## Jurnal Kejuruteraan 36(2) 2024: 811–816 https://doi.org/10.17576/jkukm-2024-36(2)-38

# Characterization and antimicrobial Properties of Cotton Fabric Loaded with green Synthesized Silica Nanoparticles

Muhammad Amira\*, Muhammad Sajid Ali Asgharb & Saeeda Nadir Alic

<sup>a</sup>Department of Textile Engineering, NED University of Engineering and Technology, Karachi, 75270, Pakistan

<sup>b</sup>Department of Materials Engineering, NED University of Engineering and Technology, Karachi, 75270, Pakistan

<sup>c</sup>Department of Chemistry, NED University of Engineering and Technology, Karachi, 75270, Pakistan

\*Corresponding author: email: qureshi@neduet.edu.pk

Received 17 April 2023, Received in revised form 6 August 2023 Accepted 30 October 2023, Available online 30 March 2024

#### ABSTRACT

During the past decade, improvement in the functionality of textile materials has become an important research field due to their significant health and safety benefits. Here we report, simple and cost-effective green synthesis of silica nanoparticles from fibrous residue of sugarcane bagasse followed by investigating the applicability on cotton fabric. The characteristics of synthesized nanoparticles were determined by techniques including FTIR (exhibited characteristic peak at 791.01 attributed to vibration of Si-O and the absorption band at 1072.48 cm<sup>-1</sup> owing to asymmetric vibration of Si-O-Si confirming the formation of SNPs), SEM ( $40\pm2nm$ ), and XRD (Examined spectra with JCPDS 36-1451 indicate well-defined peaks of Silicon Oxide). Debye Scherrer equation was used to calculate the mean crystallite size (19.79 nm) of powder Silica NP sample at the full width half maximum (FWHM) of the diffraction peaks of 2 $\Theta$  positions. Applicability of synthesized nanoparticles was determined by assessing thermal stability and antimicrobial property of treated and untreated cotton fabric. It was observed that silica nanoparticles coated fabric showed substantial thermal stability at 1 % Silica NPs comparatively treated with 2% and 3% silica NPs due to clustering phenomena. Antimicrobial property, confirmatory test as positive control of treated fabric shown substantial increased in antibacterial activity in solutions containing silica NPs as compared to the ethanol solution. Moreover, 76 green profiling score of synthesized method was evaluated by Analytical Eco-Scale tool.

*Keywords: Silica nanoparticle; sugarcane bagasse; functional fabric; thermal stability; cotton fabric; antimicrobial activity* 

#### INTRODUCTION

Clothing, the basic human necessity, protects the body from hot and cold weather conditions. In perspective of safety against fire and heat, suitable clothing is the most important shield. There are different types of fibres in textile industry and the most commonly used fibre is "Cotton" due to its soft and comfortable nature. Cotton fibre is widely used for admired fabric but its application is limited in different fields due to its low Ultra violet protectively, hygroscopicity and non-conductive nature. In order to eliminate these limitations while retaining its wear comfort and impart additional desired functionalities (Bhattacharjee, Macintyre et al. 2020), various coatings are applied on cotton fabrics as revealed in different literature; superhydrophobicity of cotton fabrics treated with silica nanoparticles (silica NPs) and water-repellent agent, enhanced ballistic performance in kevlar fabric (Bablu and Manimala 2022), use of photo reactive silica NPs for super-hydrophobic cotton fabrics application (Liu, Wang et al. 2015).

