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Research Article

Nano-Coating on Different Patterned and Unpatterned Light-Weight and Delicate Curtain and Lacy Fabrics; Multifunctional Features and Comparing ICP-OES and UV-vis Spectroscopy to Determine Washing Fastness

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Abstract

Regarding the wide range of application of different patterned and unpatterned light-weight and delicate fabrics for designing curtain, it is worthwhile to evaluate novel technologies for promotion their practical properties without impact on their appearance. This paper aims at nano-finishing of curtain and lacy fabrics and inspecting the resulting properties. To this end, treatment was done using a commercially available colloidal solution of titanium dioxide and cross-linkable polysiloxane resin (XPs) as an excellent mass production promising technique. Then, different properties such as surface friction, hydrophobicity, water drop contact angle, washing fastness, stain repellency, air permeability, abrasion resistance, bending length, wrinkle recovery angle and resistance to sunlight degradation were studied. Based on the final results, a desirable super-hydrophobicity on samples was achieved by a significant droplet absorption time up to 6 hours and a proper droplet contact angle of 150°. A good stain-repellency was confirmed after standard drops of methylene blue slipped on the samples with no trace left behind. Based on the results, the treatment serves as a protective layer against sunlight. Nano-coating associated with XPs resin has no statistically significant effect on air permeability, abrasion resistance and bending length. However, it improved wrinkle recovery angle and balanced surface friction. XPs coating was able to save the nanoparticles on the surface in a washing process after 20 cycles of laundering. Inductively coupled plasma optical emission spectroscopy (ICP-OES) and ultraviolet/visible spectroscopy were used as two analytical techniques to determine nanoparticles concentrations leached out in washing effluents. The results of these two techniques in evaluating washing fastness were compared to each other.

Keywords: Lace; Curtain; Nano-functionalization; Light fabrics; Hydrophobicity; ICP-OES-based washing fastness; contact angle

Introduction

Curtain and lace, which are light-weight, and delicate fabrics, are widely used for decorative purposes. Lace has also other applications such as pavilions and mosquito nets and is placed in front of windows as a first curtain layer to design or protect the main curtain against sunlight. Due to maintenance problems such as washing and cleaning, modifying the surface of fabrics by applying some useful features such as stain-repellency, hydrophobicity and sunlight resistance seems to be of benefit. However, treatment of delicate fabrics is particularly more sensitive than that of other types of fabrics. Moreover, this process may face some difficulties due to the especial patterns on the fabrics which should be also investigated. Therefore, this study aims at durable treatment on these fine and sensitive kinds of fabrics using nanomaterials and XPs resin, as the most industrially-compatible and the best textiles modification method.

In fact, application of nanomaterials has largely increased due to their unique characteristics [1-10]. This has encouraged researchers to extensively study this topic in recent publications. Using nanostructures for modification of textiles [11-17] has caused great promotions in many applications. Among nanomaterials, titanium dioxide is used extensively due to its remarkable features [18-22]. However, applying these materials in the finishing process is accompanying with some problems.

Generally, nanoparticle stability on textiles is poor. Several methods have been proposed to solve this problem; for example, pretreatment *via* physical or chemical phenomena like Ultraviolet (UV), plasma [23-25], laser [26] or oxidants [27] to create some radical or functional groups on the surface. Incorporation of nanoparticles into a polymer melt in the extruder and applying nanocomposite for trapping nanoparticle in a polymeric matrix is another technique used in some research works [28, 29].

Another way considered more effective is utilizing resin for consolidation of nanomaterials on textiles, such as using acrylic binder [30] or polyurethane binder [31]. However, most resins are

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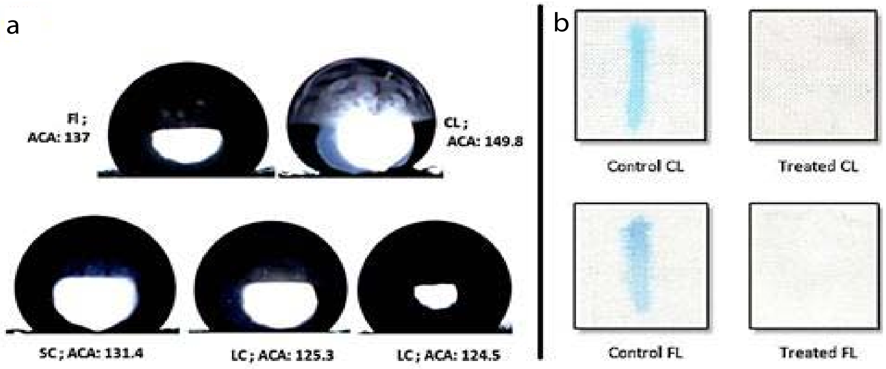

