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Multi-functional finishing of cotton fabric with Nanoparticles by sol-gel coating

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Abstract

Cotton fiber is the most widely used natural fiber owing to its superior comfort. Additionally, its abundant availability and decent mechanical and thermal characteristics give it an edge over its counterparts. However, improving its surface properties to endow desired functionalities for targeted applications remains a challenging task. Herein, we report the multifunctional finishing of cotton fabric using the sol-gel coating technique. For that purpose, a composite solution containing different ratios of silver, Titanium, and silica nanoparticles was prepared. The prepared solution was uniformly applied *via* dip-pad-dry-pad-cure process and crosslinked with cotton fabric using citric acid as a cross-linking agent. Sol-gel coated fabric exhibited decent antibacterial, high ultraviolet (UV) resistance, considerable self-cleaning and soil release, and excellent water repellency. Furthermore, sol-gel coated samples offered improved whiteness and durable performance against multiple washing cycles.

Keywords: cotton fabric, sol-gel finishing, antibacterial activity, UV resistance, soil release, water repellency

Introduction

Recently, there has been growing concerns about the hazards caused by the textile industry to the environment. Use of nanotechnology in the textile industry offers a potential solution to address these concerns (Afzali & Maghsoodlou, 2016). The development of UV blocking, anti-bacterial and self-cleaning textiles using **Feb. 2022** nanotechnology have gained importance in recent years for posing no significant threat to the human health and environment (Yuzer *et al.*, 2022).coated cotton fabric with titania nanoparticles (TNPs) doped by Cu(II) for designing an antibacterial and self-cleaning textile fabric (Pakdel, Wang, Kashi, Sun, & Wang, 2020). Pakdel et al. (2020) used TNPs and hollow glass microspheres for producing Multifunctional cotton fabric with UV protection, flame retardancy, thermal insulation, etc.(Zahid, Rashid, Akram, Rehan, & Razzaq, 2018) Zahid (2018) reported the simple and low-cost production of functional textile fabric with multifunctional properties by using TNPs doped with Manganese (Mn) (El-Naggar, Shaheen, Zaghloul, El-Rafie, & Hebeish, 2016). El-Naggar et al. (2016) investigated the use of TNPs for producing the antibacterial cotton fabrics. They achieved the excellent durable antibacterial performance with 95% bacterial reduction for up to 20 washing cycles.

Various metals and metal oxides including CuO, ZnO, Fe_2O_3, etc., in nanoparticles form offer considerably high antibacterial performance (Abidi, Hequet, Tarimala, & Dai, 2007). Amongst various nanoparticles, Cu, Zn and Ag nanoparticles have been extensively studied to explore their potential for realizing the antibacterial textiles. However, sliver nanoparticles (AgNPs) have been the most investigated nanoparticles for designing materials for keyboards, wound dressings and biomedical applications because of their excellent biocompatibility and nontoxic nature. Reported outcomes of AgNPs exhibit excellent and stable antibacterial performance (El-Naggar *et al.*, 2016; Tang *et al.*, 2015; Zhao, Milanova, Warmoeskerken, & Dutschk, 2012). Moreover, their easy and sustainable fabrication, and reasonable production cost has attracted massive attention around the globe.

Another most widely used nanoparticles are TNPs and silica nanoparticles (SNPs). They have been extensively studied due to their stable and non-toxic nature along with relatively low production and application cost. Besides that, oxidation reduction potential of TNPs to eradicate the organic stains attracts the attention of researchers (Montazer, Alimohammadi, Shamei, & Rahimi, 2012; Roe & Zhang, 2009; Stanssens *et al.*, 2011).Moreover, TNPs performance relies on their structural, morphological, and optical properties. It has been reported that at high temperature treatment enhances the photocatalytic performance of TNPs, however such treatments are unsuitable for Textile Applications(Wong, Yuen, Leung, Ku, & Lam, 2006). Therefore, it has been suggested to add metals or metal oxides into the bulk of a photocatalyst inhibits reunion of excited electron-hole pair resulting in reduced bandgap energy, and ultimately leads to an enhanced photocatalytic performance. On other hand, Silica-based nanomaterials possess excellent biocompatibility and tunable nanostructures. Furthermore, SNPs have been widely used for producing super hydrophobic fibrous materials by turning their surface roughness (Montazer *et al.*, 2012; Roe & Zhang, 2009).