Silica NPs have widespread applications in various industries with some known effects on textiles. It was reported that to enhance flame retardance of cotton fabric by applying nano-silica (Fanglong, Qun et al. 2016). In fact, an optimal nano-silica amount increased the LOI and reduced the thermal decomposition of the cellulosic materials. It was also established that a mixture of nanosilica and traditional intumescent flame retardant showed synergistic effects on the flame resistance of cellulosic textiles. In addition, application of silica NPs through the layer-by-layer coating as a flame-retardant material and found impressive changes in the principle factors of flammability. Further, silica NP films reduced burning time, delayed rate of heat release ignition and eliminated melt dripping of polyester fabric (Carosio, Laufer et al. 2011). Implication of nanoparticles such as nano-clay (Najarzadeh and Montazer 2023), carbon nanotubes(Qi, Cai et al. 2023) and silica NPs (Zhang, Li et al. 2023) on the flame retardant properties of various textile fibers were reported. Silica NPs and cross-linking agents were applied to cotton to make a hydrophobic surface. The thermal properties and flame retardation of the coated fabrics increased due to the high heat resistance, heat protection effect, and mass transport limitation of the silica NPs in the coating (Norouzi, Zare et al. 2015).

Bagasse, a biodegradable industrial waste obtained during sugar manufacturing process, is treated and utilized for paper manufacturing (Rana, Malik et al. 2021) as well as fuel generation (Carvalho, Segundo et al. 2019). It is a rich source of silica having potential for green synthesis of silica NPs. Literature survey reveals synthesis of SNPs from sugarcane bagasse (Athinarayanan, Periasamy et al. 2017, Mohd, Wee et al. 2017) and bagasse ash (Boonmee, Sabsiriroht et al. 2019, Falk, Shinhe et al. 2019). In the proposed study, sugarcane bagasse is used as a natural source of silica NPs. Synthesis via this approach is beneficial due to use of fewer chemicals. In addition, metallic contaminants are eliminated which leads to production of high purity silica NPs. Therefore, the objective of proposed study was to develop environmentally friendly silica NPs using sugar industry waste. The synthesized nanoparticles were characterized by Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM) and X-ray diffraction (XRD). Applicability of the synthesized nanoparticles was demonstrated in terms of thermal stability and antibacterial property by investigating the untreated and silica NPs treated cotton fabric. Furthermore, greenness evaluation of the proposed method for the synthesis of silica NPs was carried out by using Analytical Eco-Scale tool.

# METHODOLOGY

#### MATERIALS AND REAGENTS

The plain-weave 100% cotton fabric (1 mm thickness, 160 (gram m<sup>-2</sup>) was purchased from local market of Karachi. Sugarcane bagasse was obtained from local cane juice vender in Karachi. Hydrochloric acid, sodium hydroxide, nitric acid and ethanol were purchased from Merck. Darmstadt Germany used as received without further purification. Distilled water used throughout the experiments was prepared fresh in laboratory using deionizer (Stedec CSW 300).

#### SYNTHESIS OF SILICA NPS

Sugarcane bagasse was soaked in distilled water for 12 hours, followed by extensive cleaning with distilled water to remove grime and soil. It was then dried and pre-treated with 1N HCl on water bath at 75°C. The content was then filtered, washed to remove metallic ion followed by drying at dried at 90°C. Into 8gram sugarcane bagasse (pretreated), 250 mL of 1M NaOH was added followed by heating on water bath at 90°C for an hour. The obtained sodium silicate solution was then filtered and vigorously agitated at for 45 min. While whisking vigorously, nitric acid was steadily added into the solution until the pH of the solution reached 8.0. Then 20 mL ethanol was added with continuous stirring till further 20 min. The contents were then centrifuged for 20 min at 4000 rpm. The resultant precipitates were separated, washed with distilled water and kept in furnace at 600°C for 30 min. The obtained nanoparticles were then characterized by FTIR, XRD and SEM analysis. For comparative studies, procedure was followed for synthesis of silica NPs using 8 mL of commercial grade sodium silicate.

#### CHARACTERIZATION OF SILICA NPS

FTIR spectra were carried out on the Nicolet iS10 Mid-Near Infrared FTIR spectrometer with ZnSe ATR Crystal and recorded on OMNIC 9.2.46 software. X-ray diffraction analysis was performed on X'Pert PRO loaded with software X'Pert PRO high score. Surface morphology of synthesized nanoparticles was observed using a scanning electron microscope (Jeol JSM-6380A, Japan) at x18000 and x30000 magnification.



