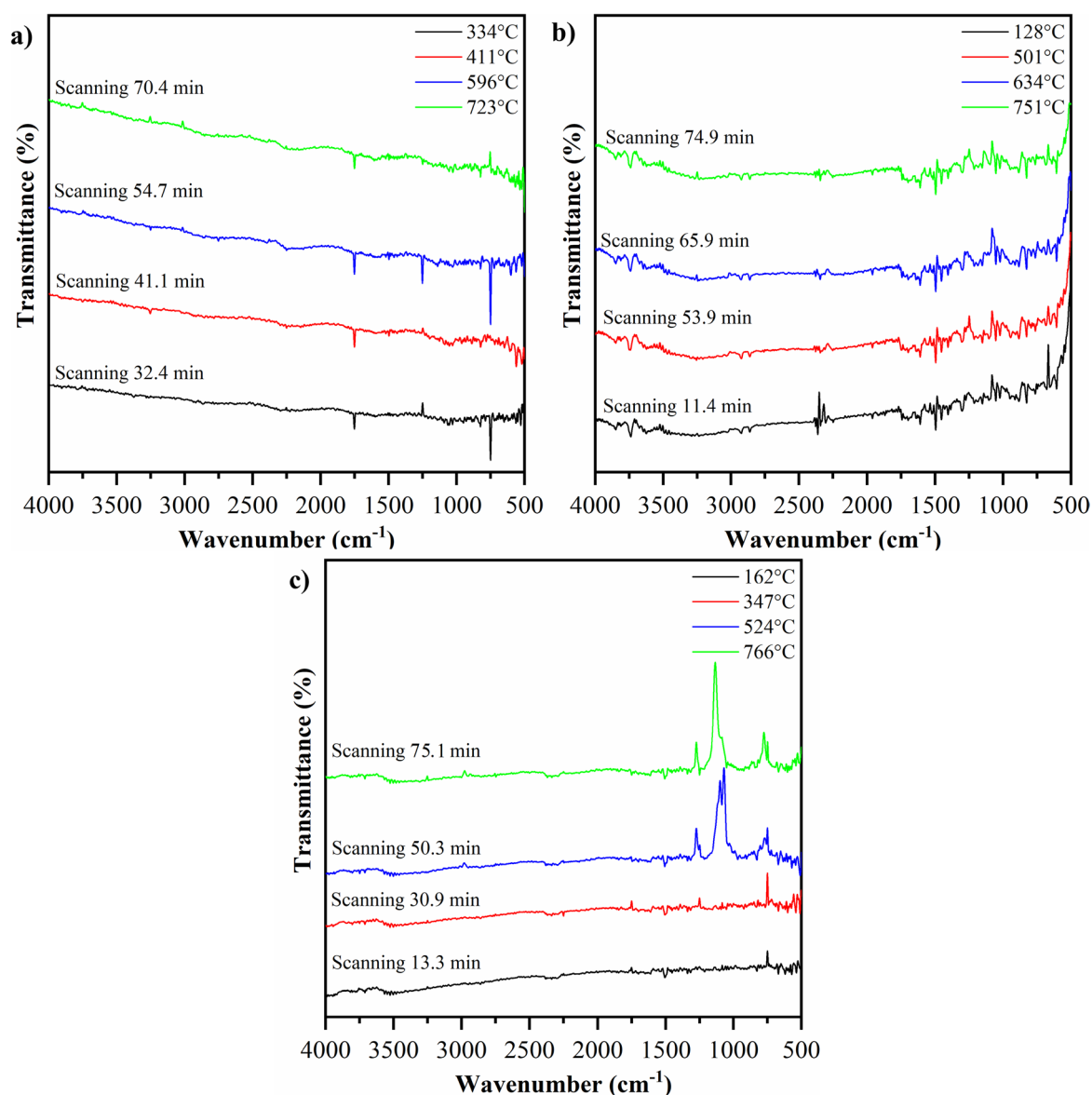


Fig. 11 TGA-FTIR analysis spectra of **a** MSQ aerogel, **b** the 10 wt% MSQ aerogel blanket and the **c** 30 wt% MSQ aerogel blanket