Herein we report a strategy to develop the multi-functional finishing (i.e., antibacterial, UV resistance, self-cleaning, water repellency, and soil release) of cotton fabric by sol-gel technique. The cotton fabric, the most widely used textile fabric, was selected because of its easy and abundant availability. The AgNPs, TNPs and SNPs were used for creating the functionalities such as antimicrobial, self-cleaning, and hydrophobic properties, respectively. The sol-gel coating process was selected because it renders better physical and chemical properties in the fabric. …The factors such as temperature, concentration and time were analyzed to achieve the optimized conditions (Brinker & Scherer, 2013; Chinta, Landage, & Swapnal, 2013; Guo, Wen, Peng, & Guo, 2017; Moafi, Shojaie, & Zanjanchi, 2010; Tomšič 2008; Ulrich, 1988). The synthesized fabric was carefully assessed, to achieve optimum multifunctional performance.

Experimental Section

Materials

Woven cotton fabric with GSM of 181 was supplied by Yunus Textile Mills (Pvt.) Ltd., Pakistan. Silica nanoparticles (SNPs, Aerosil® 200) and titania nanoparticles (TNPs, Aeroxide® TiO_2P25) with average particle diameter 15 ± 5 nm and 25 ± 5 nm, respectively, were procured from, Evonik Germany Ltd.,. Sliver nanoparticles (AgNPs) were obtained from Rudolf Pakistan (Pvt.) Ltd. Commercial hydrophobic Dodecyl-tri-methoxysilane (DTMS, KH-580) was purchased from Hangzhou Feidian Chemicals China. Ethanol, citric acid (CA), and sodium hypophosphite (SHP) of analytical grade were used. All the chemicals were used as received without any further purification. Deionized water was used in all the experiments.

Application Procedure

First, SNPs were dispersed in water under rigorous magnetic stirring. Afterwards, TNPs and AgNPs were added to SNPs dispersion, and composited dispersion was stirred magnetically for 30 minutes followed by sonication treatment of 30 minutes to ensure uniform dispersion of NPs. Another solution was prepared by dissolving CA and SHP in water. Later, the two solutions were mixed together and sonicated for 1 hour. The cotton fabric samples were immersed in the mixed solution for 3 minutes and padded with 100% pick-up on horizontal padding machine (Mathis Switzerland) and dried in mini dryer machine (Rapid Taiwan) at 100°C for 180 seconds. Then dried samples were re-padded at 100% pick-up with DTMS solution dissolved in ethanol followed by curing at 150°C for 180 seconds in laboratory dryer, as reported in our earlier work (Jhatial, Khatri, Ali, & Babar, 2019).The complete process of solution preparation and application is given in the scheme 1.

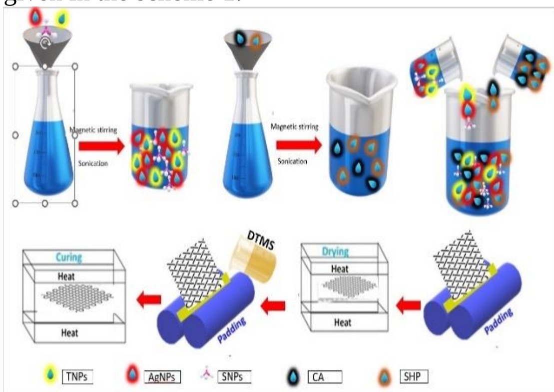

Fig 1. Illustration of the Solution preparation and application procedure of NPs.