FIGURE 1. Screen printing of silica NPs on cotton fabric preparation<sup>1</sup>, incorporation<sup>2</sup> on mesh, application<sup>3</sup> of coating paste on cotton fabric and dragging and squeegee of fabric for uniform application<sup>4</sup>

## SILICA NPS LOADING ON COTTON FABRIC

Before being used, cotton fabric was washed and dried. Cotton fabric samples ( $5 \times 8$  cm) were treated with coating solutions containing Silica NP, binder (5%) and thickener (2%). Manual out- of-contact screen printing method was applied to incorporate synthesized SNPs in different ratios i.e. 1, 2 and 3% on 100% cotton fabric (Figure 1). The treated fabric samples were then dried in oven (UN30) Memmert GmbH & Co. KG at 150°C for 5 min.

# THERMAL STABILITY ANALYSIS

Thermal stability analysis and sustainability of treated and untreated cotton fabric was investigated by physical method. Analysis was performed by introducing the silica nanoparticle treated and untreated cotton fabric separately in to boiled water placed in a beaker covered with aluminum foil to entrap the heat inside. The change in temperature was recorded after every 5 min till 45 min. Analysis was performed in triplicate.

# ANTIMICROBIAL ACTIVITY OF SILICA NPS LOADED COTTON FABRIC

The antimicrobial activities of silica NPs loaded cotton fabric were assessed by disc diffusion AATCC-147 test method. The untreated cotton fabric was used as a control.

# **RESULTS AND DISCUSSION**

### FOURIER TRANSFORMS INFRARED SPECTROSCOPY (FTIR)

Bonding in silica NP was evaluated at 1000-4000 cm<sup>-1</sup> wavenumber against up to 95% transmittance. The FTIR spectra of synthesized SNPs exhibited characteristic peak at 791.01 attributed to vibration of Si-O and the absorption band at 1072.48 cm<sup>-1</sup> owing to asymmetric vibration of Si-O-Si confirming the formation of SNPs. FIGURE 2 represents FTIR spectra of Silica NPs synthesized by green method.



FIGURE 2 FTIR of silica NPs synthesized by green method

X-RAY DIFFRACTION (XRD)

Fingerprint of periodic atomic arrangement of synthesized material was examined by the X-ray diffraction pattern (See Figure 3). Card number 36-1451 is exercised guided by the Joint Committee on Powder Diffraction Standard (JCPDS). Examined spectra indicate well-defined peaks of Silicon Oxide. This shows that the synthesized solids are crystalline. Traditionally, particle size effects have been blamed for the broadening of peaks in solid XRD patterns. Debye Scherrer equation (Amir, Hasany et al. 2023) was used to calculate the mean crystallite size (19.79 nm) of powder Silica NP sample at the full width half maximum (FWHM) of the diffraction peaks of 2 $\Theta$  positions.



FIGURE 3. XRD pattern of silica NPs synthesized by green method

## SCANNING ELECTRON MICROSCOPY (SEM)

Figure 4(a) and 4(b) evident that SEM present clear, high-resolution image of the size and surface texture of plastic-

like particles, allowing researchers to discriminate NPs from interfering particles in the environment matrices. Particle size obtained from SEM 40±2nm. Spherical shaped silica NPs are obtained.



