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High Throughput Synthesis of Silica Nanoparticles and Its Influence on Mechanical, Comfort, and Handle-Related Properties of Woven Fabrics.



Alsaid Ahmed Almetwally¹, Hazem A. Yassen ², Fayez M. Eissa³

¹ Textile Research and Technology Institute, National Research Centre, Dokki, Cairo, Egypt.
 ²Industrial Education Department, Faculty of Education, Helwan University, Cairo, Egypt
 ³Aswan University, Faculty of Science, Chemistry Department, Aswan 81528, Egypt

Abstract

The main goal of this paper was to investigate the effect of silica nanoparticle size and concentration on cotton woven fabric. Silica nanoparticles (SiO₂NPs) were synthesized using the Sol technique. Three sizes and three concentrations for each particle size were prepared and cotton woven fabrics were coated using the nine patches. Using SEM, TEM, and FTIR, the prepared particles were analyzed. The influence of Silica nanoparticles' size and concentration on antibacterial activity, roughness values, tensile strength, and air permeability of the coated woven fabrics were also investigated. The statistical analysis was conducted to assess the significant effects of SiO₂NPs' size and concentration on fabric properties. The findings of this study revealed that the fabric handle and its tensile properties are significantly affected by the concentration and sizes of the silica nanoparticles. The treated fabrics' air permeability and roughness characteristics were negatively affected by the concentration and sizes of SiO₂NPs. By contrast, the tensile properties of the treated fabrics are enhanced substantially by the treatment with silica nanoparticles.

Keywords: Silica nanoparticles, Sol-gel, antibacterial activity, roughness value, Air permeability.

1. Introduction

Due to their diverse applications in commercial and industrial areas, metal oxide nanoparticles are widely utilized. Nowadays, it was reported that the production of metal oxide nanoparticles has been increased substantially compared to the last decade [1].

Because it releases reactive oxygen species (ROS) which comprises O-2 and OH, some of the nanostructures impart antibacterial activity to textile materials [2]. Due to its porous structure, adsorption characteristics, and very high surface activity, SiO₂NPs have become an ideal antimicrobial agent [3]. Also, among other metal oxide nanoparticles, Sio2 NPS proved themselves as an ultraviolet protection agent and water repellent [4-6].

Numerous studies [7-16] have looked into the physical, mechanical, and comfort aspects of cottonwoven fabrics. Also, the finishing of this type of fabric with metals and their oxides nanoparticles has been accomplished in formidable research works. On the contrary, the finishing of cotton fabric with SiO₂NPs and their mixtures with other metal oxides nanoparticles is extremely limited. In general, SiO₂NPs are synthesized using the sol-gel technique, and tetraethyl orthosilicate (TEOS) is the chemically available precursor from which their base material can be obtained [17, 18]. But, because of thermal degradation, rice hulls which are taken from a natural source, is considered the simplest, most cost-effective, and scalable technique to produce SiO₂NPs [19].

In order to impart high and durable hydrophobicity and antibacterial activity to cotton woven fabrics, Shagufta, et al. [20] developed a new approach to modify SiO₂NPs using silane coupling agents, such as trimethoxy-silane (3-Glycidoxypropyl), and 3-(Trimethoxysilyl) propyl-N,N,N-dimethyl octadecyl ammonium chloride. The modified SiO2NPs increased antibacterial activity and water contact angle up to 147.6^o of cotton fabrics. Besides, the comfort characteristics of the fabrics understudy were enhanced substantially. Nabil, A. Ibrahim, and coauthors [21] have coated woven polyester fabrics with titanium dioxide, zinc oxide, Zirconium dioxide, and silicon dioxide nanoparticles with two different binding materials. The influence of these different ingredients and their interaction with binding materials' type on functional properties such as selfcleaning capability, antibacterial activity, and UPF and

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softness characteristics were examined. The results obtained from this study revealed that the functional characteristics of the woven fabrics are governed by the type of metal oxide nanoparticles as well as the kind of binding material.

In another work [22], in the melt spinning process of Nylon-6 multi-filaments, the dry SiO₂NPs were mixed with Nylon-6 pellets in the screw extruder. The influence of this metal oxide nanoparticle on the tensile and thermal characteristics of Nylon-6 filament was investigated. It was found that tensile properties of Nylon-6 multifilament such as ultimate tensile strength, yield strength, and tensile modulus, were all improved substantially. It was also detected that the glass transition and decomposition temperature of Nylon-6 filaments have been greatly increased with the inclusion of the SiO₂NPs.

