

Photostability and Durability Properties of Photochromic Organosilica Coating on Fabric

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ABSTRACT

Photochromic fabrics were prepared by a dip-coating method using a silica sol-gel solution containing photochromic dyes. The coated fabric showed a rapid photochromic response. Three methods; incorporating a UV stabilizer in the coating layer, hydrophobic treatment of the porous surface, and covering the coating layer with an additional silica layer; were used to improve the photostability and durability. All three treatments improved the photostability without noticeably changing the photochromic response/fading speeds. Most of the treatments reduced the washing and abrasion durability. The extra coating layer increased the fabric rigidity.

INTRODUCTION

Photochromism is a photo-induced transformation between two optical absorption states [1-4]. Since the first report in 1867, this fascinating color-changing property has received great attention in science and industry [1]. It has been used in areas such as sunglasses and ophthalmic lenses, security printing, cosmetics, optical data storage, memories, optical switches, sensors, and displays [5-10]. Recently, there has been some interest in developing photochromic smart textiles [8, 9, 11-14]. Conventional methods to apply photochromic substance on fabrics include exhaustion dyeing and coating the dye microcapsules onto fabric surface. However, the first method fails to provide sufficient space for photochromic molecules to accomplish the structural transformation, and consequently the photochromic effect is insignificant. The second method often leads to a harsh fabric handle.

The sol-gel method has been recently used for producing photochromic hybrid materials [15]. The possibility of producing transparent, porous solids at a low processing temperature is one of

the great advantages of this technique. Organically modified silica (organosilica) can be processed through the sol-gel method to develop microporous matrices, which are excellent hosts for organic dopants. The inclusion of organic functional groups in hybrid silica helps exploit the superior thermal and mechanical properties of the inorganic constituents, as well as create a favorable environment for photochromic dye [16].

Previously, we reported on developing a hybrid organosilica coating with a photochromic dye embedded through a sol-gel method [17]. Although UV irradiation is essential in producing the photochromic effect, prolonged exposure to UV light leads to degradation of photochromic dyes, resulting in low photostability. To solve this problem, three strategies were developed. These include incorporating UV photo-stabilizers, modifying surface wettability with a low surface energy silane, and blockading the dye-encapsulating pores with an additional silica coating layer. The effect of the treatments on photochromic properties, photostability, durability against washing and abrasion, fabric rigidity, and wettability of the coatings were examined.

EXPERIMENTAL

Materials

A list of chemicals used in this work is given in *Table I*. An undyed double jersey knitted interlock fabric (245 grams per square meter) was used in this work¹. The fabric consisted of 19.5 μm Australian merino wool top, chlorine/Hercosett treated to machine washable standards. A visual assessment of the front and back side of the fabric indicated 12 and 10 stitches per cm in the wale and course directions, respectively.

¹ Obtained from AIM Sports Pty Ltd (Australia)

TABLE I. List of chemicals.

Type	Chemical name	Abbreviation
Photochromic dye	5-chloro-1,3-dihydro-1,3,3-trimethylspiro[2H-indole-2,3'-[3H]-naphth [2,1-b] [1,4] oxazine]	-
Silica precursor	octyltriethoxysilane	OTES
Silica precursor	phenyltriethoxysilane	PhTES
Silica precursor	3-Aminopropyl triethoxysilane	APS
Silica precursor	(3-Trimethoxysilyl) propylmethacrylate	MPS
Silica precursor	Tridecafluorooctyl triethoxysilane	FAS (fluoroalkylsilane) ²
Catalyst	tetracetoxy silane	TAS
UV quencher	2,2'-thiobis(4-tert-octylphenolato)-Nbutylamine nickel (II)	UVQ ³
UV absorber	2-hydroxy-4-methoxy-benzophenone	HMBP
UV absorber	2-(2Hbenzotriazol-2-yl)-4-(1,1,3,3-tetramethylbutyl) phenol	BTMBP ⁴
Free radical scavenger	poly(4-hydroxy-2,2,6,6-tetramethyl-1-piperidine ethanol-alt-1,4-butanedioic acid)	HALS ⁵ Hindered Amine Light Stabilizer)

² FAS was donated by Evonik Degussa. All the other chemicals were obtained from Sigma-Aldrich and used as received.

³Commercial name: UV-1084

⁴Commercial name: Tinuvin-329

⁵Commercial name: HALS-622

Silica Sol-Gel Synthesis and Coating

Silica sols were prepared by mixing TAS, alkyltriethoxysilane (RSi), and ethanol, and stirring until TAS crystals were dissolved. Water was subsequently added using a syringe pump at a 1 ml/h rate and the mixture was stirred for 24 h. The molar ratio of TAS: RSi: H₂O: ethanol was 1:32:100:320. TAS was selected as catalyst to slowly release acetic acid into the mixture, to avoid rapid hydrolysis. Alkyltriethoxysilanes OTES and PhTES, with a ratio of 1:2 (mol/mol) were used for making all the coating solutions. The chemical structures of OTES and PhTES are shown in *Figure 1*. 10 mL of photochromic dye-ethanol solution (Dye: RSi molar ratio of 1:100) was then mixed with 10 mL of synthesized sol-gel, and stirred at about 70 °C until a clear solution was obtained. A 20×20 cm² piece of wool fabric was laid flat in the solution, air-dried horizontally to reduce coating nonuniformity, and cured at 110 °C for 10 min. The coated fabric was left for 1-2 days before measurement to ensure complete stabilizing of the silica matrix while avoiding wool yellowing caused by excessive heat treatment.

