



Super-hydrophobic blended needle-punched nonwovens integrated with silica-aerogels for PM_{2.5} filtration

Hanur Meku Yesuf^{1,2}, Syed Rashedul Islam^{1,3,4}, Xueping Zhang^{1†}, and Xiaohong Qin^{1†}

¹Key Laboratory of Textile Science & Technology, Ministry of Education, College of Textiles, Donghua University, Shanghai 201620, China

²Textile Engineering, Ethiopian Institute of Textile and Fashion Technology, Bahir Dar University, P.O. Box 1037, Bahir Dar, Ethiopia

³Department of Textile Engineering, Uttara University, Dhaka 1230, Bangladesh

⁴Department of Textile Engineering, Apparel Manufacture and Technology, BGMEA University of Fashion and Technology, Dhaka 1350, Bangladesh

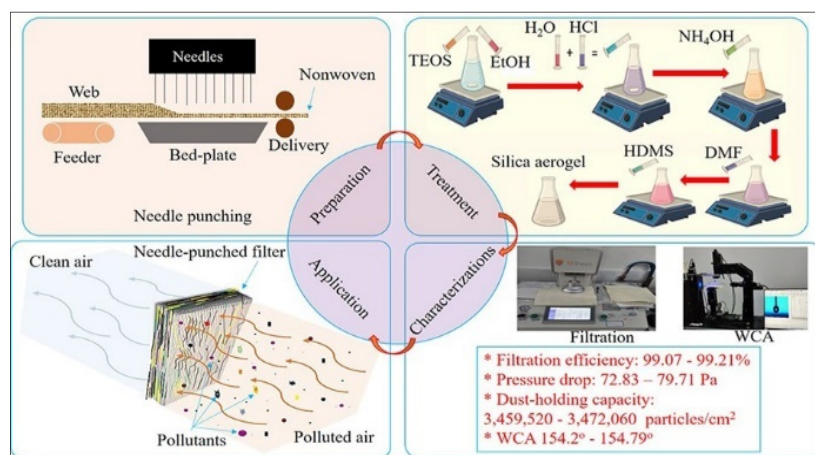
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ABSTRACT

This study investigated the hydrophobic and PM_{2.5} filtration properties of blended needle-punched nonwovens with the treatment of silica-aerogel. The treatment was done by padding. The BET, SEM and FTIR studies were used to investigate the influence of silica-aerogel on needle-punched nonwovens such as surface properties and molecular interaction. All the treated samples showed super-hydrophobic properties with water contact angle (WCA) of around 154.2 - 154.79°, filtration efficiency of 99.07 - 99.21%, pressure drop of 72.83 - 79.71 Pa and dust-holding capacity of 3,459,520 - 3,472,060 particles/cm². The findings demonstrated that silica-aerogel played a vital role in enhancing the hydrophobicity, filtration efficiency, dust-holding capacity and fabric density of blended needle-punched nonwovens with statistically significant performance ($p < 0.05$). There was no statistically significant difference in pressure drop, air permeability, porosity, and fabric thickness, which indicated that the treated samples could perform without structure loss as compared to the untreated.

Keywords: Air filtration, Blending, Needle-punched nonwovens, Silica-aerogel, Super-hydrophobic

Graphical Abstract



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[†] Corresponding author

E-mail: xhqin@dhu.edu.cn (X.Q.), xpzhang@dhu.edu.cn (X.Z.)

Tel: +86-21-67792702 (X.Q.), +86-21-67792601 (X.Z.)

ORCID: 0000-0003-4663-903X (X.Q.), 0000-0003-1873-876X (X.Z.)

1. Introduction

Air pollution has become a world serious issue because of its bad impact on the environment and public health. Fine particulate matter, especially $PM_{2.5}$ (fine particulate matter with aerodynamic diameter less than $2.5\ \mu m$), is a mix of harmful components (sulfate, nitrate, carbon) that enter the bloodstream and contribute to millions of premature deaths worldwide [1-7]. Around a total of 7 million people deaths per year have been estimated and declared due to ambient air pollution by the World Health Organization (WHO) [8]. Air filters are essential for improving indoor air quality by capturing particulate matter like dust, pollen, mold spores, and harmful fine particles such as $PM_{2.5}$, ensuring a healthier living environment in residential, commercial, industrial, and automobile spaces [9-12].

Key performance characteristics that air filters should possess include filtration efficiency, pressure drop, dust-holding capacity stability and durability, resistance to moisture and chemicals, and self-cleaning [10-14]. High filtration efficiency is essential for capturing fine particulate matter like $PM_{2.5}$, improving air quality, while stability, durability, and resistance to moisture or chemicals ensure long-term performance, reducing the need for frequent replacements. A low-pressure drop, high dust-holding capacity, and self-cleaning properties help to maintain airflow efficiency and minimize maintenance in filtration systems. These characteristics ensure air filters deliver effective, long-term solutions for maintaining indoor air quality in a cost-effective and energy-efficient manner.

The use of textile filter material in air filtration is critical for pollution management [5, 15-17]. Nonwoven fabrics are a vital component in various industrial applications, including filtration, due to their unique structure and properties. Among the many methods of producing nonwoven fabrics, needle-punching is particularly notable for its ability [18-23]. Hydrophobic filters can improve water-based particle capture in humid air while maintaining filtration efficiency due to minimal pressure drop and reduced clogging by moisture.

A superhydrophobic surface, characterized by a water contact angle greater than 150° and a rolling angle less than 10° , is achieved through micro-nano rough structures and low surface energy substances, applicable to various substrates including metals, inorganics, and textiles [24-27]. Jinde et al. studied embedding nonwovens fabrics with nanoporous amorphous silica and offered promising avenues for the development of innovative, high-performance fabrics with enhanced functional properties [28]. Talebi et al. studied addition of nanoporous amorphous silica to the nonwoven induced significant changes in several key properties of the fabric like fabric thickness, density, porosity and hydrophobicity [29]. Silica-aerogel treatment can effectively modify the hydrophobicity of needle-punched nonwoven fabrics. Yang et al. showed in their study that the addition of hexamethyldisilazane (HMDS) into silica sols during the sol-gel process can enhance the hydrophobic properties of coatings, increasing the contact angle for water [30]. Przybylak et al. investigated the fabrication of superhydrophobic cotton fabrics through a simple chemical modification, measuring the water contact angle by drop profile tensiometry and examining surface topography changes with scanning

electron microscopy, demonstrating that the fabrics maintained their superhydrophobic properties even after multiple washings [31].

Hao et al. highlighted the growing potential of silica nanoparticles as versatile modifiers for enhancing the crystalline properties, strength, and fluorescent capabilities of high-performance polyester composites [32]. The impregnation of silica-aerogel onto polyester fabric had a significant impact on its wettability and soil release properties. Altay et al. showed that aerogel impregnated fabrics provided superhydrophobic properties with a contact angle of around 170° , indicating excellent water repellency [33]. The findings of the research on synthesis of water-repellent coating for polyester fabric approved that the sol-gel method offered advantages such as uniform coverage, adhesion to the substrate, and the ability to tailor surface properties like hydrophobicity and resulted in a water contact angle of 136.2° [34]. Hu et al. enhanced resistance to moisture, thermal insulation performance, and durability of SiAG/NNW composite material at high temperatures for applications requiring both hydrophobicity and thermal insulation properties by combining the hydrophobic and thermal properties of silica-aerogel with the PPS needled nonwoven [35].