Polyphenylene sulfide fibers (PPS) are a type of highperformance one which has powerful corrosion resistance, flame resistance, and heat resistance. On the other hand, once this type of fiber is exposed to sunlight its breaking strength seriously degrades because of its poor UV-resistance [23]. The influence of silica and titanium dioxide nanoparticles on the UV resistance and mechanical characteristics of this type of fibers has been addressed [24]. It was proved that the inclusion of the metal oxide nanoparticles enhanced significantly both the UV resistance and mechanical characteristics of PPS.

As per the aforementioned studies, the influence of metal oxides nanoparticles on woven fabrics including SiO_2NPs has been researched using a mixture of them. It was also noticed that SiO_2NPs aren't used alone. The literature examining the size-dependent characteristics of SiO_2NPs is not available to a large extent. Therefore, the novelty of this work lies in studying the influence of SiO_2NPs of different sizes. Furthermore, the interaction effect between the concentrations of SiO_2NPs and their sizes has never been investigated before.

2. Experimental

2.1. Materials

In this study, desized, scoured, and bleached 100% cotton-woven fabrics with a plain weave structure and a weight of 120 g/m² were used. The woven fabric samples were produced with a yarn of count 78/2 Ne and with a density of 105 ends/inch and 72 picks/inch respectively. These woven fabrics will be designed and tailored in a form of men's shirt.

Tetraethylorthosilicate (TEOS) (Si(OC₂H₅)₄, ethanol, and ammonia which are used as starting materials were purchased from Sigma-Aldrich.

2.2. Preparation of silica nanoparticles (SiO₂NPs)

Throughout this study, SiO₂NPs with three different sizes, namely, 52 nm, 90 nm, and 135 nm respectively were synthesized using sol-gel technique. This technique is outlined as follows: The preparation of sol was performed by mixing 240 ml of ethanol along with 50 ml of distilled water to different volumes of the precursor: tetraethylorthosilicate (TEOS: 100 ml, 150 ml, and 200 ml). The addition of ethanol and water to TEOS was carried out in a glass beaker (1000 mL) to ensure the homogenization of these components with TEOS. To avoid the rapid hydrolysis of TEOS, ethanol and distilled water were added dropwise to TEOS. At the end of ethanol and distilled water addition, ammonia solution (200 mL) was added to precipitate the formed silica oxide nanoparticles (SiO₂NPs). The solution is subjected to filtration, followed by washing the precipitate several times with an excess amount of water to remove the residual ammonia. The end product is silica nanoparticles of three different sizes. Ultimately, three different concentrations of SiO₂NPs were formed and varied depending on the three different of the utilized TEOS (100, 150, and 200 ml) respectively. The samples were kept in closed glass bottles for further characterization and application for different samples of cotton fabrics.

The reactions can be schematically represented as follows:

Hydrolysis: $Si(OC_2H_5)_{4+}$ $2H_2O \longrightarrow Si(OH)_{4+}$ 4CH₃CH₂OH

Condensation: $(HO)_3Si-OH + HO-Si(OH)_3 \longrightarrow$ $(HO)_3Si-O-Si(OH)+H_2O$

The hydrolysis rate of TEOS depends on the concentrations of H_2O and NH_3 . As the concentration of NH_3 in the solution rises, H_2O dissociates, releasing more OH– ions, which attack the Si atoms and speed up the hydrolysis process. While it has long been known that the amount of NH_3 and H_2O is used to manage the equilibrium between TEOS hydrolysis and condensation.

2-3: Treatment of cotton fabrics with different concentrations of SiO₂NPs-1, SiO₂NPs-2 and SiO₂NPs-3

Accurate weight; 0.5 g, 1 g, and 1.5 g of the resultant powder (SiO₂NPs-1, SiO₂NPs-2, and SiO₂NPs-3) were dispersed in 100 mL of distilled water containing 2 ml of Tween 80 as an emulsifying agent. the dispersion was performed under the aid of magnetic stirring (30 min) followed by ultrasonication for 15 min. After complete dissolution, cotton fabric samples were submerged in the aforementioned solutions for 2 min and squeezed using pad dry-cure-method with pickup 100 %. After that, the squeezed cotton fabrics samples were dried at 80 °C for 3 min and cured for 2 min at 130 °C. The scheme of producing SiO₂NPs is also shown in Figure 1.