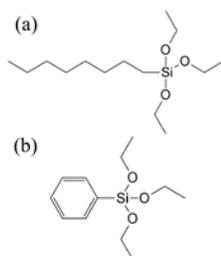


FIGURE 1. Chemical structures of (a) OTES, and (b) PhTES.

To add UV stabilizer to sols, a stabilizer was dissolved in ethanol. After ultrasonication for 5 minutes, dye-containing sol was added, and stirred at about 70 °C to produce clear solutions. The molar ratio of photochromic dye: UV stabilizer was 1:1. To adjust the wettability of the coating, FAS was added to the silane at the beginning of synthesis (before adding water). To seal off the dye-containing pores, a second layer (20 mL) of sol containing no dyes was applied after air drying the first for 24 hours, and was heat treated 24 hours later.

Photochromic Measurements

A SUB4000 optical fiber UV-VIS spectrometer and a DH2000-BAL UV-light source with a wavelength range of 230-2000 nm², and a personal computer were employed to measure the photochromic properties of the coatings. The color changes are expressed using the differential absorption between the state of presence and absence of UV light, $\Delta A = A_{\text{light}} - A_{\text{dark}}$, at the wavelength of maximum absorption (~627 nm).

The photochromic response and fading were recorded in real time as the ascent and descent of absorption when UV light was turned on and off, respectively. The response and fading rates were calculated from the time required for absorption to rise and subsequently fall to half of its maxima. These were expressed as response and fading half-life times, $t_{1/2}$ and $t'_{1/2}$, respectively, and were

² Both from Ocean Optics

measured using the absorption versus time graph obtained from the software³.

To measure photostability, samples were illuminated by strong UV light from an Hg metal halide lamp⁴ and their photochromic absorption was recorded after each exposure period (approximately 3.2 hours). The required time for the coated fabric to lose its entire photochromic effect (i.e. for absorption to no longer rise with UV stimulation) was considered the lifetime of the photochromic coating. For comparison, the photostability of a selection of coatings was also measured in a Suntest XLS+ from ATLAS. The samples were exposed to simulated sunlight for 5 hour periods at a 300 W/m² energy level.

Durability and Rigidity Measurements

The durability of the coatings was calculated from Eq. (1):

$$\text{Durability (\%)} = \Delta A_2 / \Delta A_1 \times 100 \quad (1)$$

where ΔA_1 and ΔA_2 represent the differential maximum absorption of the coating before and after the durability test, respectively. The Martindale machine was used for the durability to abrasion measurement. A 9 kPa load was applied and changes in absorption were recorded after 1000 abrasion cycles. The wash fastness measurement was carried out in accordance with Australian standard AS 2001.4.15.C. A M003B Shirley Stiffness Tester was employed for measuring the bending properties of the coated fabrics. The test was conducted in accordance with BS 3356 BS9073 part 7 and ASTM D1388. The Flexural Rigidity (G) and Bending Modulus (Q) were calculated from Eq. (2) and Eq. (3) in mg·cm and kg/cm², respectively:

$$G = 0.1MC^3 \quad (2)$$

where M is the mass per unit area (g/m²) and C is the bending length (cm) of the fabric.

$$Q = (12G \times 10^{-6}) / g_2^3 \quad (3)$$

³ SpectraSuite

⁴ Type OH N30/50E from Heraeus Noblelight GmbH; UV-A: 350-400 nm, relative irradiance around 90-120, and an intensity of approximately 80 mW/cm² at 365 nm

where g_2 is the thickness of the coated fabric (cm), measured under a 1 N weight. Water contact angle was measured on a KSV Model CAM101 Contact Angle Meter.

RESULTS AND DISCUSSION

The chemical structure and photochromic transformation of Photorome II, the spiro-oxazine (SPO) chromophore used in this study, are shown in Figure 2. This type of chromophore is colorless in its closed form, and undergoes a ring opening reaction when subject to UV radiation, transforming into a colored merocyanine in its open form. It subsequently returns back to its stable colorless state thermally or via visible light.

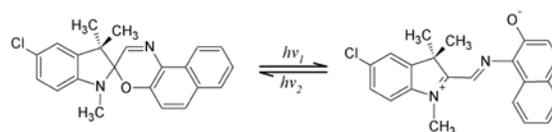


FIGURE 2. Structure and photochromic reaction of spirooxazine; left: closed colorless form; right: open colored form.