of hydrogen from small oligomers during combustion reactions. Strong characteristic peaks around 2300–2200 cm^{-1} were due to the formation of CO_2 and the solvent's degradation [46, 68]. At 65.9 min and 634 °C, asymmetric Si–O–Si vibrations corresponding to 1120 cm^{-1} reveal the maximum intensity of Si–O–Si. Afterward, a significant peak for increasing temperatures implies that combustion reactions have reached an equilibrium and are slowing down [69, 70]. The overall 10 wt% MSQ aerogels blankets weight loss was about 5.1%. Furthermore, in Fig. 11c, spectra obtained during the 30 wt% MSQ aerogel-blanket composite degradation are presented. At 30.9 min, a Si–O–C peak appears at 820 cm^{-1} , which indicates the resulting thermal degradation [71]. Also, distinctive and intense peaks were observed at

1100 cm^{-1} corresponding to Si–O–Si asymmetric vibrations starting at a temperature of 524 °C while methane peaks were revealed at 1300 cm^{-1} and intensified, being present until 766 °C. The effect of increasing temperature, inert atmosphere, and side groups form methane. Such particular reactions usually happen toward the end of the experiment [68, 69]. In addition, methane-induced combustion peaks at 1300 cm^{-1} and Si–O–Si asymmetrical absorption peaks at 1100 cm^{-1} were observed at 75.1 min. Also, the specific peak of Si–O–C was revealed at 820 cm^{-1} . Unlike both previous figures, the intensity of the peaks obtained here is relatively high. The reason for this situation can be evaluated as the spread of the combustion reaction to the inner layers of the aerogel insulation blanket. Thus, volatile gases

accelerate the degradation of siloxane structures by feeding remaining CH_3 groups with inner layers [45]. For this reason, the remaining mass had the highest final weight loss with 32.91%.

The mechanical performance of the samples was evaluated with tensile and three-point bending tests concerning the amount of MSQ content in the aerogel in the synthesized composites. Results from the tensile test are given in Fig. 12, while three-point bending tests are depicted in Fig. 13. Maintaining the structures' integrity under external forces for the MSQ aerogel insulation blankets with various MTMS ratios is crucial. Aerogel showed very brittle characteristics with low mechanical strength due to its morphology and nanostructure. Due to aerogels' porous and open-cell nature, it is very brittle and fragile to sustain external pressure. Moreover, the pristine ceramic blanket did not show a yield point and started disintegration from the beginning of the test. Aerogel-ceramic blanket composites, however, showed measurable and promising mechanical properties. The tensile test showed a linear region, where the composites only go through elastic deformation up to the yield point, increasing as the strain increases. The maximum tensile stress of aerogel blankets with 5, 10, 15, 20, 25, and 30 wt% additives are 5.62, 9.89, 7.14, 14.89, 14.34, and 22.90 MPa, respectively. The tensile stress graph (inset of Fig. 12) indicates that the 5 wt% MSQ ceramic blanket material shows a three-stage degradation curve: (I) elastic behavior, (II) covers propagation of various failure modes including matrix cracking, (III) delamination and friction

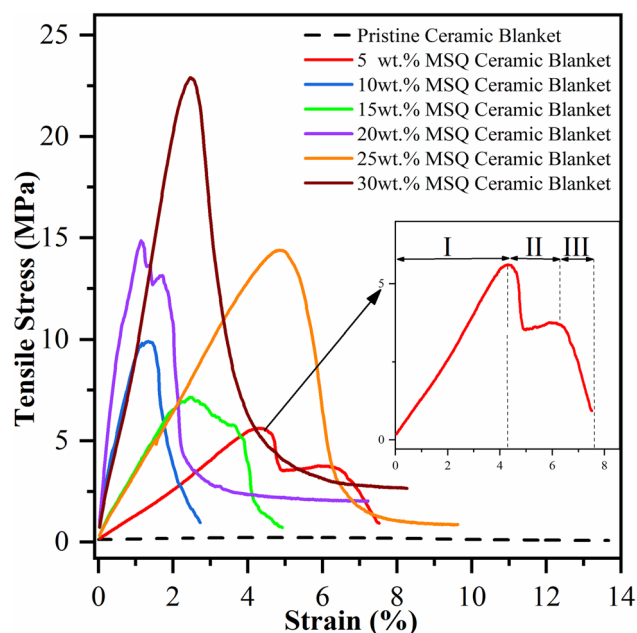


Fig. 12 Tensile stress-strain curves for pristine and MSQ aerogel-coated ceramic blankets