Figure 1: Schematic representation of synthesizing SiO₂NPs.

2.3: Characterization of Silica Nanoparticles (SiO₂NPs)

The Transmission Electron Microscope (TEM) of model JEM-2100 was used to characterize the morphology of silica nanoparticles. Fourier Transform Infrared Spectroscopy (FTIR) was also used to evaluate the functional groups of silica nanoparticles using Nicolet iS50 FTIR (American Thermo Nicolet Company). In order to achieve the elemental analysis of the prepared particles, this device was equipped with Energy Dispersive Spectroscopy (EDS) with -30 KV accelerating voltage.

2.4. Characterization of coated fabrics

In order to examine the influence of SiO₂NPs and their sizes, and concentrations on the functional characteristics of the finished fabrics, air permeability, tensile strength, roughness values, and antibacterial activity of the finished fabrics were investigated. Air permeability was measured using Permeameter instrument No. 869 according to standard ASTM D737-18. Tensile strength of the treated and blank fabric samples was also evaluated using the Instron measuring device of model 4465 according to ASTM D5035-11 (2019). The fabric roughness of the fabrics was recoded using Atomic Force Microscopy (AFM). The average values of tensile strength and air permeability of the untreated (Blank) woven fabrics are 305 Newton and 30.2 cm³/cm².sec respectively. According to standard test method AATCC 147-1988, antibacterial activity of coated woven fabrics was tested qualitatively against gram-positive and gramnegative bacteria, namely Staphylococcus aureus and Escherichia coli, respectively.

2.5. Statistical analysis

To explore the effects of SiO_2NPs sizes and concentrations on the properties of the woven cotton fabrics, a 3^2 full factorial design was conducted. The different control factors and their selected levels were shown in table 1.

To detect the significant effect of control factors and also their interaction effect on the functional properties of the woven fabrics, Analysis of Variance (ANOVA) was conducted. It should be noted that the significance of each control factor was assessed at a significance level, namely at $\alpha \leq 0.05$.

In order to explore and derive the regression relationship between each functional property for the finished fabrics at the different levels of the control factors, a regression analysis was implemented as well. The regression relationship which correlates the control factors with each functional fabric property has the following form:

Z=a+b X+c Y+d XY+e X2+f Y2

Where,

Z= functional fabric property (fabric tensile strength,

Air permebility, ..., etc)

X= SiO₂NPs size

 $Y = SiO_2NPs$ concentration

a = constant

b, c, d, e, f = regression coefficients.

To assure the degree of reliability of each regression equation, the coefficient of determination (R^2 value) was calculated. This coefficient can be anywhere between 0 and 1. Whenever the coefficient of determination approaches one, this means that the regression equation is reliable.

Table 1:	Control	factors	and	their	levels	used	in	the
Study (A	3^2 full fa	actorial	desi	gn).				

Sample No.	Diameter of SiO ₂ NPs (nm)	SiO ₂ NPs concentration (g/l)		
1	52	5		
2	52	10		
3	52	15		
4	90	5		
5	90	10		
6	90	15		
7	135	5		
8	135	10		
9	135	15		
10	Blank- not treated			

3. Results and discussion

3.1. Characterization of SiO₂NPs

The TEM images of silica nanoparticles with different diameters were illustrated in Figure 2. As shown from this figure, the SiO_2NPs have a solid sphere. It is also shown that the different sizes of nanoparticles are completely synthesized without any agglomeration. Also, for all sizes, the nanoparticles are all equiaxed in shape to each other.

It should be noted that the prepared silica nanoparticles' size was controlled by controlling the synthesis parameters in the sol-gel technique. In general, smaller nanoparticles are effectively obtained by controlling the polycondensation reaction of the sol-gel process. On the other hand, it was reported that as the ammonia concentration increases, the particle diameter decreases [25-28].



Figure 2: TEM images of SiO_2NPs with different diameters obtained by controlling the reaction parameters: (a) 52 nm (b) 90 nm and (c) 135 nm.