Arat et al. achieved integration of hydrophobic silica-aerogel with polyacrylonitrile (PAN) nanofibers by synthesizing the aerogel and incorporating it into the nanofiber structure, resulting in increased surface roughness and hydrophobicity with a water contact angle of 142° [36]. Lu et al. developed the self-assembled silica aerogel-coated polylactic acid membrane, with its specific molecular arrangement, effectively separates water-in-oil emulsions by selectively allowing the passage of one phase while blocking the other based on differences in hydrophilicity and pore size [37]. Another study synthesized a desirable silica aerogel/polypropylene composite via a two-step sol-gel process by immersing a PP nonwoven fabric in silica sol [38].

In this research, polylactic acid (PLA), polyphenylene sulfide (PPS), polyacrylonitrile (PAN), polypropylene (PP), and polyester (PET) fibers were blended in equal proportions to produce needle-punched nonwoven samples. Each fiber was chosen for its unique properties such as PAN for its high strength and thermal stability, PET for its durability and resistance to various chemicals, PLA for its biodegradability, PP for its lightweight and hydrophobic nature, and PPS for its excellent chemical resistance and thermal stability. The combination of these fibers aimed to leverage their individual strengths to create a robust and versatile nonwoven fabric. To further enhance the functionality of these nonwoven samples, a silica-aerogel (SiGel) treatment was applied. This study particularly focused on evaluating the changes in dust-holding capacity, filtration efficiency, pressure drop, and water contact angle before and after the silica-aerogel treatment. These properties were crucial for applications such as air filtration, where material performance was directly related to efficiency and durability. By analyzing these properties before and after the silica-aerogel treatment, this study aimed to understand how the addition of silica-aerogel influences the performance of needle-punched nonwoven fabrics. The results of this investigation provided valuable insights into the potential of these materials for advanced filtration applications, highlighting the benefits of combining different fiber types and functional treatments to achieve desired properties.

2. Experimental Work

2.1. Materials

Three kinds of needle-punched nonwoven samples with different web arrangements and pre-needling (N0 with cross-laid web arrangement, N1 with parallel-laid web arrangement and N2 with parallel-laid pre-needling web arrangement) were prepared at Donghua University, Shanghai, China. All samples are made from the blend of polylactic acid (PLA), polyphenylene sulfide (PPS), polyacrylonitrile (PAN), polypropylene (PP), and polyester (PET) fibers. The comprehensive details were presented in our previous publication [22, 23]. Tetraethylorthosilicate (TEOS >99%), ethyl alcohol (EtOH $\geq$ 99.5%) and N, N-dimethyl-formamide (DMF $\geq$ 99.0%) were purchased from Shanghai Mindel Biochemical Technology Co.Ltd, Sinopharm Chemical Reagent Co.Ltd, and MACKLIN, respectively. Hydrochloric acid (36-38%), ammonium hydroxide (NH₄OH 25%) and hexamethyldisilazane (HMDS $\geq$ 99%) were purchased from Shanghai Titan Technology Co. Ltd. All the chemicals and reagents were applied without any further rectification.

2.2. Sample Preparation

To create the silica-hydrogel via a two-stage sol-gel process [39] (detailed in Fig. 1), TEOS was first hydrolyzed in acidified ethanol (stage 1). 111.1 ml of TEOS was dissolved in 694.4 ml of ethanol and stirred for 30 minutes. Then, an acid solution (27.8 ml HCl in 83.3 ml water) was slowly added while stirring at 60°C for 60 minutes. Stage 2 involved gelation and pore formation. Next, 27.8 ml of NH₄OH was added and stirred for 30 minutes. 27.8 ml of DMF was then added to create nanopores, followed by the introduction of 27.8 ml of HMDS and a final stirring for 120 minutes at 60°C, resulting in the formation of silica-hydrogel.

The needle-punched nonwoven samples were first equilibrated at 20°C and 65% relative humidity for 24 hours (as detailed in Fig. 1) [40]. Then, 35 cm $\times$ 35 cm squares were cut and fully submerged in silica-hydrogel until saturated. Excess gel was removed by hanging the samples for 8-10 minutes. To ensure proper integration with the fabric structure, the wet-gelled samples were held at 25°C for an additional 24 hours [41]. Finally, to transform into silica aerogel and achieve better fixation with the samples, a two-step drying process was applied: first, drying at 50°C for 5 hours, followed by curing at 100°C for an additional 5 hours. Silica-aerogel modified samples are coded N0-SiGel, N1-SiGel and N2-SiGel to represent samples after treatment.

Reproducibility of the silica-aerogel preparation process can be a challenging aspect in ensuring consistent quality. Several factors may affect the uniformity of the aerogels, leading to variability in their performance. The silica-aerogel preparation process requires careful control of factors like pH, catalyst concentration, and gelation rate, as minor deviations can cause inconsistencies in pore size, density, and mechanical properties. Additionally, variations during solvent exchange and supercritical drying can affect the aerogel's texture and porosity, while batch-to-batch variability can arise in large-scale production due to differences in raw material quality and processing conditions. To improve reproducibility, refining each step of the process and employing precise control systems is essential for minimizing variability and ensuring consistent, high-quality silica-aerogels.

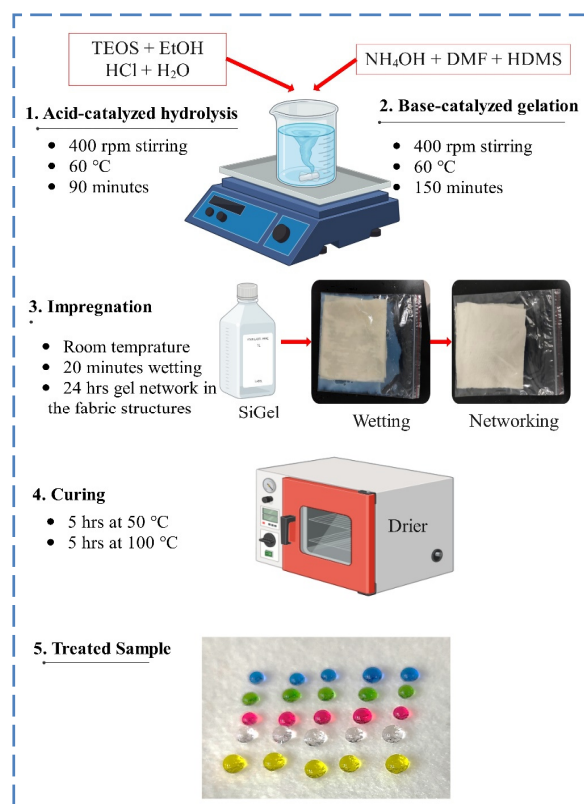


Fig. 1. Preparation and integration of silica-aerogel with needle-punched nonwoven samples.

2.3. Characterization

2.3.1. Standard test methods

The test method and the experimental procedures were performed based on the standard test method. Before the test, all samples were conditioned under a standard atmosphere at a temperature of $20 \pm 2^\circ\text{C}$ and a relative humidity of 65% for 24 h.

2.3.2. SEM analysis

A Hitachi Flex-SEM (scanning electron microscopy) 1000 (SU1000) was used to examine the morphology of the needle-punched fabric fibers. To enhance image quality, all samples were sputter-coated with a thin layer of gold before analysis.

2.3.3. FTIR test

The American Nicolet iNTM 5700 FT-IR (Fourier Transform Infrared) spectrometer was used to analyze the chemical composition and confirm the uniformity of fiber distribution within the nonwoven samples. The analysis employed Attenuated Total Reflection (ATR) to obtain spectra across a wavenumber range of $400\text{--}4000\text{ cm}^{-1}$.