Characterization

The antibacterial activity of the coated and control samples were investigated using disc diffusion methods. One of the most widely used bacteria family *E-Coli, gram negative strain 25923* was used for analyzing the bacterial growth. The coated and uncoated (control) fabric samples with 6 mm diameter were used. Before antibacterial testing samples were placed in an autoclave for sterilization (121 °C, 15 minutes) under constant pressure (Table 1. for Further details).Sterilized samples were then subjected to incubation at 37 °C for 24 hours and then inhibition zones were carefully observed.

Table 1: Test variables	
Test organism	***E. coli*** **(Gram-negative bacteria)**
Dilution medium used	Saline water
Agar used	Eosin methylene blue (EMB) agar
Method of sterilization	Autoclaving at 121 ^{0}C for 15 min
Sample dimension	6 mm diameter disc cut from large fabric
Number of swatches per sample	3
Control	100% mercerized cotton without AgNPs
Test martial	100% mercerized cotton with AgNPs
Microbial incubation time	24 hours
Deviation from standard test method	The average value was taken

resistance against full range of UV radiation of the sol-gel coated cotton samples. For observing the self-cleaning characteristics, the coated samples (2cm × 8cm) were stained with methyl orange dye (0.5% w/v), and then irradiated with high intensity Xenon light for 24 hours. Half of each stained sample was exposed directly to UV light, whereas other half of samples was covered with opaque material. The change in shade was assessed by using Blue wool reference scale. Moreover, water repellency of samples was investigated by following the AATCC Spray Test Method 22 [27]. Samples were exposed to water spray at 45o angle and assessed with standard qualitative photo scale. AATCC Test Method 130 standard method was followed to evaluate the soil release performance. The durability of synthesized fabric sample was examined using Gyro Wash (James H. Heal, UK) according to AATCC-61-2013-2A.

The breaking force of pristine and coated fabrics was measured according to ISO 13934-1:2013 Test Method using Titan universal tester (Titan 3-910, UK).The air permeability of fabrics was analyzed *via* Air Permeability Tester (MO12S SDL Atlas, UK) according to ASTM D 737-18 standard test method. The Shirley stiffness tester was used to determine the stiffness of fabric samples following standard ASTM D 1388. sample dimensions of width (25 mm) and length (200 mm) were used to assess stiffness in both warp as well as weft directions. Finally, the surface morphology of the pristine and sol-gel coated samples were determined by scanning electron microscope (SEM, JSM 6380LV, JEOL Japan) at magnification of 500x and accelerating of 5kV voltage.

Results and Discussion

Antibacterial Performance.

To observe antibacterial activity, cotton fabric samples were sol-gel coated with AgNPs, it was noticed that sol-gel coated cotton specimen exhibited maximum inhibition zone of 12 mm at 0.5% concentration (Fig: 02). This decent antibacterial activity of the designed sol-gel coated cotton fabric may be attributed to the release silver ions from the nano-coating of AgNPs. Furthermore, coated samples displayed minor change in the antibacterial activity when subjected to 05 rigorous commercial washing cycles. This minor change in the antibacterial performance of the sol-gel cotton fabrics was credited to the loss of some of the AgNPs from the surface. Fig. 3 demonstrated the impact of citric acid concentration on antibacterial activity and wash durability of cotton fabric. Generally, it was shown that an increase in the citric acid concentration caused an increase in the zone of inhibition simultaneously. It was observed that E. coli did not show any significant growth in acidic medium. However, the apparent growth was observed in mild alkaline medium (pH 6-8). Maximum inhibition (i.e., 12 mm) zone was observed at 8% concentration of citric acid due to enhanced number of ester bond formation between

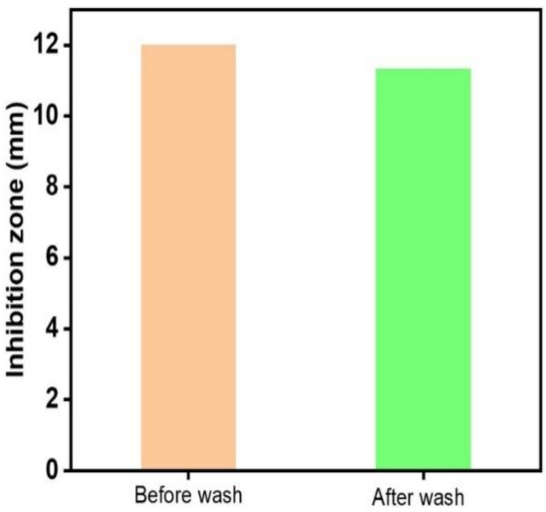

Fig 2. Before and after wash; antibacterial activity of sol gel coated cotton fabric against