Figure 3: (a) size distribution, (b) PDI values, and (c) zeta potential of the prepared SiO₂NPs

Figure 3 (a, b) depicts the sizes distribution of the resulted SiO₂NPs and their corresponding polydispersity index (PDI). The PDI value is used to evaluate the homogeneity of the prepared SiO₂NPs. From this figure, it is clear that the prepared sizes of the silica nanoparticles are centered on 52, 90, and 135 nm respectively. Also, the associated PDI is 0.923, 0.516, and 0.328 respectively. It is also verified that the high stability, good quality, and homogeneous dispersion of the prepared colloidal solution of silica nanoparticles are associated with low values of PDI. Zeta Potential of the prepared silica nanoparticles as shown in Figure 3 c gives us the prediction about the stability of the formed nanoparticles. From this figure, it is shown that all zeta potential had narrow distribution with a negative charge. It is well known that the value of the zeta potential above ± 30 is an indicator of the good stability of the obtained nanoparticles. As depicted in this figure, SiO₂NPs with diameters 52 nm, 90 nm, and 135 nm are formed with values of Zeta potential of -33, -38, and -22 respectively. The presence of oxygen atoms, which is the most essential ingredient in generating the negative charge, results in a negative value in general.

Throughout this study, FTIR spectroscopy has been conducted to investigate the sol-gel silica nanoparticles. This analysis gives an important

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indication of the effect of the processing conditions on the microstructure of the prepared nanoparticles. The FTIR spectra of the obtained SiO₂NPs with different sizes are depicted in Figure 4. It is clear from this figure that the outstanding absorption bands are located at 447cm⁻¹, 795 cm⁻¹, and 1049cm⁻¹ which complies with the structure of the SiO₂ bands. The bands detected at these wavenumbers are ascribed to the bending modes between Si-O-Si bonds. Also, the peak located at 3384 cm⁻¹ is assigned to H-O bonds. It is worth noting that increasing the TEOS content leads to shifting in the absorption bands' position which results in the O-Si-O network perturbation. Also, it is evident that the different sizes of SiO₂NPs don't affect significantly the location of the absorption bands.



Figure 4: FTIR spectra of SiO₂NPs with different sizes: a) 52 nm, b) 90 nm, and c) 135 nm.

3.2. Characterization of coated cotton fabrics.

Scanning Electron Microscope of untreated and treated cotton fabric surfaces with different sizes and concentrations of silica nanoparticles is depicted in Figure 5. From this figure, it can be seen that SiO_2NPs are clearly and well distributed on the cotton fiber surface. The concentration and diameter of silica nanoparticles play a vital role in their attachment to the cotton fiber. As the nanoparticle size and its concentration increase, the nanoparticles can agglomerate on the fiber surface. By contrast, with a low diameter and high concentration, the SiO_2NPs can penetrate deeper and strongly adhere to the fiber surface.

The chemical composition of the coated cotton fabrics' surface was examined using Energy Dispersive Spectroscopy (EDS) analysis. In Figure 6 (a), only carbon and oxygen peaks were detected on the surface of untreated cotton fabrics. After coating cotton fabrics with SiO₂NPs, a peak of Si was noticed as depicted in Figure 6 (b). and Figure 6 (c). The weight percent of Si was 3.87%, 5.83%, and 8.75% at

concentrations of 100 ml and 200 ml of TEOS respectively assuring that the cotton woven fabrics were covered with SiO_2NPs .



Figure 5: SEM images of blank and treated cotton fabric with different sizes and concentrations of SiO_2NPs : a) blank, b-d) treated with silicon nanoparticles of size 52 nm with concentrations 100 ml, 150 ml and 200 ml of TEOS respectively, e, f) treated with SiO_2NPs of sizes 90 and 135 nm respectively, and with concentration of 200 ml of TEOS.



Figure 6: EDS of blank and coated cotton fabrics with SiO₂NPs.