Figure 1: Hydrophobisity of Sample; a) The water droplet status on "TiO_2+XPs" samples; Average Contact Angle (ACA) has been reported for each sample, b) Effect of slipping of methylene blue drop; left samples are control and right samples are treated samples.

not suitable because applying them on the surface decreases clothing comfort, air permeability, draping, abrasion resistance, and softness. It also poses some other problems. For instance, it requires curing, demands an acidic condition damaging the substrates during the curing process, and needs neutralization of pH after the treatment [32].

Consequently, if an industrially adaptable process can be offered, it is worth to be investigated for different types of fabrics. The results will provide a vital perspective to researchers and specially industries to any future plan on the mass production. According to our search, there is no any report on nano-coating of the patterned and unpatterned light-weight and delicate curtain and lacy fabrics and its effect on different properties of treated fabrics in the literature.

This article aimed at finishing light-weight and delicate fabrics with two issues in focus; lace and curtain fabrics. To cover different types of fabric structures, three curtain samples and two non-patterned lacy samples were chosen and treated by nano TiO_2 and XPs resin, respectively. Then, their physical and mechanical properties were evaluated, reported and discussed.

Experimental Section

Materials and methods

Patterned and unpatterned curtain and lacy fabrics: Several Polyester fabrics (PET) with an area weight less than 100 g/m^2 were used in this study. They were selected due to presence of these useful fabrics in new-fashioned textiles industries as well as home textiles usages, etc. The details about the samples are given in (Table 1).

Treating materials: Water-based nano-sized colloidal TiO_2 Aerodisp W 740X (40% TiO_2 P25 nano titanium dioxide in water and polysiloxane CT 208 E emulsion) were kindly provided by Evonik Degussa Corporation and Wacker Finish, respectively. Evaluating the particle size by zeta-sizer has confirmed the size of about 85.95 nm for TiO_2 nanoparticles [5].

Samples preparation: The cut samples were exposed to a pre-wash preparation to remove any impurities such as dust particles from their surfaces. A washing bath was prepared with 1g/L of a anionic detergent with a liquor-to-goods ratio (L:G) of 80:1 at 50°C. The samples were immersed in the bath for 20 minutes and rinsed completely. To ensure removing the excess detergent, the samples were rinsed twice more with distilled water at 50°C for 5 minutes.

Fabric treatment: The nano-sized colloidal TiO_2 and the polysiloxane emulsion were diluted using distilled water at an ambient temperature (25°C) to achieve a specific concentration. All the pre-washed samples were immersed in the prepared bath (TiO_2) for 20 seconds and squeezed by a pad, down to a 100% wet pick-up. The padded samples were dried at 100°C in an oven for about 3 minutes.

A subsequent treatment bath was prepared with distilled water and a specific concentration of resin. All the pre-treated samples were treated in the same condition again but within a shorter immersion time (i.e. 3-5 seconds). An associated control sample treated with only XPs was also prepared. To facilitate reporting the results, all the samples were introduced by proper codes. The samples treated with both TiO_2 and XPs were subject to an after-wash process in the same conditions as mentioned for the pre-wash.

Test Methods

Water droplet absorption time

Water drops (10 microliter) were dropped from a distance of about 10 mm of the fabric surface, and its absorption time was recorded at a controlled temperature and relative humidity (20°C and 30%).

Water droplet contact angle

After releasing water drops on the sample surface, the droplet statue was examined by mean of a self-developed goniometer apparatus equipped with a high-resolution camera and a suitable lens [33] and the corresponding contact angles were measured manually by using MB-Ruler software.

Stain repellency

Stain repellency was evaluated according to the modified method AATCC 22 [32]. Stain drops of methylene blue (0.005%) were released on the samples at an angle of 45° with respect to the horizon. At the end, trace of stains on the samples was scanned and reported.