fiber and silver nanoparticles. Currently, increasing use of functional textiles which need no washing/laundering have resulted in enhanced demand for self-cleaning textiles(Huang, Gurney, Wang, & Liu, 2018; Pakdel & Daoud, 2013).

UV resistance and self-cleaning Performance

UV radiation is believed to be harmful for humans. Consistent and prolonged exposure to UV light can damage eyes, cause erythema and even sunburn (Ren *et al.*, 2018; Xin, Daoud, & Kong, 2004). Corresponding samples are depicted in Fig: 4. Self-cleaning behavior of the resultant samples was evaluated using standard greyscale for staining.

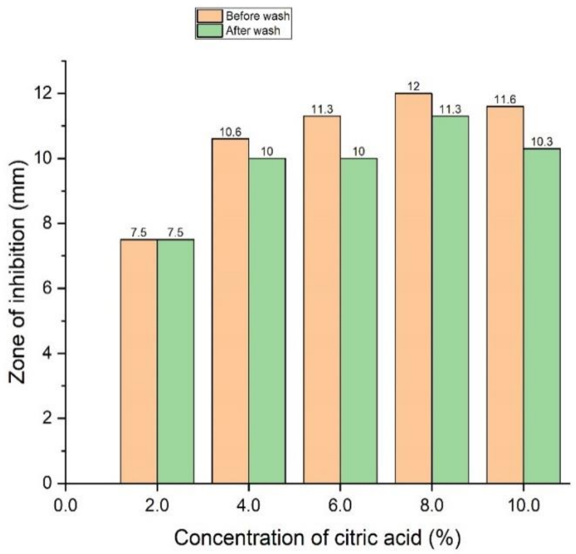

Fig. 3: Effect of citric acid concentration against bacterial activity and wash durability

performance even after 5 cycles of commercial washes. Moreover, UV radiations can also wreck the textile fabrics and ruin the color shades of textiles dyed with different dyes (Yang, Zhu, & Pan, 2004). Therefore, it is essential to design fabrics capable of blocking maximum UV radiations. Therefore, to analyze the UV resistance, cotton fabric samples with various concentrations of TNPs (0.5-2.5 wt./vol %) were irradiated with UV radiations range from 200-400 nm. Compared to uncoated cotton sample, sol gel coated fabric transmitted lower UV radiations. It was observed that when concentration of TNPs increased transmittance of UV radiations in the resultant fabric sample declined. Further analysis of results revealed that sol-gel coated cotton fabrics exhibited maximum UV blockage measured by minimal transmittance of UVA radiations (i.e., 320-380 nm) at 2% concentration of TNPs (Table:2). Further increase in TNPs concentration resulted in reduced UV protection because of formation of floccules, indicating that the large aggregated TNP scatter less UV light than even distribution of TNP (Abidi *et al.*, 2007).

Table: 2. Effect of TNPs concentration on UV transmittance

TNP concentration	UV Transmittance percent (%) on the wavelength (nm)							
	260 nm	280 nm	300 nm	320 nm	340 nm	360 nm	380 nm	400 nm
Uncoated	107.88	98.49	98.28	100.21	100.68	100.34	100.32	99.56
0.5%	78.66	68.9	67.6	65.56	65.86	67.8	73.27	74.99
1%	87.52	66.13	65.31	61.14	60.54	61.85	65.77	76.32
1.5%	64.07	46.29	44.61	44.84	43.29	44.63	48.08	50.68
2%	51.82	33.42	32.25	42.03	39.71	39.94	44.51	47.12
2.5%	47.07	38.21	32.79	38.16	39.23	41.82	46.35	49.76