3.3. Air permeability

The air permeability of cotton woven fabrics coated with different sizes and concentrations of SiO_2NPs was illustrated in Figure 7. The results of the Two-Way ANOVA listed in table 2 revealed that the size and concentration of SiO_2NPs have a considerable impact on the air permeability of coated cotton fabrics at a significant level of 0.01. The statistical analysis also disclosed that the particle size and its concentration account for 24% and 46% respectively of the effects on fabric air permeability. As seen in Figure 7, decreasing trends were detected for both independent variables. In a general sense, as both variable levels increase, the air permeability of the coated fabrics decreases. It was also estimated that increasing particle size from 52 nm to 135 nm leads to a reduction in the coated fabric air permeability by about 7%. While the increase in concentration from 100 ml to 200 ml of TEOS leads to a reduction in air permeability by approximately 10%.

It should be noted that the air permeability of treated cotton fabrics with SiO_2NPs is generally less than untreated fabric by about 10%. The reduction of air permeability of coated fabric with increasing the particle size and its concentration may be related to the penetration and crosslinking of nanoparticles to the cotton fibers that cover and block the fiber pores and yarns' interstices which in turn reduces the passage of the air through the coated fabrics.

The Multiple linear regression which correlates air permeability of coated fabrics to the levels of particle sizes and their concentrations has the following form: Z = 31.9 - 0.016 X - 0.2 Y

Where.

Z= Air permeability $(cm^3/cm^2.s)$,

X= nanoparticle size (nm), and

Y= nanoparticle concentration (g/l)

The statistical analysis confirmed that the coefficient of determination of this linear model equals 0.84 which means that model fits the data very well. Also, the standard error of this model was estimated to be 0.09 which confirms that this predictive model is very reliable.



Figure 7: Response surface of the air permeability of cotton woven fabrics coated with different sizes and concentrations of SiO₂NPs.

Table 2: Analysis of variance results of the effects of particle size and concentration on air permeability of coated fabrics

Variation source	SS	DF	MS	F-value	P- value
Concentration	5.8256	2	2.912	16.584	0.011
Particle size	3.1773	2	1.588	9.045	0.032
Error	0.7025	4	0.175		
Total	9.7055	8			

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3.4: Antibacterial activity

Agar diffusion was used to test the antibacterial activity of the coated cotton fabrics with SiO_2 NPs against gram-positive and gram-negative bacteria, namely *Staphylococcus aureus* and *Escherichia coli*, respectively (Figure 8). Antibacterial activity was qualitatively assessed using an inhibition zone of the analysis. In general, the inhibition zone with a higher diameter which can be formed around the fabric sample placed on the agar inoculated with positive and negative gram bacteria are evidence that the coated fabrics have a better antibacterial impact against both types of bacteria.

The results of the antibacterial activity of the untreated and treated cotton fabrics with different sizes and concentrations of SiO₂NPs were portrayed in Figure 8. From this Figure, it is clear that there is no inhibition zone was formed around any fabric sample whether the untreated or the treated ones. This means that there is no antibacterial agent that has been transferred from cotton fabrics. In other words, there is no antibacterial effect of SiO₂NPs at any size or degree of concentration.



Figure 8: Antibacterial activity of fabrics coated with SiO₂NPs.

3.5: Roughness values

AFM images of cotton fabrics treated with SiO₂NPs at different sizes and concentrations were depicted in Figure 9. From this figure, it can be seen that as the size and concentration of silica nanoparticles increases the roughness of the treated cotton fabrics' surface reacts in the same manner. This is because of the inclusion of SiO₂NPs onto the fabric surface. The response surface of the influence of both nanoparticles' size and their concentration on the average roughness value of the treated cotton fabrics was depicted in Figure 10. The results of the analysis of variance of both independent variables on the surface roughness were also listed in table 3. From Figure 10 and statistical analysis, it is noticed that the size of nanoparticles and their concentration have a

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positive impact on the roughness of the treated fabrics. As the levels of both variables increase, the average roughness values also increase. The statistical analysis also showed that increasing SiO₂NPs size from 52 nm to 135 nm leads to an increase of the average value of the fabric roughness by about 44%. It was also found that the increase of nanoparticles' concentration from 100 to 200 results in the increase of the roughness average values by approximately 58%. From this result, it can be concluded that the concentration of nanoparticles is more influential than nanoparticle size with respect to fabric roughness. Finally, it should be noted that finishing cotton fabrics with SiO₂NPs increased the average values of their roughness from 0.03 μ m to 0.05 μ m.