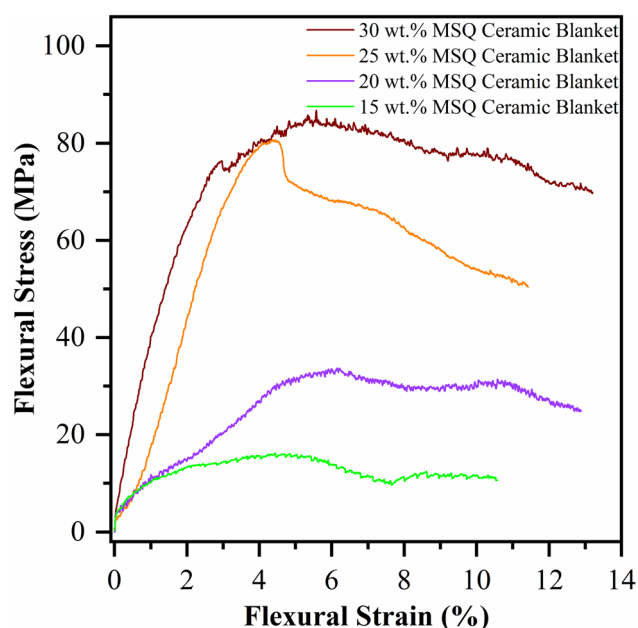


Fig. 13 Flexural stress-strain curves in three-point loading tests for MSQ ceramic blankets

slip zone. At the end of stage I, the deformation and crack formation started. However, the aerogel blanket could still carry the tensile strength until delamination started, after which the carrying capacity gradually decreased, and eventually, a rupture occurred. The tensile stress of MSQ aerogel silica blankets and the mechanical performance increased with increasing MTMS content while the elongation values decreased. They also filled the gaps in the ceramic fibrous matrix and connected the fibers, thus, increasing the rigidity of composites.

The three-point bending test was conducted to determine the flexibility of the aerogel blanket, and the results are shown in Fig. 13. Pristine and composite samples loaded with less than 15 wt% MSQ aerogel did not provide enough strength in the flexure testing setup [27]. The yield strengths of the aerogel blankets from the highest to the lowest were 84.32, 68.14, 33.67, and 15.34 MPa for the 30, 25, 20, and 15 wt% MSQ aerogel loaded blankets, respectively. The matrix provides flexibility for requirements in relevant aerospace applications. Thanks to the structure of the fibers, it provides macro flexibility, performance, and compatibility with the environment, which is needed when processing or installing the insulation product on potential aerospace platforms. As the weight of silica aerogel in the insulation blanket increases, the flexural modulus gradually increases; thus, the rigidity increases. It also indicates that silica aerogel restricts the movement of fibers in the matrix under stress direction. Overall, the strain capacities of the samples were close to each other. The total flexural stress develops significantly as the aerogel increases within the insulation

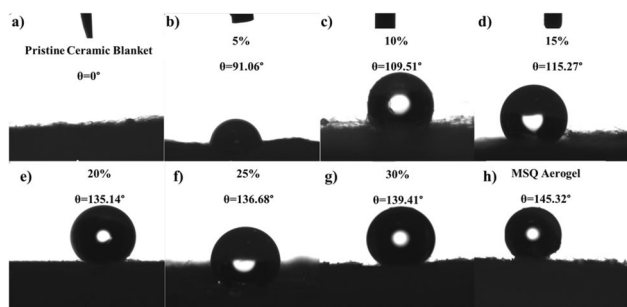


Fig. 14 Water contact angle on the surface for **a** the pristine ceramic blanket and **b** 5 wt%, **c** 10 wt%, **d** 15 wt%, **e** 20 wt%, **f** 25 wt%, **g** 30 wt% MSQ aerogel blankets as well as **h** MSQ aerogel

blanket. Moreover, fibers get too close to each other and improve rigidity. When the samples reach the failure point, fluctuations happen, and the samples' structural integrity is lost. Remarkably, similar results were also found in the literature [23].