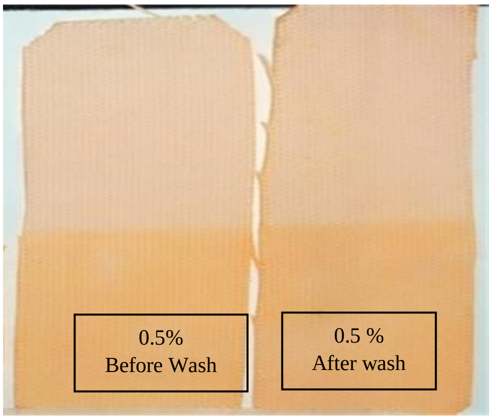

Fig. 4: Stain removal of coated samples at a varying concentration of TNP

Optimized sample exhibited the stain degradation 4/5 rating on greyscale at 2% concentration of TNPs. Moreover, it was observed that optimized sol-gel coated samples showed no significance change in performance indicating stable self-cleaning

Water repellency and soil release of sol-gel coated fabrics

It is well-known that water repellency is very essential for efficient self-cleaning and soil-release performance, and hydrophobic coatings can significantly alter the water absorbency as well as water repellency of textile fabrics (Chinta *et al.*, 2013). Therefore, water repellency of the sol-gel coated fabric samples was analyzed using SDL spray tester and the results of corresponding samples were carefully assessed using standard photographic scale. As expected, pristine cotton fabric sample showed nearly no water repellency and readily absorbed the water droplets when water spray was showered on it. Whereas sol-gel coated fabric samples to subject to water spray, water could easily roll of the sample surface. It was noticed that fabrics coated with 3% concentration of DTMS offered optimal water repellency with 90 rating. Further increment in the DTMS concentration displayed no significant change in the water repellency of the coated samples(Brinker & Scherer, 2013).

Like self-cleaning, there is rising demand for textiles

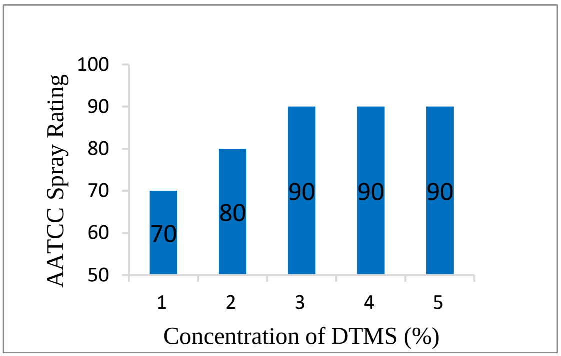

Fig: 05 Effect of DTMS Concentration

having soil-release feature. Textile and material scientists are trying to make it an integral and complementary feature of home textiles. Table 3 depicts the outcomes of soil-release performs of pristine and sol-gel coated textiles. It was witnessed that simultaneous increase in the concentrations of SNPs and TNPs resulted in improved soil-release performance of the corresponding sol-gel coated textiles. Optimum soil-release performance (i.e., rating4) was achieved for sol-gel coated fabrics at 1% and 2% concentrations of SNPs and TNPs, respectively (Pakdel & Daoud, 2013).

Table 3: Effect of SNP and TNP on soil release

S.No	Conc: of SNPs	Soil Release Rating	S.No	Conc: of TNPs	Soil Release Rating
1	0.5%	2	1	0.5%	3
2	1 %	3	2	1 %	3
3	1.5%	2-3	3	1.5%	4
4	2 %	2-3	4	2 %	4
5	2.5%	2-3	5	2.5%	2

Comparison of sol-gel coated and uncoated cotton fabric

Compared to uncoated cotton fabrics, all the sol-gel coated fabrics exhibited better for performance in all the desired characteristics. Furthermore, sol-gel coated were then subjected to multiple industrial washes to evaluate the durability of sol-gel coating.