To quantify the results, another study was designed [34] and done. Accordingly, every sample was immersed into a methylene blue (0.005%) container lying on a vibrating plate (shaker) for 10 seconds at the rate of 60 rpm. The characteristics of color (L, a, b)

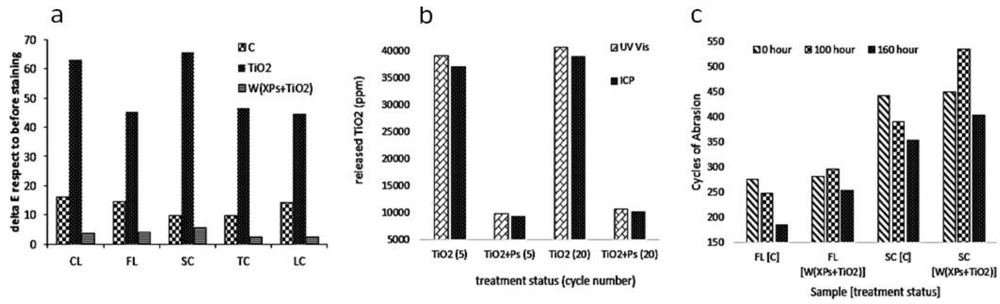

Figure 2: a) calculated color difference (ΔE) after staining as compared to same samples before staining, b) comparing the results of two effluent analysis techniques to determine released TiO_2 in washing effluents, c) sunlight resistance of samples (end points in abrasion resistance test).

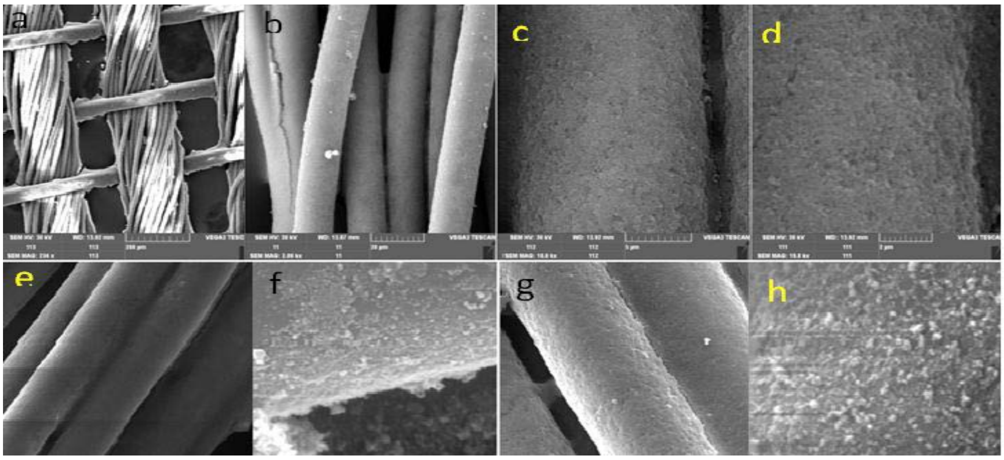

Figure 3: SEM micrographs of nanofunctionalized fabrics with different magnifications.
1st row a-d) TC, 2nd row e-f) FL and g-h) CL.

were extracted by an X-rite Spectrophotometer. The color difference between before and after staining was calculated for each sample by the following well-known equation:

$$\Delta E = [(L_2 - L_1)^2 + (a_2 - a_1)^2 + (b_2 - b_1)^2]^{0.5}$$

Washing fastness

Determination of titanium quantity, released in each wash, was considered as the most accurate measure of washing stability [32]. Washing test was done 20 times for every sample according to standard the AATCC 61(2A)-1996. Each sample was immerged in a washing solution (5 g/l of detergent with L: R=1:80) and shaken at 40°C for 40 minutes. Washing effluents of 1-5th and 6-20th with proper dilution factors were studied separately by ICP-OES (Perkin Elmer Plasma 400 Sequential ICP-OES Spectrometer) and a transmission spectrophotometer (UV mini-1240) in the ultraviolet region near visible (UV-vis.) through transition spectroscopy at λ_{max}= 318 nm. The released TiO_2 was calculated considering the dilution factors and calibration series. For FL samples, titanium quantity released in each wash was also determined by inductively coupled plasma spectrometry test ICP-OES, and the results compared to the data resulted UV-vis spectrometry.