Hydrophobic properties of materials primarily result from their physical and chemical properties. The physical hydrophobicity originates from void defects inside the microstructure, while the chemical characteristic comes from the $-\text{CH}_3$ methyl functional group, which interacts with water [61]. Contact angle measurements were performed, and the results are presented in Fig. 14. Hydrophobicity samples were characterized by measuring the contact angle (θ) of a water droplet (5 μl). According to the literature, surfaces of aerogels and composites obtained from MTMS precursors are expected to be hydrophobic. This is due to the MTMS precursor's chemical structure with trifunctional methyl groups. Due to these groups present in the precursors, the composite materials are hydrophobic without any post-treatment or specific surface modification steps. In Fig. 14a, the pristine ceramic blanket does not show any water contact angle and simultaneously absorbs the water droplet. SEM results depicted in Fig. 2c show that pristine ceramic blankets microstructure is spacious between fiber structures and support this observation. Furthermore, these results agree with the observations presented in FTIR results. Remarkably, MSQ aerogel gives a 145.32° typical water droplet on the surface contact angle, indicating that it is superhydrophobic, as shown in Fig. 14h. When these results are examined together, the results correlate with the water contact angle of the insulation blanket (Fig. 14b–g) obtained by increasing the addition of MSQ in the aerogel developing hydrophobicity [25, 47].

The effect of air permeability of the synthesized MSQ aerogel insulation blankets was investigated. Results are presented in Fig. 15. We can consider the air permeability test under two aspects: surface area and mechanical properties (flexibility). Composites may have low-medium (for aerogel

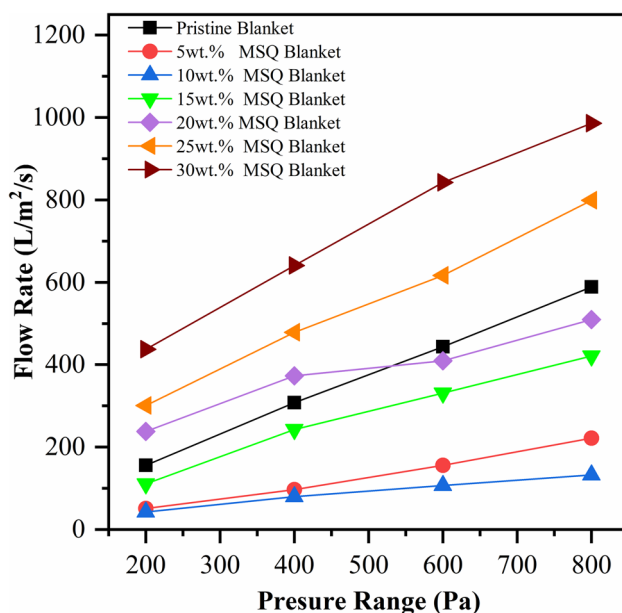


Fig. 15 Air permeability of pristine and MSQ-coated blankets is given as flow rate at various pressure ranges