FIGURE 4 (a). SEM images of silica NPs synthesized by green method at X18000 magnification (b). SEM images of silica NPs synthesized by green method at X30000 magnification

#### GREENNESS EVALUATION

Analytical Eco-scale approach was used to evaluate the greenness of the proposed method to develop silica nanoparticle. According to this approach, the method is considered to be green which involves benign reagents and solvent with < 0.1 kwh power consumption per sample, without affecting environment and generating waste. On analytical eco-scale, the score of ideal method is hundred. Penalty points are allocated on deviation from ideal conditions which are subtracted from 100. Resultant score > 75 represents remarkable green method, score >50represents an acceptable green method, whereas a score <50 indicates a poor green method (Gałuszka, Migaszewski et al. 2012). The proposed method collected 24 penalty points, thus analytical eco-scale score is 76 confirming the greenness of the proposed method for silica nanoparticle synthesis (See Table 1).

TABLE 1. Greenness evaluation of proposed method forsynthesis of silica NPs using Analytical Eco-scale Tool

S.No	Parameters	Penalty points	
1	Reagents		
	Sodium hydroxide	6	
	Nitric acid	4	
	Ethanol	6	
2	Instrument	3	
3	Occupational Safety	0	
4	Waste	5	
		24	

# APPLICABILITY OF SYNTHESIZED SILICA NPS ON COTTON FABRIC

#### THERMAL STABILITY

Thermal insulation behavior of treated samples was assessed and compared with that of pure (untreated) cotton. Table 2 represents the thermal insulation observations of treated and untreated fabric. The untreated fabric showed initial temperature 80°C at 0 min. and the temperature drop was about 55°C, at time interval of 45 min owing to 31% thermal reduction, whereas, cotton fabric treated with varying concentration of silica NPs i.e. 1%, 2% and 3% separately showed 19 %, 24% and 28% decrease in temperature respectively. It was established that silica NPs coated fabric have substantial thermal stability as compared to the untreated fabric. The fabric treated with 1% silica NPs showed the thermal insulation behavior comparatively greater than that treated with 2% and 3% silica NPs may be due to clustering (Fanglong, Qun et al. 2016, Zhang, Li et al. 2023).

TABLE 2. Thermal Insulation observations of treated and untreated fabric

S.No	Time	Untreated fabric (°C) -	Treate	Treated fabric (Mean (°C))		
	(min)		1%	2%	3%	
1	0.0	80	80	80	80	
2	5.0	75	77	77	76	
3	10.0	73	74	74.5	73	
4	15.0	70	71	72	70	
5	20.0	68	69	71	69	
6	25.0	65	68	67	66	
7	30.0	64	66	66.5	65	
8	35.0	61	65	65	62	
9	40.0	58	64	62	61	
10	45.0	55	64.5	61	58.5	

#### ANTIBACTERIAL ACTIVITY

Disc diffusion method for the evaluation of antibacterial activity (bacteriostatic activity; inhibition of multiplication) was followed by AATCC-147 test method. In this test used gram positive Staphylococcus Aureus (American type culture collection no 6538) and gram negative Klebsiella Pneumoniae (American type culture collection no 4352). Figure 5 represents confirmatory test as positive control (red mark)  $\rightarrow$  antibiotic. In clockwise manner, next mark shows negative control  $\rightarrow$  ethanol (solvent). Next mark shows the solution of silica NPs in ethanol. The highest increase in antibacterial activity is seen in solutions containing silica NPs as compared to the ethanol solution (Imam and Abdelrahman 2023, Kodaloglu and Demiralay 2023).



FIGURE 5. Antibacterial activity of treated cotton fabric by disc diffusion method

# CONCLUSION

The cost-effective, 76 green profiling score by Analytical Eco-Scale tool, green silica NPs have been synthesized using sugarcane bagasse and characterized by FTIR (absorption band at 1072.48 cm<sup>-1</sup>), XRD (mean crystallite size (19.79 nm) and SEM ( $40\pm2nm$ ). Applicability of synthesized NPs on cotton fabric shows improved thermal stability and antibacterial property at 1% against untreated fabric. It is concluded that proposed treatment can be employed in manufacturing thermally stable antimicrobial textiles.

#### ACKNOWLEDGMENT

Present sincere thanks to NED University of Engineering and Technology, Pakistan for kindness and laboratory work support.