Abrasion resistance test

This test was conducted according to the standard ASTM-D4966/D4970 using Shirley Martindale Abrasion Tester. The number of rub cycles until the first break point was considered as the standard end point.

Sunlight resistance test

The abrasion resistance of the samples exposed to the sunlight was considered as the criterion of sunlight resistance [29] and was compared with the primary sample. For this purpose, the samples were exposed to the sunlight of Yazd city, Iran, for 100 and 160 hours in summer during several days and at certain times of the day (from 10 a.m. to 4 p.m. in July, 2013). Then, abrasion resistance of each irradiated sample was determined according to the standard ASTM-4966/D4970.

Crease recovery angle

Crease recovery angle was determined by a Shirley Crease Recovery Tester according to the standard AATCC Method 66.

Surface friction

This test was done based on the standard test method BS3424 using a Shirley instrument. The results were reported as the static angle of surface drag (Ds) according to the standard test method.

Air permeability

The air permeability is one of the important features of delicate fabrics, especially lacy fabrics. To investigate the effect of nano-treatment on air permeability, a test was performed according to the standard BS-5636 on four folded samples under 1mm H_2O column

Table 1: Raw samples structures.

Sample code	Fabric type	Fabric texture	Warp yarn	Weft yarn	Pattern yarn
FL	Fine lacy	warp knitted	multi-filament		-
CL	Coarse lacy	warp knitted	multi-filament		-
SC	Striped curtain	woven	mono-filament	mono-filament	intermingled
TC	Tiled curtain	woven	multi-filament	mono-filament	multi-filament
LC	Limbed curtain	woven	mono-filament	mono-filament	multi-filament

Table 2: Water droplet absorption Time (s) of control and $XPs+TiO_2$ treated samples with 1wt% and 1.5 wt% of resin. Data has been reported in "Average (SD)" form.

Sample	Control	Treatment	Resin 1%	Resin 1.5%
CL	180 (11)	$XPs+TiO_2$	5484 (150)*	4080 (34)
		$W(XPs+TiO_2)$	26040 (2508)	3936 (20)
FL	456 (66)	$XPs+TiO_2$	3840 (252)	4200 (44)
		$W(XPs+TiO_2)$	21720 (1368)	4020 (52)
SC	8500 (59)	$XPs+TiO_2$	9480 (66)	3900 (0)
		$W(XPs+TiO_2)$	5280 (288)	4140 (66)
TC	660 (48)	$XPs+TiO_2$	5460 (114)	3660 (0)
		$W(XPs+TiO_2)$	4920 (384)	3480 (0)
LC	480 (42)	$XPs+TiO_2$	5220 (288)	4020 (24)
		$W(XPs+TiO_2)$	4200 (606)	3660 (30)

pressure.

Bending length

This test was done according to the standard ASTM-D1388 using a Shirley Stiffness Tester.

Scanning electron microscopy

Scanning Electron Microscope (SEM) micrographs were obtained using a Vega 3, Tescan scanning electron microscope with different magnifications.

Results and Discussion

Hydrophilicity-hydrophobicity

The Samples, treated with an optimal percentage of titanium dioxide (obtained in prior studies), were treated with different concentrations of XPs resin, and then the absorption time was considered as a hydrophobicity index. Overall, evaluation of the hydrophobicity of the samples treated *via* this method can be considered as a criterion for determining whether or not to form a thorough layer of resin on the surface [32]. Hence, an appropriate concentration of resin to form an optimal resin layer covering the surface should be determined. Therefore, hydrophobicity has an optimal point [32]. Actually, maximum hydrophobicity is obtained when a film of resin is formed on the surface. Incomplete coverage reduces hydrophobicity with respect to the optimal status. Similarly, the excess resin, due to rearrangement of hydrophilic groups, can also increase hydrophilicity. The appropriate resin concentration also depends on other treatments as well as the fabric structure. The study revealed that a 1% concentration of resin is the optimal amount to form a thorough layer on the surface (Table 2). In fact, the optimum concentration of resin was achieved at maximum hydrophobicity (Table 2). Considering achieving maximum droplet absorption time at the concentration of 1% in all the cases, this concentration was chosen as the selected concentration of resin to treat all the delicate fabrics.