2.3.4. Thickness test

The thickness of the nonwoven samples was measured according to the established standards GB/T 3820-1997 and ISO 5084:1996. A high-precision thickness gauge was used, capable of measuring the distance between the presser foot and a reference plate with an accuracy of 0.01 mm.

2.3.5. GSM test

Following ASTM D6242 standards, the needle-punched nonwoven fabric's gram per square meter (GSM) was determined. This involved cutting ten 10 cm × 10 cm samples from various locations and measuring their mass using an electronic balance with 0.001 g accuracy. The final GSM value was obtained by averaging the ten measurements.

2.3.6. Density test

Nonwoven fabric density, or bulk density, is the weight per unit volume of the nonwoven fabric (kg/m³). It is determined by the following Eq. (1):

$$D = \frac{\text{GSM}}{T} \quad (1)$$

where D is the fabric density (Kg/m³), GSM is gram per square meter, and T is the thickness (mm).

2.3.7. BET specific surface test

Nitrogen gas adsorption measurements, employing the Brunauer-Emmett-Teller (BET) method, were performed to determine the specific surface area, pore diameter, and pore volume of the samples [42-47]. The specific surface area of the needle-punched nonwoven samples was determined using N₂-adsorption experiments on an ASAP2010 instrument (Micromeritics, USA). Before testing, the samples were degassed at 150°C.

Porosity test

Porosity was determined by gravimetric analysis. Dry samples were weighed and then immersed in water for 24 hours. After removing excess water from the sample surface, they were weighed again. The porosity, expressed as a percentage, was calculated using Eq. (2) [39]. This equation represents the ratio of the water absorbed (difference between wet and dry weight) to the initial dry weight of the sample, averaged over ten measurements:

$$P = \frac{W_w - W_d}{W_d} \times 100 \quad (2)$$

where P is the porosity ratio in %, W_w is the wet weight in g, and W_d is the dry weight in g.

2.3.8. Air permeability test

The air permeability test, as per the relevant provisions of ASTM D737-96, determined the velocity of airflow passing perpendicularly through a test specimen under specific conditions. The test was carried out using YG461E automatic air permeability tester, with the test surface area set at 20 cm². A pressure drop of 200 Pa was applied across the fabric test area during the test.

2.3.9. Filtration efficiency, pressure drop and dust-holding capacity

Air filtration performance was evaluated using a U-Test automatic digital tester (Model: F003, Uti Intelligent Technology Co., Ltd., Suzhou, China) following the ISO 29463 assessment standard. The tester introduced sodium chloride (NaCl) particles with a size range of PM_{2.5} (aerodynamic diameter ≤ 2.5 μm) into the filtration system at a constant airflow rate of 32 L/min, simulating dust exposure. Tests were conducted under controlled conditions of 20°C and 45% relative humidity. Fabric moisture content, follow-

ing ISO 939, was determined before testing. Filtration efficiency and dust-holding capacity were then calculated using Eq. (3) and (4) [22, 23, 48]:

$$F_e = \frac{D_c}{D_f} \times 100 \quad (3)$$

$$D_h = \frac{D_c \times D_f}{F_A} \times 100 \quad (4)$$

where Fe is filtration efficiency (%), Dh is dust-holding capacity (particles/m²), Dc is dust collected, Df is dust fed and FA is filter area (m²).

2.3.10. Water contact angle test

An optical video contact angle instrument (Model KINO SL200KS) was used to determine the water contact angle of the fabric samples. A 10 ml water droplet was dispensed onto each sample, and the contact angle was measured over 180 seconds to capture any dynamic changes. The average contact angle for each sample was calculated based on measurements from five different areas. All testing was conducted at a controlled temperature of 20 ± 2°C.

2.3.11. Silica-aerogel add-on percentage

Following drying at 107°C for 60 minutes, both treated and untreated needle-punched nonwoven samples were analyzed to determine the percentage of silica-aerogel added (add-on %). This calculation was performed using Eq. (5) [49]:

$$A = \frac{W_t - W_u}{W_u} \times 100 \quad (5)$$

where A is add-on percentage, W_u is the weight of untreated samples in g, and W_t is the weight of treated samples in g.

2.3.12. Statistical analysis

The statistical methods employed for analyzing the data, specifically ANOVA and Tukey tests, were crucial in determining the significant differences between untreated and treated samples across various properties (Fig. 6 and Table 2). ANOVA and Tukey tests depended on several key assumptions to ensure valid results. Firstly, normality was crucial, as ANOVA assumed that the data from each group was normally distributed, especially when comparing means across multiple groups. Validation of this assumption was carried out using graphical methods by histograms. Secondly, homogeneity of variances was another essential assumption, which means that the variance within each group should be approximately equal. Levene's test is typically employed to assess this equality. These necessary assumptions of normality and homogeneity of variances were validated using appropriate tests confirming that the data met the requirements for ANOVA.

3. Results and Discussion

3.1. Silica-Aerogel Add-on Percentage

Scanning electron microscopy (SEM) Shows how the silica-aerogel bonded with the structure of the treated needle-punched nonwoven samples as indicated by surface morphological view (Fig. 2: a,