Table: 04. Comparison of uncoated and coated fabric properties

Properties	**Uncoated Sample**	**Optimize Sample**	**After washing**
Antibacterial (zone	0	11 mm	10.98 mm
UV transmittance %	99.56	47.12	58.67
Water repellency	AATCC 50	AATCC 90	AATCC 70
Soil release	1-2 Rating	4 Rating	2-3 Rating
Tensile strength	744.8 N	478.5 N	344.7 N
Whiteness CIE	64.60	67.26	37.47
Bending length (Warp direction	5.5 cm	8.3 cm	6.6 cm
Bending length (Weft direction)	5 cm	5.5 cm	5.3 cm
Air permeability (S)	200 ml/s	185 ml/s	190 ml/s
Air permeability (D)	120 ml/s	67 ml/s	70 l/s

It was found out that sol-gel coated fabrics offer considerably high antibacterial, UV resistance, self-cleaning and soil-release etc., performance even after 5 industrial washes. No significant change in antibacterial performance was observed before and washing, indicating that coated samples offered durable antibacterial performance. Minor increase in UV transmittance, self-cleaning, and soil-release performance displays that UV resistance of the fabrics deteriorated after washing because some of TNPs and SNPs were removed because of intensive mechanical agitation during laundering process (Teli & Annaldewar, 2017).

Surface morphology of cotton fabric

Fig. 6 shows the SEM photographs of cotton samples. It was observed that fibers of uncoated cotton fabric showed uneven and convoluted fibers with smooth surface (Fig. 6A and 6B).However, sol-gel coated fabric displayed relatively rough fiber surface which was credited to the presence of nanoparticles aggregates throughout the fiber length (Karimi, Mirjalili, Yazdanshenas, & Nazari, 2010). It was further noticed that sol-gel coated fabrics retained their roughness on the surface of fibers after multiple industrial washing which validates the durability of sol-gel coating on the fabric surface(Huang *et al.*, 2018) (Fig. C).

Conclusion

In summary, a combination of AgNPs, TNPs and SNPs was successfully crosslinked on the surface of woven cotton fabric by sol-gel (dip-pad-dry-pad-cure) method using citric acid as crosslinker. Excellent antibacterial performance was achieved at 0.5% concentration of AgNP. Moreover, high UV resistance (i.e., UV transmittance of <50%) and considerably high self-cleaning with rating of 4/5 (greyscale for staining) was obtained at 2% concentration of TNPs. Additionally, moderate soil-release performance and water repellency with rating of 3 (greyscale) and 90 (photographic scale) were observed at 1% and 3% concentration of SNPs and DTMS, respectively.

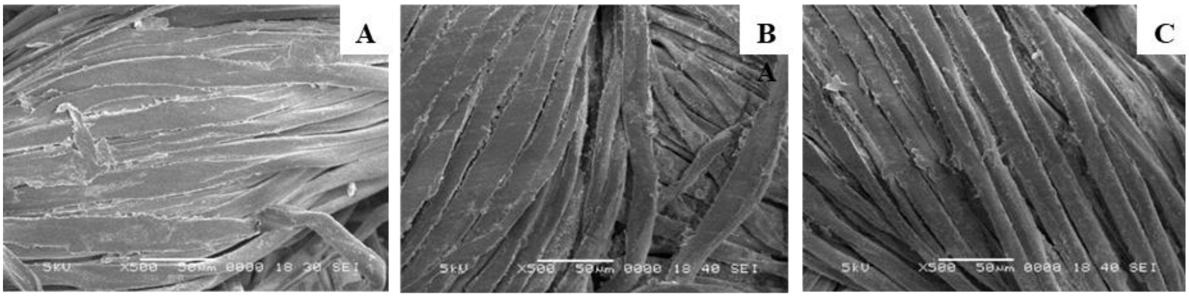

Fig.6. SEM photographs of cotton fabric (A) uncoated (B) coated with sol-gel, and (C) washed

Finally, before and after industrial washing of sol-gel coated samples confirmed the durability of sol-gel coating for up to 5 industrial washes. The tensile strength of sol-gel coated fabric was improved, and negligible change in air permeability, softness of fabric reveals that inherent comfort properties of cotton remained unaffected after sol-gel coating.

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