amounts of 5–15 wt%) or high surface areas (for aerogel amounts of 20–30 wt%), respectively [72, 73]. In the first case, the air permeability decreased significantly compared to the pristine ceramic blanket, which is the most flexible material compared with the MSQ aerogel blankets (5–15 wt%) since aerogels form a new layer. This layer did not previously exist in the pristine ceramic blanket, as confirmed by SEM and BET results presented in Fig. 2; Table 2, respectively. Due to the layers' indented and protruding internal structure, it inhibited traversing air molecules. Therefore, air permeability was significantly reduced in all three samples (5–15 wt%). Both surface area and flexibility are two crucial conditions for attaining low air permeability values measured, especially for 5–10 wt% of the composites. Moreover, 20 wt% MSQ blanket values confirm the air permeability performance since, in this case, an important phenomenon is taking place. At altering air pressures, the material's air permeability performance also fluctuates. Between 200 and 500 Pa, the material shows nearly close but high air permeability compared to the pristine ceramic blanket, but less air passed among 500–800 Pa. Considering this situation, aerogel particles in the composite material fall over from the structure because of the pressured airflow, which results in lowered air permeability at increasing pressures. Correspondingly, missing aerogel indirectly declines surface area. Subsequently, for 25–30 wt% MSQ blankets, the air permeability increases significantly due to the rigidity and high surface area. High rigidity presents a low damping ability. Increased surface area leads to an enhanced layered interconnected structure resulting in less free space and gaps in the

matrix forcing air molecules to pass more rapidly through the material. Thus, air permeability increases significantly.

4 Conclusion

In this manuscript, MSQ silica aerogel blankets were synthesized in situ on an alumina ceramic insulation blanket using an anionic surfactant (SDS) with the acid-base sol-gel process followed by drying at ambient pressure. With the addition of SDS, the electrostatic and hydrophobic attraction in aerogel was developed, resulting in better interaction between fiber and aerogel. Characteristics of insulation blankets differ from each other with dip-coating of various weights of MSQ aerogel. Results show that the multifunctional properties of aerogel blankets, including mechanical and thermal performances, affect the aerogel weight properties, which are also affected by application areas. As a result, two different multifunctional structures were obtained. As a first type, blankets with low thermal conductivity and diffusion were combined with the lowest density values (5–10 wt%). Considering the aerogel insulation blanket in SEM results, the gap between fibers of the pristine ceramic blanket decreased gradually with the addition of MSQ aerogel. This way, the thermal conductivity decreased (0.12 to 0.094 W/mK). While achieving these results, it was crucial not to increase the density (pristine ceramic blanket 0.1280 g/cm³, 5–10 wt% MSQ ceramic blanket 0.119 and 0.122 g/cm³) as the mechanical properties (tensile stress 9.89 MPa) were also enhanced. Also, these samples are pretty flexible and have a moderately graded hydrophobic surface character (91.06 to 109.51°). The lowest air permeability was obtained among these samples in which flexural stress increased gradually for 15–30 wt% insulation blankets; these samples are also much more rigid. Furthermore, pristine ceramic insulation blankets are initially hydrophilic and turn superhydrophobic due to the water contact angle being 139.41°. Thus, these materials can be classified as structures with a high potential for various applications on aerospace platforms. Note that these materials might be flame retardant which can be expected due to the presented TGA results. A simple classification would require UL 94 tests. However, aerospace applications require additional sophisticated tests and analyses, which might pave the way for further research. In this regard, studying the effects of the SDS amount on thermal conductivity is also relevant. Distinct peaks at 9 and 22° were established through XRD analysis, aligning with the Si-CH₃ and Si-O-Si functional groups in the MSQ aerogel. The introduction of SDS increased these peaks, signifying an elevated organization of functional groups within the aerogel's structure. This intensification results in correlation and complementarity in Raman and TEM analyses. In addition, the SEM cross-section of the sample confirms the

comprehensive integration of aerogel across the material and immersion from the surface to the inner sections. Besides, Raman and FTIR results indicate no solvent traces peaks in the study, likely attributed to the material's considerable thickness and confirmed convenient drying at 150 °C. In addition, both presented compositions can be used depending on the field of application, as the production is easy to modify and open to scale-up, which is essential for serial procedures in various sectors like the aerospace industry. Furthermore, there is no production limitation, such as supercritical drying.

Acknowledgements We thank the Scientific and Technological Research Council of Turkey (TUBITAK) for supporting Project No: 118C100. This study was also jointly funded by Bursa Uludag University (Grant # FDK-2021-697) and the Turkish Aerospace Scientific Research Projects program (TUSAŞ-BAP).

Author contributions AT prepared and characterized materials, visualized and analyzed data, and wrote the manuscript draft. YO characterized materials, analyzed data, and wrote the manuscript. HIA, provided the conceptual experimental plan, analyzed data, and wrote the manuscript. All authors reviewed the manuscript.