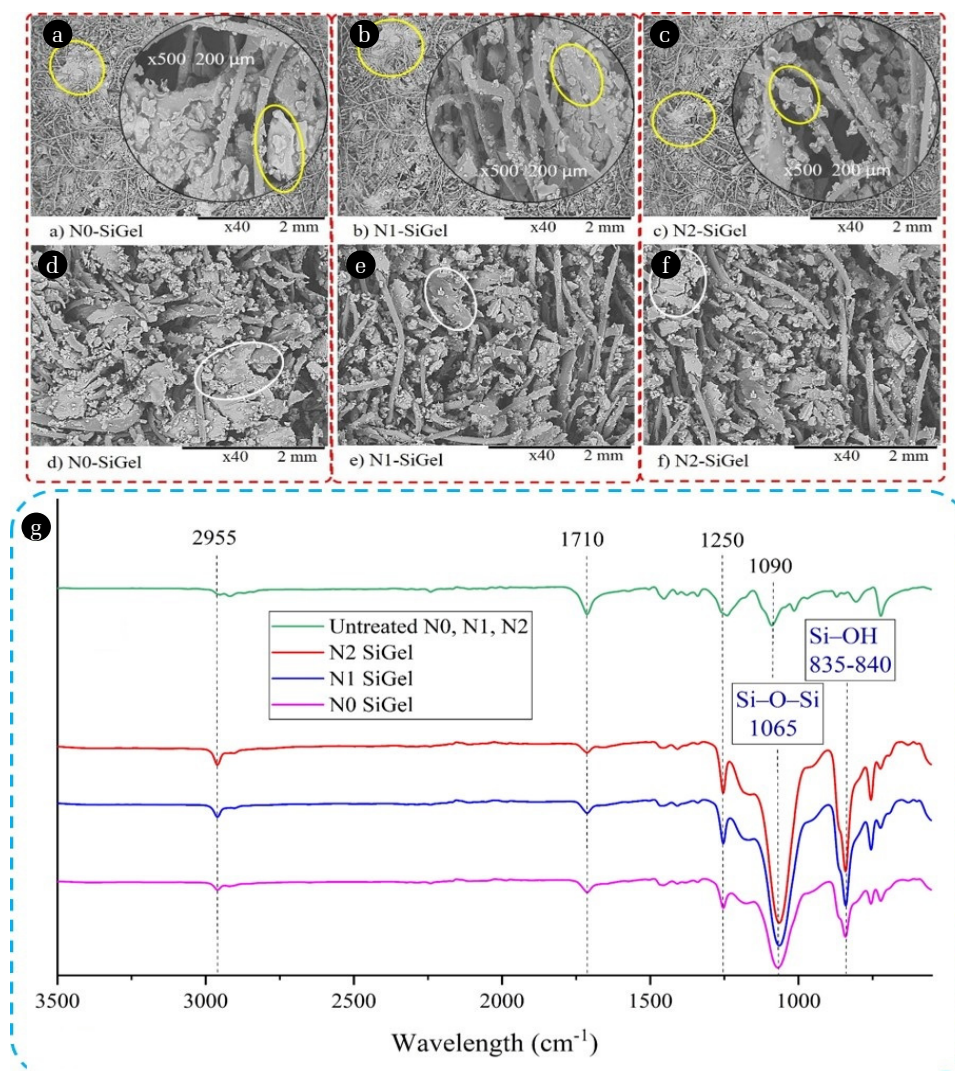


Fig. 2. SEM images of silica-aerogel treated needle-punched nonwoven samples; surface views (a, b, c) and cross-sectional views (d, e, f); FTIR graphs of untreated and silica-aerogel treated needle-punched nonwoven samples (g).

b, c) and cross-sectional view (Fig. 2: d, e, f). The bonding between the fibers and silica-aerogel in the treated samples, denoted as N0-SiGel, N1-SiGel, and N2-SiGel, can occur through various chemical interactions. Silica-aerogel, a highly porous form of silicon dioxide, possesses hydroxyl (OH) groups on silanol groups (Si-OH) present on the surface of silica-aerogel, making it capable of forming hydrogen bonds with polar functional groups (hydroxyl, carbonyl, -NH, cyano groups (-CN)) present in the fibers (PAN, PET, PLA). PP and PPS fibers, being less polar, primarily interact with silica-aerogel through van der Waals forces and physical entanglement rather than strong chemical bonds. However, some levels of adhesion can still occur due to the surface roughness and porosity of silica-aerogel, enhancing mechanical interlocking between the fibers and the gel. Although individually weak, Van der Waals forces can collectively contribute to adhesion between fibers and the silica-aerogel. These forces are weak intermolecular forces that arise from temporary fluctuations in electron distribution.

The add-on percentage of the silica-aerogel on N0-SiGel, N1-SiGel and N2-SiGel is calculated based on the Eq. (5), and resulted 22.51%, 27.71% and 32.60% respectively. The higher amount of add-on percentage in the case of N2-SiGel is due to its lower density and higher porosity. Overall, the bonding mechanism between the fibers and silica-aerogel involves a combination of hydrogen bonding, coordination interactions, and physical entanglement, contributing to improved adhesion and bonding strength in the treated nonwoven samples.

3.2. Effect of Silica-Aerogel Coating on the Molecular Structure

In the FTIR (Fourier Transform Infrared) Spectroscopy spectrum of silica-aerogel compounds, the Si-O-Si stretching vibrations are typically observed at wavelengths around 1000 to 1100 cm^{-1} [50]. This range corresponds to the stretching vibrations of the silicon-oxygen-silicon (Si-O-Si) bonds in the silica-aerogel structure. Si-OH stretching vibration is also usually observed in the range

of 800 to 950 cm^{-1} . The existence of Si-O-Si and Si-OH network constructions in the treated samples induces constant material distribution on the fabric surface, resulting in increased surface area (Fig. 3 (d)), thus, enhancing the hydrophobic properties of the treated samples [42, 51, 52]. These vibrations are characteristic peaks in the FTIR spectrum and provide information about the chemical bonds present in the silica-aerogel compound. Fig. 2 (g) presents the FTIR analysis results, highlighting the presence of specific functional groups in all fabric samples. An absorption peak around 1050 cm^{-1} indicates the stretching and bending of C-O bonds, which is observed in both untreated and treated samples.

The peaks at 1090 cm^{-1} in untreated samples were attributed to S-C₆H₄-S's C-S bond stretching [22]. However, after the silica-aerogel treatment, the peak at 1090 cm^{-1} is modified, becoming a high sharp peak. This is because silica-aerogel treatment facilitates the condensation reaction between the silanol (Si-OH) groups from the silica-aerogel and the hydroxyl groups from the polymers, leading to the creation of Si-O-Si bonds at 1065 cm^{-1} . A new peak at 835 to 840 cm^{-1} is present only in the treated samples and is attributed to the stretching vibration of Si-OH bonds. This confirms the successful formation of a nanoporous silica network structure on the treated samples [53]. The absence of this peak in the untreated samples differentiates their spectra at this specific wavenumber. The presence of Si-O-Si bonds in the treated samples' spectra is linked to their improved hydrophobic properties, as reported elsewhere [54]. The reduction of certain peaks (e.g., around 1710 cm^{-1}) in the treated samples suggests chemical modifications consistent with the hydrophobization process using hexamethyldisilazane. This process replaces hydrophilic groups with hydrophobic groups, enhancing the water-repellent properties

of the material. Absorption bands near at 1250 cm^{-1} and 2955 cm^{-1} correspond to the stretching and bending of C-H bonds.

3.3. BET Specific Surface Area (SSA) of Needle-Punched Nonwoven Samples

The BET results provided in Fig. 3 (d) include data on the specific surface area, pore diameter, and total pore volume for the untreated and silica-aerogel treated samples. The significant increase in specific surface area upon silica-aerogel treatment suggests more available surface area for particle capture. Specifically, for N0 to N0-SiGel, the specific surface area increases from 2.333 to 4.200 m^2/g ; for N1 to N1-SiGel, it rises from 2.230 to 3.969 m^2/g ; and for N2 to N2-SiGel, it enhances from 2.066 to 3.801 m^2/g . This enhancement is critical for PM_{2.5} air filtration as it improves the material's ability to trap fine particles, leading to higher filtration efficiency. A larger surface area also contributes to better adsorption of contaminants. The reduction in pore diameter upon treatment indicates a more compact structure. Specifically, for N0 to N0-SiGel, the pore diameter decreases from 3.773 to 3.019 nm; for N1 to N1-SiGel, it decreases from 4.194 to 3.271 nm; and for N2 to N2-SiGel, it decreases from 4.453 to 3.740 nm. Smaller pores enhance the filtration efficiency by providing a tighter mesh for trapping smaller particles, including PM_{2.5}.