Water droplet contact angle

Since the samples without resin had a rapid absorption (about 20s for untreated control sample and about 5s for only TiO_2 treated samples) water drops had a contact angle less than 90 degree and were not stable on the surface. Thus, the contact angle of the samples treated with resin was evaluated. Super hydrophobicity was obtained by reaching the contact angle up to over 130 degrees in the FL and SC samples and up to 150 degree in the CL sample. It has been realized that lowering the surface tension cannot increase contact angle more than 120° [35]. However, a hydrophobic nano-roughness can enhance the contact angle significantly [36]. In this paper, hydrophobicity can be accomplished by using the optimal concentration of resin on the created nano-roughness. (Figure 1-a) shows the drops and their evaluated contact angle.

Stain repellency

As shown in (Figure 1-b), in the control sample, stain drops were thoroughly absorbed during moving on the surface. However, in the samples treated with resin, the drops rapidly slipped without any trace. Investigation of the stain repellency of the samples was also pursued by another method. The samples were turned into a dye solution, and then color changes were studied. Figure 2-a shows the color difference between after and before staining for each sample.

TiO_2 samples, which had super-hydrophilic features, absorbed methylene blue more than control samples did, while sample which have XPs treatment varied less and resisted staining. According to (Figure 2-a), the color differences between the after and before staining for XPs-treated samples were very low. Therefore, the positive performance of XPs is confirmed. Figure 2-a shows ΔE values, the color difference between before and after staining calculated for each sample by the formula mentioned in the test method section.

Scanning electron microscopy

Scanning Electron Microscopy (SEM) was used to observe the particle dispersion on the treated samples. As (Figure 3), SEM observations of different magnifications, showed the nanoparticles had good dispersibility. It was also confirmed that an even coating had formed on the fiber surfaces.

Washing stability

As mentioned, there is no affinity between TiO_2 and fabrics. Nanoparticles are reluctant to react chemically with polymer surfaces. Therefore, they were absorbed physically and their stability was completely related to XPs resin to characterize the released TiO_2, proper diluted washing effluents were analyzed by ICP-OES. For

Table 3: UV-Vis based effluent analysis and calculated fastness.

Samples	Treatment Status	Washing cycle	Released TiO_2 (ppm)	Remained TiO_2 on fabrics (%)
FL	TiO_2	5	39180	21.64
	TiO_2+Ps	5	9776	80.45
	TiO_2	20	40619	18.76
	TiO_2+Ps	20	10552	78.90
TC	TiO_2	5	27132	45.74
	TiO_2+Ps	5	7500	85.00
	TiO_2	20	28268	43.46
	TiO_2+Ps	20	10700	78.60
LC	TiO_2	5	31544	36.91
	TiO_2+Ps	5	8544	82.91
	TiO_2	20	32452	35.10
	TiO_2+Ps	20	8803	82.39
SC	TiO_2	5	49150	1.70
	TiO_2+Ps	5	2426	95.15
	TiO_2	20	49714	0.57
	TiO_2+Ps	20	2499	95.00

this purpose, FL samples were chosen to examine. The final results have been reported in (Figure 2-b). The results demonstrated that XPs resin could save about 81% and 80% of nanoparticles after 5th and 20th washing cycles, respectively, while the TiO_2 remaining on the sample without XPs was about 26% and 22% after 5th and 20th washing cycles, respectively.

Because of such features as availability and low cost, UV-vis spectroscopy was used to evaluate the washing stability of the samples. To investigate the competency of such an alternative, all the data obtained by both methods were compared (Figure 2-b). The results showed a good consistency, thus it can be concluded that UV-Vis spectroscopy is a good alternative to follow the ICP-OES method in this case.

Table 3 reports the amount of the released titanium dioxide in all the effluent samples with "TiO_2" and "(XPs+TiO_2)" treatment as an index for measuring nanoparticle stability. The percentage of the remaining TiO_2 on each washed sample was also calculated to investigate the stabilizing efficiency of XPs treatment.

Based on the results, there was a significant loss in the amount of titanium dioxide on the sample without resin after 5th wash, as compared to the sample treated with TiO_2 and resin. Calculating the titanium dioxide remaining on the SC fabrics revealed that about 95% of nanoparticles still existed on the fabric treated with resin after 20 times of home laundering. As predicted, in the samples treated only with TiO_2, nanoparticles were easily separated from the surface. Here, the role of resin, as a good holder is clear because, even in the five primary effluents of samples treated with resin and titanium dioxide (Xps+TiO_2), the concentration is extremely lower than that of the sample without the resin.