Funding The Scientific and Technological Research Council of Turkey (TUBITAK) [Grant number 118C100], Bursa Uludag University [Grant number FDK-2021-697], Turkish Aerospace Scientific Research Projects Program [TUSAŞ-BAP].

Data availability The data supporting this study's findings are available from the corresponding author upon reasonable request.

Declarations

Competing interest The authors declare that they have no conflict of interest or competing interests.

Ethics approval Not applicable.

Consent to participate Not applicable.

Consent for publication Not applicable.

References

1. S.S. Kistler, *Nature* **127**, 741 (1931)
2. Y. Wu, X. Wang, L. Liu, Z. Zhang, J. Shen, *Gels* **7**, 122 (2021)
3. N. Bheekhun, A.R. Abu Talib, M.R. Hassan, *Adv. Mater. Sci. Eng.* **2013**, 1 (2013)
4. U. Berardi, L. Ákos, *Energy Build.* **185**, 307 (2019)
5. A.C. Pierre, *Adv. Sci. Technol.* **65**, 174 (2010)
6. N.A. Zubair, E. Abouzari-Lotf, M. Mahmoud, Nasef, E.C. Abdullah, *E3S Web Conf.* **90**, 01003 (2019)
7. W.C. Hung, R.S. Horng, R.E. Shia, *J. Sol-Gel Sci. Technol.* **97**, 414 (2021)
8. U. Berardi and S (ed.), (Mark) Zaidi, *Energy Build.* **198**, 542 (2019)
9. S.A. Lermontov, V.M. Buznik, N.A. Sipyagina, A.S. Bespalov, A.N. Malkova, D.V. Grashchenkov, A.E. Baranchikov, *J. Porous Mater.* **28**, 1237 (2021)