The isotherm graphs in Fig. 3 (a, b and c) show N₂ adsorption-desorption isotherm curves of samples with a hysteresis loop at $P/P_0 > 0.5$. The isotherm exhibits characteristics of type IV hysteresis. The narrow, lower-pressure region suggests an open pore structure, while the hysteresis loop at higher-pressure regions indicates the presence of macropores. Therefore, it can be con-

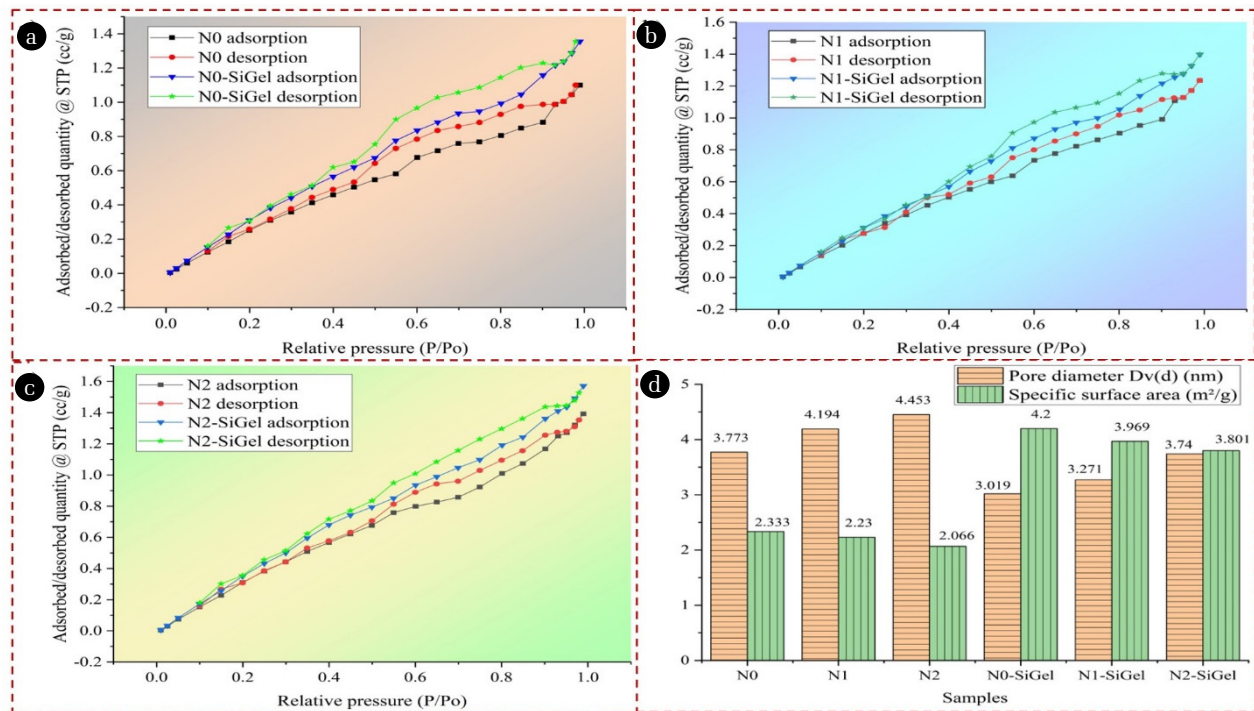


Fig. 3. N₂ adsorption-desorption isotherms of treated and untreated samples; a) Samples N0 and N0-SiGel, b) Samples N1 and N1-SiGel, c) Samples N₂ and N2-SiGel, d) Pore volume, pore size and specific surface area of Samples.

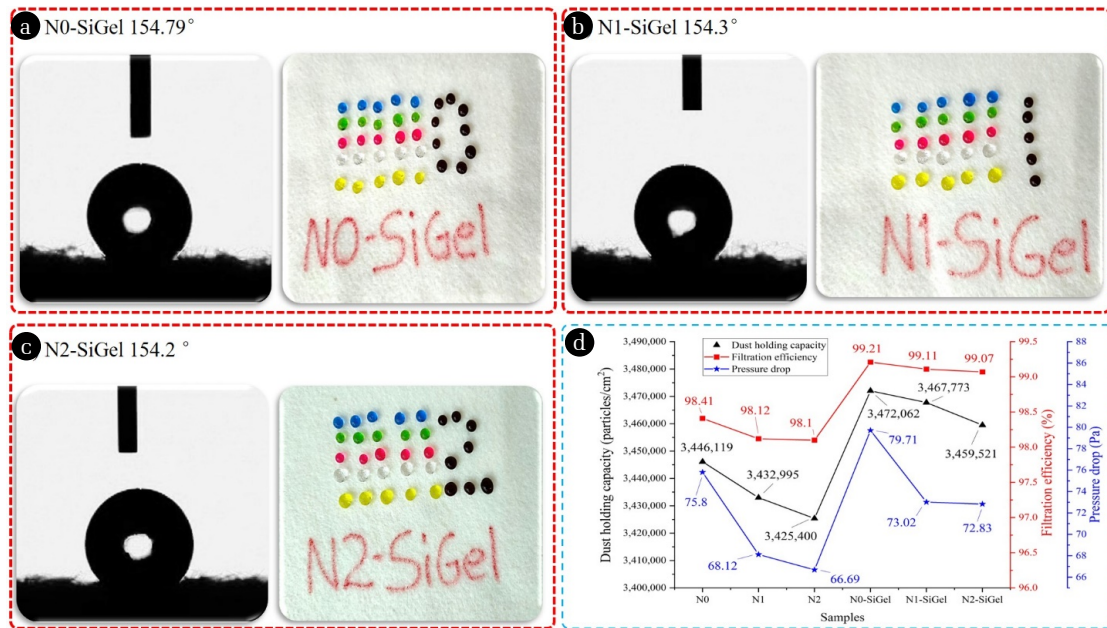


Fig. 4. Water contact angle and water droplets on the surfaces of silica-aerogel treated needle-punched nonwoven samples (a-c); dust-holding capacity, filtration efficiency and pressure drop of Silica-aerogel treated needle-punched nonwoven samples (d).

cluded that the samples are mesoporous [44-47]. For the silica-aerogel treated samples, there is an increase in the adsorbed quantity at STP across various pressures. This behaviour indicates enhanced adsorption capacity. The slight increase in total pore volume suggests that while the pores are becoming smaller, the overall volume available for capturing particles is maintained or slightly increased. This balance is crucial for maintaining filtration efficiency without drastically increasing the pressure drop. The combined effects of increased specific surface area, decreased pore diameter, and stable total pore volume suggest that silica-aerogel treatment enhances the filtration efficiency of the nonwoven fabrics. The increased surface area and reduced pore size create a more effective barrier against PM_{2.5} particles.

3.4. Effect of Silica-Aerogel Treatment on Thickness, GSM, Density, Porosity and Air Permeability

The addition of silica-aerogel to the nonwoven samples, denoted as N0-SiGel, N1-SiGel, and N2-SiGel, induces significant changes

in several key properties of the fabric as indicated by surface morphological view (Fig. 2: a, b, c) and cross-sectional view (Fig. 2: d, e, f). Firstly, silica-aerogel treatment leads to a slight decrease in fabric thickness across all samples (-0.74%, -0.55% and -1.45% respectively), indicating a compression of protruding fibers within the fabric structure. This compression effect is accompanied by a substantial increase in fabric weight (22.52%, 27.72% and 32.61% respectively), as silica-aerogel adds mass to the fabric, resulting in denser and heavier materials.

Consequently, the fabric density increases (24.11%, 29.28%, 35.18% respectively) proportionally with the addition of silica-aerogel, reflecting the tighter packing of fibers within the fabric structure. Simultaneously, the porosity of the fabric increases (2.23%, 1.32% and 1.29% respectively), indicating an increment in pore volume due to the incorporation of silica-aerogel, which itself is porous material [42, 51, 52]. Air permeability decreases (-3.45%, -3.01% and -8.09% respectively) insignificantly in N0-SiGel and N1-SiGel, indicating reduced airflow through the fabric due to the decrease in pore diameters.