# DECLARATION OF COMPETING INTEREST

None

# REFERENCES

- Amir, M. et al. 2023. "Modification of bentonite nanoclay for textile application." *Polimery* 68(2): 79–85.
- Athinarayanan, J. et al. 2017. "Synthesis and biocompatibility assessment of sugarcane bagassederived biogenic silica nanoparticles for biomedical applications." *Journal of Biomedical Materials Research Part B: Applied Biomaterials* 105(2): 340– 349.
- Bablu, M. A. and J. M. Manimala 2022. "Mechanisms of ballistic performance enhancement in silica nanoparticle-treated kevlar fabric." *Journal of Composite Materials* 56(14): 2253-2266.
- Bhattacharjee, S. et al. 2020. "Nanoparticles incorporated graphene-based durable cotton fabrics." *Carbon* 166: 148–163.
- Boonmee, A., et al. 2019. "Preparation and characterization of rice husk ash for using as a filler in natural rubber." *Materials Today: Proceedings* 17: 2097–2103.
- Carosio, F. et al. 2011. "Layer-by-layer assembly of silica-based flame retardant thin film on PET fabric." *Polymer degradation stability* 96(5): 745–750.
- Carvalho, M. et al. 2019. "Carbon footprint of the generation of bioelectricity from sugarcane bagasse in a sugar and ethanol industry." *International Journal of Global Warming* 17(3): 235–251.

- Falk, G. et al. 2019. "Synthesis of silica nanoparticles from sugarcane bagasse ash and nano-silicon via magnesiothermic reactions." *Ceramics International* 45(17): 21618–21624.
- Fanglong, Z. et al. 2016. "Influence of nano-silica on flame resistance behavior of intumescent flame retardant cellulosic textiles: remarkable synergistic effect?" Surface Coatings Technology 294: 90–94.
- Gałuszka, A. et al. 2012. "Analytical Eco-Scale for assessing the greenness of analytical procedures." TrAC *Trends in Analytical Chemistry* 37: 61–72.
- Imam, M. S. and M. M. Abdelrahman. 2023. "How environmentally friendly is the analytical process? A paradigm overview of ten greenness assessment metric approaches for analytical methods." *Trends in Environmental Analytical Chemistry*: e00202.
- Kodaloglu, F. A. and H. Demiralay. 2023. "Application of grape seed coating for antibacterial cotton fabric." *Industria Textila* 74(3): 332–336.
- Liu, B. et al. 2015. "Synthesis and characterization of photoreactive silica nanoparticles for superhydrophobic cotton fabrics application." *Textile research journal* 85(8): 795-803.
- Mohd, N. K. et al. 2017. Green synthesis of silica nanoparticles using sugarcane bagasse. *AIP conference proceedings, AIP Publishing.*
- Najarzadeh, H. and M. Montazer 2023. "Impact of nanoclay on improving the properties of cotton/nylon fabric." *Applied Nanomaterials Smart Polymers* 1(1): 29–37.
- Norouzi, M. et al. 2015. "Nanoparticles as effective flame retardants for natural and synthetic textile polymers: application, mechanism, and optimization." *Polymer Reviews* 55(3): 531–560.
- Qi, L. et al. 2023. "Application of Silver-Loaded Halloysite Nanotubes in Flame Retardant and Smoke-Suppressive Coating for Polyester-Cotton Fabric." ACS Applied Materials Interfaces.
- Rana, V. et al. 2021. "Preparation of alpha cellulose from sugarcane bagasse and its cationization: Synthesis, characterization, validation and application as wetend additive." *International Journal of Biological Macromolecules* 170: 793–809.
- Zhang, X. et al. 2023. "Improved thermal stability and flame retardancy levels of cotton fabrics treated by GEL/AMP layer-by-layer assembly and silica gel composite coatings." Cellulose: 1–24.