Sunlight resistance

To evaluate the protection efficiency of nanomaterial against sunlight, the samples were exposed to sunlight for 100 and 160 hours and then the abrasion resistance was tested as an index for evaluating the resistance of fabrics against structural degradation under UV. The results (Figure 2-c) showed that increasing the time of exposure to sunlight weakened the fibers of the control samples (in the case of FL and CL which were tested), and their abrasion resistance decreased, while nano-treatment could control the harmful effects of sunlight. It seems that applying this feature in upholstery, especially curtains, is significantly constructive.

Air permeability

FL and CL fabric series were examined in terms of air permeability. The results were reported in (Table 4). The statistical analysis indicated that the nano-functionalization has no significant effect on air permeability.

Bending length

Bending length was evaluated as a criterion of stiffness and handle for FL and CL fabric series. The results were reported in (Table 4). Although the results showed TiO_2 treatment made surfaces stiffer, XPs had an opposite effect. However, according to the statistical analysis, the differences were not significant.

Crease recovery angle

An experiment was performed to evaluate FL and CL crease recovery angle. As reported in (Table 4), TiO_2 treatment significantly decreased the crease recovery angle; however, XPs significantly improved the anti-wrinkle properties of both kinds of fabrics as compared to both control samples and TiO_2 treated samples.

Surface friction

FL and CL fabric series were also examined for surface friction properties (Table 4). According to the results, TiO_2 treatment increased friction coefficient as compared to the control samples. XPs had compensated for this effect. According to the statistical analysis, the differences between all the samples were significant in

Table 4: Physical properties of lace fabrics (Data is reported in "Average (S.D.)" form).

Physical factor	FL samples			CL samples		
	C	TiO_2	W(XPs+TiO_2)	C	TiO_2	W(XPs+TiO_2)
Air permeability (ml.S^{-1}/5cm^{-2})	276 (10.6)	-	266 (9.7)	214 (10.1)	-	208.2 (4.45)
Bending length (cm)	1.93 (0.4)	1.96 (0.2)	1.8 (0.2)	3.18 (0.4)	3.3 (0.3)	2.8 (0.6)
Crease recovery angle (degree)	148.2 (2.4)	69.6 (11)	162.6 (1.7)	141 (1.6)	104.6 (1.8)	152.2 (2.6)
Static angle of surface drag (D_s) (degree)	40.3 (4.5)	47.9 (1.8)	36.5 (4.9)	39.8 (1.4)	47.9 (1.8)	44.3 (1.8)

the case of CL series fabrics; however, in the case of FL series, only the differences between TiO_2 and TiO_2+XPs were significant. However, the treatments had no significant effect as compared to the control FL sample.

Conclusions

In this study, different patterned and unpatterned light-weight and delicate fabrics and lacy fabrics were treated by titanium dioxide and polysiloxane resin (XPs), and their final properties were evaluated. By applying TiO_2 and XPs treatment, significant hydrophobicity and super hydrophobicity were achieved. The results revealed that the textiles constructions significantly affected on the results. In the ideal case, absorption time of water-drop was approximately 6 hours. Stain droplets slipped on the samples without any trace; and thus, the desirable stain repellency was achieved. The maximum obtained value of contact angle was about 150 degrees, which is another sign of good hydrophobicity of the treated sample.

Based on results, XPs treatment was able to effectively prevent separation of particles from the fabric surface during washing. After calculating the percentage of titanium dioxide remaining on the fabrics, it was found that, in the case of the best fastness, about 95% of the particles still remained after 20 times of domestic laundering. The optimal stability was directly related to silicone resin. Polysiloxane resin on TiO_2 compensated for the unpleasant variation created by titanium dioxide treatment in flexural stiffness, air permeability, as well as friction, and improved wrinkle recovery. Based on the results, the nanocomposite coating served as a protective layer against the destructive effect of sunlight. Consequently, the nano-finishing can be used as a novel effective treatment to enhance practical properties and/or add some desirable functions to the different patterned and unpatterned and lacy light-weight and delicate fabrics. However, it should be noted that the level of improvement depends on textiles constructions and it may be optimized to achieve the ideal properties.

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