Table 1. Density, porosity, and air permeability of untreated and treated needle-punched nonwoven samples

Sample	Fabric Thickness (mm)	Fabric weight (GSM)	Fabric density (Kg m ⁻³)	Porosity (%)	Air permeability (m ³ /m ² /hr)	Silica-aerogel Add-on %
N0	3.85	467.36	121.34	89.99	1722.25	22.51
N0-SiGel	3.82	572.61	150.60	92.00	1662.8	
Change in %	-0.74	22.52	24.11	2.23	-3.45	
N1	4.17	379.29	90.96	92.37	1784.61	27.71
N1-SiGel	4.15	484.45	117.59	93.59	1730.96	
Change in %	-0.55	27.72	29.28	1.32	-3.01	
N2	4.63	386.52	83.40	93.12	1830.26	32.60
N2-SiGel	4.57	512.55	112.75	94.32	1682.22	
Change in %	-1.45	32.61	35.18	1.29	-8.09	

These changes collectively contribute to enhancing the fabric's effectiveness in PM_{2.5} filtration. Decreased porosity and increased fabric density restrict the passage of fine particles, improving filtration efficiency. Additionally, reduced air permeability ensures that air passing through the fabric is more thoroughly filtered, minimizing the likelihood of PM_{2.5} particles bypassing the filtration system. Therefore, the effects induced by silica-aerogel treatment on fabric properties play a crucial role in enhancing PM_{2.5} filtration performance, making the nonwoven samples more suitable for air filtration applications in environments where particulate matter pollution is a concern.

3.5. Effect of Silica-Aerogel Treatment on Water Contact Angle

The silica-aerogel treatment significantly enhanced the superhydrophobic properties of the needle-punched nonwoven samples N0, N1, and N2, as indicated in Fig. 4 (a, b and c) by the substantial increase in their water contact angles (WCA). The untreated nonwoven fabrics were already somewhat hydrophobic. However, after treating the samples with silica-aerogel, there was a significant increase in the WCA of N0-SiGel 154.79°, N1-SiGel 154.3° and N2-SiGel 154.2°, representing a 22.1%, 22.07% and 21.84%, respectively. These increases elevated the samples into the superhydrophobic range, generally defined as having a WCA greater than 150° [24-27]. The enhancement in hydrophobicity could be attributed to the porous structure of silica-aerogel, which provided additional surface modifications and increased the surface area (Fig. 3 (d)), thereby reducing the adhesion of water molecules [29, 42, 43, 51, 52]. These increased surface modifications, combined with the inherent hydrophobicity of the samples, resulted in a characteristic feature of superhydrophobic surfaces.

The varying add-on percentages of silica-aerogel correlated with the increase in WCA. The consistent improvement across all samples suggested that the treatment was uniformly effective, regardless of the web arrangement or pre-needling process. The slight variations in percentage changes indicated a robust enhancement in hydrophobicity without significant dependence on the specific initial structure of the nonwoven fabric. This improvement was highly beneficial for applications, requiring superhydrophobic surfaces such as water-resistant textiles, filtration systems, and protective clothing, where preventing water absorption was crucial [42, 51, 52].

3.6. Effect of Silica-Aerogel Treatment on Filtration, Pressure Drop and Dust-Holding Capacity

The dust-holding capacity, filtration efficiency, and pressure drop of needle-punched nonwoven samples are shown in Fig. 4 (d). The treatment with silica-aerogel resulted in a 0.75% to 1.01% increase in dust-holding capacity for all samples, indicating a positive correlation with the add-on percentage of silica-aerogel. This increase can be attributed to the porous structure of silica-aerogel, providing additional surface area for dust particles to adhere to. Similarly, there was an improvement (0.8% to 1.01%) in filtration efficiency across all samples, likely due to the finer pore size created by the silica-aerogel, enhancing particle capture efficiency. However, the silica-aerogel treatment caused an increase (5.16% to 9.21%) in pressure drop for all samples, because the added silica-aerogel restricts airflow through the filter media. This increase in pressure drop corresponded to the increased resistance

to airflow caused by the additional material present in the samples. Overall, while silica-aerogel treatment led to improvements in dust-holding capacity and filtration efficiency, it also resulted in increased pressure drop due to the increment in fabric density, coupled with a decrement in porosity.

3.7. Relationship between Silica-Aerogel Add-on Percentage Vs. Water Contact Angle

A linear fit analysis has been conducted to examine the relationship between the silica-aerogel add-on percentage and the water contact angle of needle-punched nonwoven samples. The resulting linear Eq. (6) is presented in Fig. 5 (a). The substantial increase in the water contact angle suggests that the addition of silica-aerogel significantly enhances the hydrophobicity of the nonwoven samples. The higher the water contact angle, the more hydrophobic the surface becomes, indicating improved water repellency. The R² value of 0.95 signifies that the linear model explains 95% of the variability in the water contact angle based on the silica-aerogel add-on percentage. This high level of explanatory power suggests that the silica-aerogel add-on percentage is a major determinant of the water contact angle, with minimal influence from other variables:

$$y = 127.23 x + 0.96 \quad (6)$$

where x is silica-aerogel add-on % and y is water contact angle.

The untreated samples N0, N1, and N2 have WCA of 126.77°, 126.47° and 126.56°, respectively. After treatment with silica-aerogel, WCA of N0-SiGel 154.79°, N1-SiGel 154.3° and N2-SiGel 154.2°, representing a 22.51%, 27.71%, and 32.6% increase, respectively. The linear fit analysis reveals a strong positive relationship between the silica-aerogel add-on percentage and the water contact angle of the needle-punched nonwoven samples. As the amount of silica-aerogel increases, the water contact angle increases markedly, indicating enhanced hydrophobic properties. This finding indicates the effectiveness of silica-aerogel treatment in improving the water repellency of nonwoven materials, making them more suitable for applications where moisture resistance is critical.

3.8. Relationship between Silica-Aerogel Add-on Percentage Vs. Filtration Efficiency

A linear fit analysis has been conducted to investigate the relationship between the silica-aerogel add-on percentage and the filtration efficiency of needle-punched nonwoven samples. The derived linear Eq. (7) is presented in Fig. 5 (b). The significant slope highlights the substantial impact of silica-aerogel treatment on improving the filtration efficiency of the samples. The R² value of 0.87 signifies that the linear model explains 87% of the variability in the filtration efficiency based on the silica-aerogel add-on percentage. This high R² value indicates that the majority of the changes in filtration efficiency can be attributed to variations in the silica-aerogel add-on percentage, confirming the robustness and reliability of the linear model:

$$y = 98.24 x + 0.031 \quad (7)$$

where x is silica-aerogel add-on % and y is filtration efficiency.