Figure 9: AFM images of blank and treated cotton fabric with different sizes and concentrations of SiO_2NPs : a) blank, b-d) treated with silicon nanoparticles of size 52 nm with concentrations 5 g/l, 10 g/l and 15 g/l of SiO_2NPs respectively, e, f) treated with silicon nanoparticles of sizes 90 and 135 nm respectively, and with concentration of 20 g/l.

Table 3: Analysis of variance results of the effects of particle size and concentration on average roughness values of coated fabrics.

Variation source	SS	DF	MS	F-value	P- value
Concentration	0.00095	2	0.0004	46.49	0.001
Particle size	0.000569	2	0.0002	27.84	0.004
Error	4.09E-05	4	1.02E-05		
Total	0.00158	8			

The multiple linear regression equation which correlates the average value of cotton fabric roughness with the levels of nanoparticles' size and their concentration is as follows: Z = 0.0134 + 0.0002 X + 0.0016 Y

Where Z = average roughness value (μ m) X=nanoparticle size (nm), and Y= nanoparticle concentration (g/l)



Figure 10: Response surface of the average roughness values of cotton woven fabrics coated with different sizes and concentrations of silica nanoparticles

The statistical analysis revealed the coefficient of determination of this regression model equals 0.96 which means that this model fits that experimental data very well. It was also estimated that the standard error of the mode is about 0.05 which confirms that the regression model is very reliable.

3-6: Tensile strength

The tenacity of cotton treated fabrics with different levels of silica nanoparticles' sizes and concentrations were illustrated in figure 11. The values of the statistical analysis of the effect of both independent variables on fabric tensile strength were also tabulated in Table 4. From this figure and the ANOV values, it is clear that the concentration of silica nanoparticles has a profound influence on the treated fabric tensile strength at 0.05 significance. An increasing trend was detected assuring that as the concentration of nanoparticles increases, the tenacity of the treated cotton fabric follows the same trend. On the contrary, the size of SiO₂ NPs was found to have no significant impact on the treated fabric tensile strength. It was estimated that increasing the concentration of SiO₂ NPs from 5 g/l to 15 g/l leads to an increase in the treated woven fabrics by about 14.7%.



Figure 11: Response surface of the tensile strength of cotton woven fabrics coated with different sizes and concentrations of SiO₂NPs.

It was also detected that treating cotton fabrics with silica nanoparticles increased the tenacity of the fabrics by approximately 18%. The improvement of the treated cotton fabrics' tensile strength may be due to the binding resulting from the incorporation of silica nanoparticles.

Table 4: Analysis of variance results of the effects of particle size and concentration on tensile strength of coated fabrics

Variation source	SS	DF	MS	F- value	P- value	
Concentration	3870.83	2	1935.415	7.24	0.046	
Particle size	149.700	2	74.850	0.28	0.7694	
Error	1069.29	4	267.322			
Total	5089.82	8				

The multiple non-linear regression which correlates both nanoparticles size and their concentration with the tensile strength of the coated fabric has the following form:

Z= 375 - 1.5 X +8.4 Y+ 0.06 XY + 0.04 X² -0.5 Y² Where,

Z= Tensile strength of the treated fabric (Newton),

X= Nanoparticle size (nm), and

Y= Nano particle concentration (g/l).

The statistical analysis proved that the coefficient of determination of this regression model is about 0.84 and its standard error equals 0.082 which means that this model fits the experimental data very well and has reliable predictive power.

Conclusion

Three sizes of silica nanoparticles (SiO₂NPs) with diameters of 52 nm, 90 nm, and 135 nm were manufactured using the Sol-gel process. Cotton fabrics were coated with three concentrations of each particle size, namely 5, 10, and 15 g/l. The TEM, SEM, FTR, and Zeta potential procedures were used to evaluate the produced nanoparticles. The treated fabrics' antibacterial activity and physical properties were tested. The following were the study's main findings:

- Neither gram-positive nor gram-negative bacteria showed antibacterial action in the presence of silica nanoparticles.
- The roughness of coated cotton fabrics increased with particle size and concentration, outperforming untreated samples.
- As particle size and concentration grew, coated textiles' tensile strength increased but their air permeability decreased.

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Conflicts of interest

There are no conflicts to declare

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