10. A. Du, B. Zhou, Z. Zhang, J. Shen, *Mater. (Basel)*. **6**, 941 (2013)
11. M.A. Aegerter, N. Leventis, M.M. Koebel (eds.), *Aerogels Handbook*, 1st edn. (Springer New York, NY, New York, NY, 2011)
12. K. Kanamori, M. Aizawa, K. Nakanishi, T. Hanada, *Adv. Mater.* **19**, 1589 (2007)
13. H. Zhang, C. Zhang, W. Ji, X. Wang, Y. Li, W. Tao, *Molecules* **23**, 2198 (2018)
14. C. Luan, S. Movva, K. Wang, X. Yao, C. Zhang, B. Wang, *Funct. Compos. Struct.* **1**, 042002 (2019)
15. K.M. Saoud, S. Saeed, M.F. Bertino, L.S. White, *J. Porous Mater.* **25**, 511 (2018)
16. D.B. Mahadik, H.-N.-R. Jung, Y.K. Lee, K.-Y. Lee, H.-H. Park, *J. Microelectron. Packag Soc.* **23**, 35 (2016)
17. A. Venkateswara Rao, S.D. Bhagat, H. Hirashima, G.M. Pajonk, *J. Coll. Interfac. Sci.* **300**, 279 (2006)
18. I. Şahin, Y. Özbakır, Z. İnönü, Z. Ulker, C. Erkey, *Gels*. **4**(1), 3 (2018)
19. X. Wu, K. Zhong, J. Ding, X. Shen, S. Cui, Y. Zhong, J. Ma, X. Chen, *J. Non Cryst. Solids*. **530**, 119826 (2020)
20. X. Yue, J. Chen, H. Li, Z. Xiao, X. Yu, J. Xiang, *Mater. Chem. Front.* **4**, 2418 (2020)
21. C.A. García-González, M.C. Camino-Rey, M. Alnaief, C. Zetzl, I. Smirnova, *J. Supercrit. Fluids* **66**, 297 (2012)
22. Y. Huang, S. He, G. Chen, H. Dai, B. Yuan, X. Chen, X. Yang, *J. Non Cryst. Solids*. **505**, 286 (2019)
23. T. Zhou, X. Cheng, Y. Pan, C. Li, L. Gong, H. Zhang, *Appl. Surf. Sci.* **437**, 321 (2018)
24. A. Borba, J.P. Vareda, L. Durães, A. Portugal, P.N. Simões, *New. J. Chem.* **41**, 6742 (2017)
25. Y. Li, A. Du, J. Shen, Z. Zhang, G. Wu, B. Zhou, *J. Porous Mater.* **25**, 1229 (2018)
26. L. Wa, L. Fengyun, Z. Fanlu, C. Mengjing, C. Qiang, H. Jue, Z. Weijun, M. Mingwei, *Appl. Surf. Sci.* **353**, 1031 (2015)
27. A. Parakkulam Ramaswamy, A. Rigacci, *Mater. Chem. Phys.* **261**, 124252 (2021)
28. J.P. Vareda, P. Maximiano, L.P. Cunha, A.F. Ferreira, P.N. Simões, L. Durães, *J. Coll. Interfac. Sci.* **512**, 64 (2018)
29. J. He, H. Zhao, X. Li, D. Su, H. Ji, H. Yu, Z. Hu, *Ceram. Int.* **44**, 8742 (2018)
30. S. Jafari, *1* (2018)
31. E. Yalamaç, M. Sütçü, S.B. Baştürk, *Ceramic Fibers* (Woodhead Publishing Series, New Delhi, 2017)
32. A. Tav, Y. Öz, H.I. Akyildiz, *Eur. J. Sci. Technol.* **31**, 309 (2021)
33. M. Di Luigi, L. An, J.N. Armstrong, S. Ren, *Mater. Adv.* **3**, 2726 (2022)
34. H. Huang, Y. Yu, Y. Qing, X. Zhang, J. Cui, H. Wang, *Materials* **13**(2), 447 (2020)
35. Á. Lakatos, A. Trnák, *Energies* **13**(4), 823 (2020)
36. A.A. Trofimov, J. Atchley, S.S. Shrestha, A.O. Desjarlais, H. Wang, *J. Porous Mater.* **27**, 1791 (2020)
37. P. Li, M. Ishiguro, *Soil. Sci. Plant. Nutr.* **62**, 223 (2016)
38. E. Ul Haq, S.F.A. Zaidi, M. Zubair, M.R. Abdul, S.K. Karim, Padmanabhan, A. Licciulli, *Energy Build.* **151**, 494 (2017)
39. K.A. Cychoz, M. Thommes, *Engineering*. **4**, 559 (2018)
40. H. Nikpourian, A.R. Bahramian, *Therm. Sci. Eng. Prog.* **20**, 100751 (2020)
41. K.J. Lee, Y.J. Choe, Y.H. Kim, J.K. Lee, H.J. Hwang, *Ceram. Int.* **44**, 2204 (2018)
42. C. Henry, A.K. Criner, M. Imel, D. King, *AIP Conf. Proc.* **1949**, 140008 (2018)
43. K. Nocentini, P. Achard, P. Biwolé, *Energy Build.* **158**, 14 (2018)
44. A. Darmawan, R. Utari, R.E. Saputra, Suhartana, Y. Astuti, *IOP Conf. Ser. Mater. Sci. Eng.* **299**, 012041 (2018)