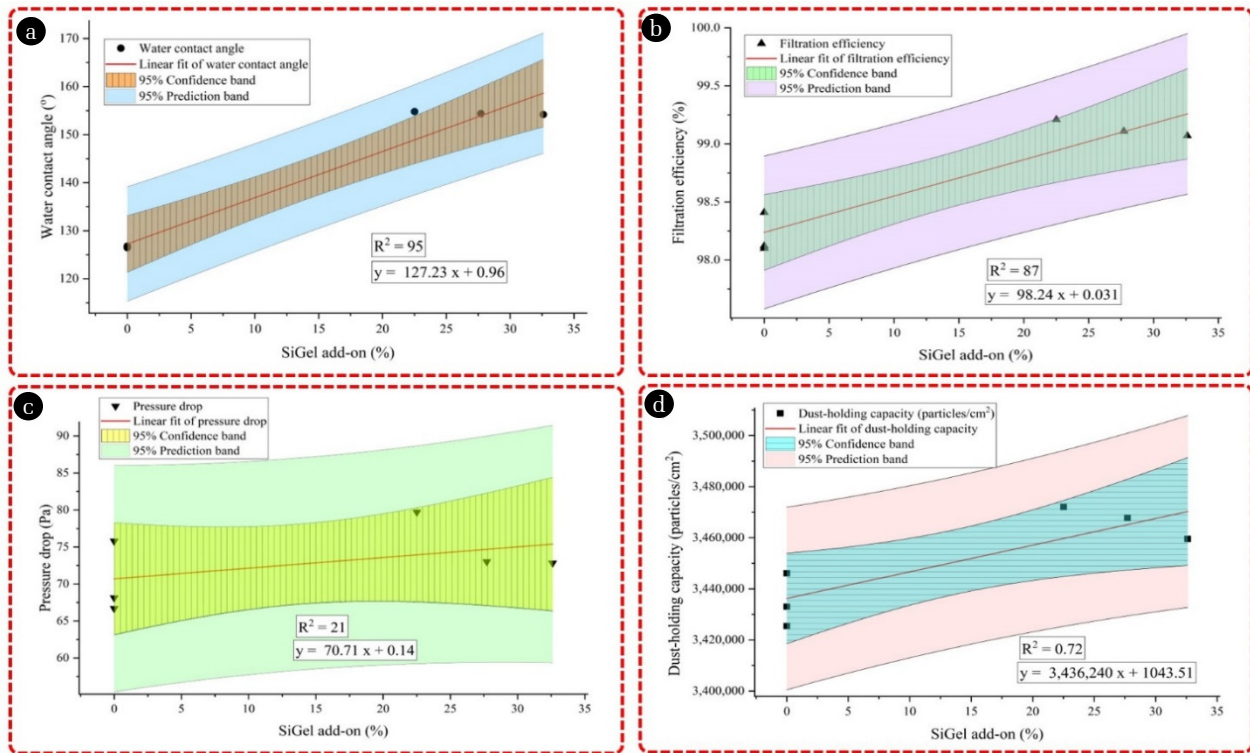


Fig. 5. Relationship between properties of needle-punched nonwoven samples: a) linear fit of silica-aerogel add-on % with water contact angle, b) linear fit of silica-aerogel add-on % with filtration efficiency, c) linear fit of silica-aerogel add-on % with pressure drop, d) linear fit of silica-aerogel add-on % with dust-holding capacity.

Initially, the untreated samples (N0, N1, and N2) exhibit filtration efficiencies of 98.41%, 98.12%, and 98.10%, respectively, with no silica-aerogel add-on. Upon treatment with silica-aerogel, the filtration efficiencies of the samples improved significantly. Specifically, N0-SiGel with a 22.51% silica-aerogel add-on shows an increased filtration efficiency of 99.21%. Similarly, N1-SiGel with a 27.71% silica-aerogel add-on exhibits a filtration efficiency of 99.11%, and N2-SiGel with a 32.60% silica-aerogel add-on achieves a filtration efficiency of 99.07%. The linear fit analysis reveals a strong positive relationship between silica-aerogel add-on percentage and filtration efficiency. As the amount of silica-aerogel increases, the filtration efficiency of the nonwoven fabrics improves significantly. However, it is important to note that while there is an overall improvement in filtration efficiency with higher silica-aerogel add-on percentages, the incremental gains in efficiency tend to diminish slightly at higher add-on levels.

3.9. Relationship between Silica-Aerogel Add-on Percentage Vs. Pressure Drop

A linear fit analysis has been performed to explore the relationship between the silica-aerogel add-on percentage and the pressure drop across needle-punched nonwoven samples. The resulting linear Eq. (8) is presented in Fig. 5 (c). The R^2 value of 0.21 indicates that the linear model explains 21% of the variability in the pressure drop based on the silica-aerogel add-on percentage. While this is a low level of explanatory power, it suggests that other factors might also influence the pressure drop in addition to the silica-aerogel add-on percentage:

$$y = 70.71x + 0.14 \quad (8)$$

where x is silica-aerogel add-on % and y is pressure drop.

The untreated samples (N0, N1, and N2) showed pressure drops of 75.8 Pa, 68.12 Pa, and 66.69 Pa, respectively. After silica-aerogel treatment, a pressure drop of N0-SiGel with a 22.51% silica-aerogel add-on increased to 79.71 Pa. N1-SiGel with a 27.71% silica-aerogel add-on increased a pressure drop of 73.02 Pa, and N2-SiGel with a 32.60% silica-aerogel add-on achieved 72.83 Pa. The linear fit analysis reveals a moderate positive relationship between silica-aerogel add-on percentage and pressure drop. As the amount of silica-aerogel increases, the pressure drop across the nonwoven fabrics also increases, reflecting higher airflow resistance. This finding highlights a trade-off when using silica-aerogel to enhance certain properties of nonwoven materials, as it can lead to increased pressure drop, which may affect the overall performance in filtration applications.

3.10. Relationship between Silica-Aerogel Add-on Percentage Vs. Dust-Holding Capacity

A linear fit analysis has been conducted to explore the relationship between the silica-aerogel add-on percentage and the dust-holding capacity of needle-punched nonwoven samples. The resulting linear Eq. (9) is present in Fig. 5 (d). The significant slope underscores the profound impact that silica-aerogel treatment has on enhancing the dust-holding capacity of the samples. The R^2 value of 0.72 signifies that the linear model explains 72% of the variability

in the dust-holding capacity based on the silica-aerogel add-on percentage. This high R^2 value confirms the reliability of the linear model and highlights that the majority of the changes in dust-holding capacity can be attributed to variations in the silica-aerogel add-on percentage:

$$y = 3,436,240x + 1043.51 \quad (9)$$

where x is silica-aerogel add-on % and y is dust-holding capacity

The untreated samples (N0, N1, and N2) have dust-holding capacities of 3,446,120 particles/cm², 3,433,000 particles/cm², and 3,425,400 particles/cm², respectively. After treatment with silica-aerogel, the dust-holding capacities for the treated samples (N0-SiGel, N1-SiGel, and N2-SiGel) increase to 3,472,060 particles/cm², 3,467,770 particles/cm², and 3,459,520 particles/cm², respectively. The corresponding silica-aerogel add-on percentages for these treated samples are 22.51%, 27.71%, and 32.6%, respectively. The linear fit analysis demonstrates a robust positive relationship between silica-aerogel add-on percentage and dust-holding capacity. As the amount of silica-aerogel increases, the fabric's ability to capture and retain dust particles significantly

improves. This finding suggests that silica-aerogel treatment is highly effective in enhancing the filtration properties of needle-punched nonwoven fabrics, making them particularly useful for applications requiring high dust-holding capacity.

3.11. Statistical Analysis

The statistical analysis of untreated and treated samples using ANOVA and Tukey Test at a significance level of 0.05, based on their Sig-value and P-value, as presented in Fig. 6 and Table 2, providing insights into the impact of silica-aerogel treatment on various properties of the needle-punched nonwoven samples. For the pressure drop, air permeability and porosity, the comparison between untreated and treated samples N0 vs. N0-SiGel and N1 vs. N1-SiGel do not show a significant difference. This suggests that the addition of silica-aerogel does not substantially alter the pressure drop for these specific samples. In contrast, for sample N2, there is a significant difference in pressure drop, air permeability, and porosity between untreated and treated samples (N2 vs. N2-SiGel), indicating that the silica-aerogel treatment had a noticeable impact on this sample. Regarding fabric thickness, all comparisons show no significant differences across untreated and

Table 2. Statistical analysis of untreated and treated samples

Fabric properties	Mean				Means Comparison using Tukey Test		0.05 level significant test	
	Untreated		Treated		Factor	Sig	P-value	Significance
Air permeability	N0	1722.25	N0-SiGel	1662.8	N0-SiGel N0	0	0.09193000	Not Significant
	N1	1784.61	N1-SiGel	1730.96	N1-SiGel N1	0	0.14294000	Not Significant
	N2	1830.26	N2-SiGel	1682.22	N2-SiGel N2	1	0.00277000	Significant
Porosity	N0	89.99	N0-SiGel	92	N0-SiGel N0	1	0.00262000	Significant
	N1	92.37	N1-SiGel	93.59	N1-SiGel N1	0	0.12662000	Not Significant
	N2	93.12	N2-SiGel	94.32	N2-SiGel N2	0	0.19553000	Not Significant
Filtration efficiency	N0	98.41	N0-SiGel	99.21	N0-SiGel N0	1	0.00000000	Significant
	N1	98.12	N1-SiGel	99.11	N1-SiGel N1	1	0.00000000	Significant
	N2	98.1	N2-SiGel	99.07	N2-SiGel N2	1	0.00000000	Significant
Pressure drop	N0	75.8	N0-SiGel	79.71	N0-SiGel N0	0	0.05978000	Not Significant
	N1	68.12	N1-SiGel	73.02	N1-SiGel N1	0	0.11617000	Not Significant
	N2	66.69	N2-SiGel	72.83	N2-SiGel N2	1	0.01880000	Significant
Dust-holding capacity	N0	3,446,120	N0-SiGel	3,472,060	N0-SiGel N0	1	0.00000756	Significant
	N1	3,433,000	N1-SiGel	3,467,770	N1-SiGel N1	1	0.00000003	Significant
	N2	3,425,400	N2-SiGel	3,459,520	N2-SiGel N2	1	0.00000000	Significant
Fabric density	N0	121.34	N0-SiGel	150.6	N0-SiGel N0	1	0.00017904	Significant
	N1	90.96	N1-SiGel	117.59	N1-SiGel N1	1	0.00009874	Significant
	N2	83.4	N2-SiGel	112.75	N2-SiGel N2	1	0.00000019	Significant
Fabric thickness	N0	3.85	N0-SiGel	3.82	N0-SiGel N0	0	0.82918000	Not Significant
	N1	4.17	N1-SiGel	4.15	N1-SiGel N1	0	0.87328000	Not Significant
	N2	4.63	N2-SiGel	4.57	N2-SiGel N2	0	0.64773000	Not Significant
Fabric GSM	N0	467.36	N0-SiGel	572.61	N0-SiGel N0	1	0.00000053	Significant
	N1	379.29	N1-SiGel	484.45	N1-SiGel N1	1	0.00000012	Significant
	N2	386.52	N2-SiGel	512.55	N2-SiGel N2	1	0.00000000	Significant
Water contact angle	N0	126.77	N0-SiGel	154.79	N0-SiGel N0	0	0.00000000	Significant
	N1	126.47	N1-SiGel	154.38	N1-SiGel N1	0	0.00000000	Significant
	N2	126.56	N2-SiGel	154.2	N2-SiGel N2	0	0.00000000	Significant

- P-value ≤ 0.05 indicates that at 0.05 level, the population means are significantly different.
- Sig equals 1 indicates that the difference of the means is significant at 0.05 level.
- Sig equals 0 indicates that the difference of the means is not significant at 0.05 level.

at 99.9%, even after being subjected to treatment at 200°C for a duration of 120 hours [57]. Zhao et al. have conducted a field test in Shanghai, demonstrating that the air filter consistently achieves a stable PM_{2.5} purification efficiency of 99.99% even during high moisture vapor transmission rate (MVTR) conditions prevalent in haze events [58]. Chen et al. have found that the screen made with 30 ml of CNFs dispersion attained a 96% removal rate for PM_{2.5}, surpassing the N95 mask's 76% under comparable conditions [59].

3.13. Limitations and Future Works

Scaling up the production of silica-aerogel treated needle-punched nonwovens for air filtration involves optimizing the manufacturing process, including fiber blending, web arrangement, and silica-aerogel treatment, to ensure consistent quality across larger batches. Automation and adaptation of equipment are essential to handle larger volumes and maintain precise control, while securing a steady supply of raw materials and managing costs are crucial for economic viability. Quality control measures must be established to monitor performance characteristics throughout production. Challenges in scaling up include ensuring reproducibility in silica-aerogel treatment, maintaining performance characteristics like filtration efficiency and oil sorption, and complying with environmental and regulatory standards. Limitations of current research include evaluating the long-term durability of materials under harsh conditions, assessing the cost of large-scale production, and understanding material reusability. Future work should focus on industrial-scale testing to address unforeseen manufacturing challenges, durability studies under various conditions, exploring alternative fiber blends for enhanced performance, and assessing the environmental impact to ensure sustainability.

4. Conclusions

Air pollution is a major health threat due to fine particles like PM_{2.5}, which can enter the bloodstream and cause premature deaths. Textile filter materials, particularly hydrophobic needle-punched nonwoven fabrics are crucial for air pollution control due to their ability to capture harmful particles. The silica-aerogel treatment resulted in add-on percentages of 22.51% for N0-SiGel, 27.71% for N1-SiGel, and 32.60% for N2-SiGel, with the highest add-on percentage observed in N2-SiGel due to its lower density and higher porosity. The integration of silica-aerogel into the nonwoven samples was achieved through a combination of hydrogen bonding, coordination interactions, and physical entanglement, leading to enhanced adhesion and bonding strength. This was confirmed by FTIR analysis, which identified the characteristic Si-O-Si stretching vibrations at wavelengths around 1000 to 1100 cm⁻¹, indicating the presence of silica-aerogel.

The silica-aerogel treatment significantly increased the water contact angle (WCA) of the samples, elevating them into the super-hydrophobic range (WCA greater than 150°). This substantial change in WCA indicated a significant modification in the surface hydrophobicity of the treated samples by increasing the surface area up to 78-84%. The treatment also significantly improved the

filtration performance of the nonwoven samples. The dust-holding capacity increased by 0.75% to 1.01%, and the filtration efficiency improved by 0.8% to 1.01% across all samples. However, this improvement came with an increased pressure drop, ranging from 5.16% to 9.21%, attributed to the increased fabric density and reduced pore diameter. The decreased pore diameters and increased fabric density enhanced the filtration efficiency by restricting the passage of fine particles, while the reduced air permeability ensured more thorough filtration of PM_{2.5} particles.

The statistical analysis indicated that silica-aerogel add-on percentage possessed a positive correlation with filtration efficiency, pressure drop, dust-holding capacity, and hydrophobicity. These results underlined the effectiveness of silica-aerogel treatment in enhancing the functional properties of needle-punched nonwoven samples, making them more suitable for applications requiring high filtration efficiency and super-hydrophobicity. Generally, the study justified that blended needle-punched nonwovens with silica-aerogel treatment are a promising solution for PM_{2.5} air filtration due to their effectiveness and ability to handle humid environments and reduce millions of deaths each year because of air pollution.

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Conflicts of Interest Statement

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.

Author Contributions

H.M.Y. (PhD student) conceptualized and conducted all sample preparation, testing, data analysis, and wrote the manuscript. S.R.I. (Assistant professor and chairman) conceptualized, visualized, edited, and revised the manuscript. Q.X. (Professor) and Z.X. (Professor) participated in conceptualization, validation, project administration, funding acquisition, editing, and supervision.

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