45. X. Wu, S. Huang, Y. Zhang, L. Shi, Y. Luo, X. Deng, Q. Liu, Z. Li, *J. Nanoparticle Res.* **22**, 1 (2020)
46. Z. Li, Y. Zhang, S. Huang, X. Wu, L. Shi, Q. Liu, *J. Nanoparticle Res.* **22**, 1 (2020)
47. S. Huang, X. Wu, Z. Li, L. Shi, Y. Zhang, Q. Liu, *J. Porous Mater.* **27**, 1241 (2020)
48. A. Lamy-Mendes, A.V. Girão, R.F. Silva, L. Durães, *Microporous Mesoporous Mater.* **288**, 109575 (2019)
49. P. Bazylewski, R. Divigalpitiya, G. Fanchini, *RSC Adv.* **7**, 2964 (2017)
50. C.Y. Kim, A. Rum Jang, *Asian J. Chem.* **24**, 4217 (2012)
51. K. Igarashi, K. Tajiri, Y. Tai, S. Tanemura, *Z. Für Phys D Atoms Mol Clust.* **26**, 207 (1993)
52. C.R. Ehgartner, S. Grandl, A. Feinle, N. Hüsing, *Dalt Trans.* **46**, 8809 (2017)
53. G.E. Walrafen, M.S. Hokmabadi, N.C. Holmes, W.J. Nellis, S. Henning, *J. Chem. Phys.* **82**, 2472 (1985)
54. J.P. Vareda, C.A. García-González, A.J.M. Valente, R. Simón-Vázquez, M. Stipetic, L. Durães, *Environ. Sci. Nano.* **8**, 1177 (2021)
55. S. Pohl, G. Kickelbick, *J. Sol-Gel Sci. Technol.* **107**, 329 (2023)
56. M. Cazacu, C. Racles, M.F. Zaltariov, M. Dascalu, A. Bele, C. Tugui, A. Bargan, G. Stiubianu, *Polym. (Basel)* **13**(10), 1605 (2021)
57. H. Yang, F. Ye, *RSC Adv.* **12**, 12226 (2022)
58. S. Yun, T. Guo, J. Zhang, L. He, Y. Li, H. Li, X. Zhu, Y. Gao, *J. Sol-Gel Sci. Technol.* **83**, 53 (2017)
59. I.A. Rahman, P. Vejayakumaran, C.S. Sipaut, J. Ismail, C.K. Chee, *Mater. Chem. Phys.* **114**, 328 (2009)
60. N. Minju, B.N. Nair, S. Savithri, *RSC Adv.* **11**, 15301 (2021)
61. S. Chakraborty, A.A. Pisal, V.K. Kothari, A. Venkateswara Rao, *Adv. Mater. Sci. Eng.* 2016(2), 1–8 (2016)
62. S. He, G. Sun, X. Cheng, H. Dai, X. Chen, *Compos. Sci. Technol.* **146**, 91 (2017)
63. Y. Huang, S. He, G. Chen, X. Shi, X. Yang, H. Dai, X. Chen, *J. Sol-Gel Sci. Technol.* **88**, 129 (2018)
64. Á. Lakatos, A. Csík, I. Csarnovics, *Case Stud. Therm. Eng.* **25**, 100966 (2021)
65. B. Merillas, J.P. Vareda, J. Martín-De, M. León Á., Rodríguez-Pérez, L. Durães, *Polymers (Basel)*. **14**(13), 2556 (2022)
66. Y.S. Park, J. Choi, B.S. Kim, S.H. Baeck, S.E. Shim, Y. Qian, *J. Therm. Anal. Calorim.* **148**, 7623 (2023)
67. P.C. Thapliyal, K. Singh, *J. Mater.* **2014**, 1 (2014)
68. G. Camino, S.M. Lomakin, M. Lageard, *Polym. (Guildf)*. **43**, 2011 (2002)
69. D. Gao, M. Jia, *High. Perform. Polym.* **24**, 546 (2012)
70. X. Liu, H. Sheng, S. He, C. Du, Y. Ma, C. Ruan, C. He, H. Dai, Y. Huang, Y. Pan, *Chin. J Chem Eng.* **58**, 266–281 (2022)
71. C. Hamciuc, G. Lisa, E. Hamciuc, N. Tudorachi, *J. Anal. Appl. Pyrolysis.* **129**, 204 (2018)
72. B. Nowak, M. Bonora, M. Zuzga, L. Werner, A. Jackiewicz-Zagórska, J.M. Gac, *J. Environ. Chem. Eng.* **10**, 108410 (2022)
73. S.J. McNeil, H. Gupta, *Polym. Test.* **106**, 107426 (2022)

Publisher's Note Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Springer Nature or its licensor (e.g. a society or other partner) holds exclusive rights to this article under a publishing agreement with the author(s) or other rightsholder(s); author self-archiving of the accepted manuscript version of this article is solely governed by the terms of such publishing agreement and applicable law.