To select a suitable silica precursor for the synthesis of the matrix, preliminary trials were carried out with different combinations of OTES and PhTES silanes. A ratio of OTES:PhTES 1:2 (O:P 1:2) was selected for optimum coating performance, encompassing the superior photostability and response/fading speed of OTES in addition to the better durability of PhTES against abrasion and washing. The chemical structures of the silica precursors are shown in Figure 1. The Octyl functional group in OTES helps create more flexible pores, which leads to less steric hindrance for the encapsulated dye. This allows better optical performance by the photochromic dye. On the other hand, the phenyl ring in PhTES creates rigid pores which inhibit the structural transformation of the photochromic dye, leading to reduced photochromic performance.

The optical absorption of an OTES: PhTES 1:2 organosilica coating doped with SPO photochromic dye in the presence and absence of UV irradiation is displayed in Figure 3a. In the absence of UV light, the absorption spectrum was a flat line, whereas a photochromic peak was observed at a wavelength of around 627 nm in the presence of UV light.

The optical absorption spectrum of the coating recorded in real time at 627 nm is shown in *Figure 3b*. The time required for the absorption to ascend and subsequently descend to half of its maxima was calculated as shown in this graph, and denoted as $t_{1/2}$ and $t'_{1/2}$, or response and fading half-life times, respectively.

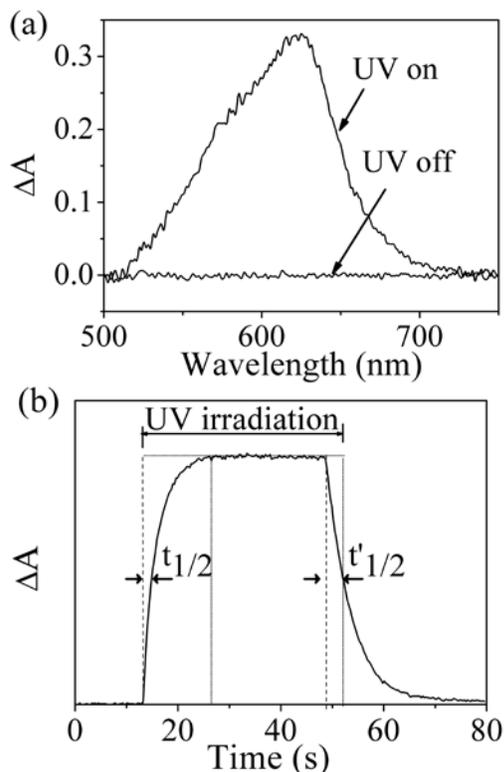


FIGURE 3. (a) VIS spectra, and (b) coloration-decoloration curve of an OTES:PhTES 1:2 photochromic coating.

The effects of three different treatments on photochromic and durability properties of the photochromic fabric were measured. The treatments included incorporating UV photostabilizers, modifying the coating's hydrophobicity by means of fluorinating the pores, and blocking the pores with an additional layer of silica coating.

In this study, three different types of light stabilizers were employed: a free radical quencher, UVQ, two UV absorbers, namely HMBP and BTMBP, and a free radical scavenger, HALS. The quencher, UVQ, captures the energy absorbed by the photochromic dye as a result of the UV irradiation, and dissipates it as heat, fluorescence, or phosphorescence emission. It has been reported that the degradation of photochromic dye is caused by photo-induced free

radicals [18]. UV absorbers, HMBP and BTMBP, block UV light, reducing the chance of generating free radicals [19, 20]. Hindered amine light stabilizers (e.g. HALS) are free radical scavengers [21]. Although they do not absorb UV light, they trap and deactivate the free radicals generated, preventing photochromic dyes from being degraded.

TABLE II. Coating conditions.

Method	Additives	Constituents
	Control	OTES:PhTES 1:2 sol + dye
Adding UV stabilizer	UVQ	O:P 1:2 sol + UVQ + dye
	HALS	O:P 1:2 sol + HALS + dye
	BTMBP	O:P 1:2 sol + BTMBP + dye
	HMBP	O:P 1:2 sol + HMBP + dye
Fluorinating	FAS	O:P 1:2 + FAS sol + dye
Double coating with extra silica	APS	1 st layer O:P 1:2 sol + dye
		2 nd layer APS sol only
	MPS	1 st layer O:P 1:2 sol + dye
		2 nd layer MPS sol only
Same silica (shown as 2xSS)	1 st layer	O:P 1:2 sol + dye
	2 nd layer	O:P 1:2 sol only

FAS enriches the pore surfaces with fluorine atoms leading to increased hydrophobicity, thus preventing moisture and degrading elements from coming into contact with the dye. Hou, *et al.* reported the positive effect of fluoro-alkyl silane on photostability, used independently or in conjunction with other additives [22-24]. In this work, FAS was combined with the sol precursors prior to hydrolysis, to further engage this additive with the matrix.

The addition of an extra layer of sol-gel silica was adopted to seal the dye-containing pores, and thus deterring degrading elements. (3-Aminopropyl) triethoxysilane (APS) and (3-Trimethoxysilyl) propyl methacrylate (MPS) were used as precursors for the additional protective sol-gel layer. The amino group also helps protect the chromophore from photobleaching [25].

The chemical structures of the UV stabilizers, fluorinated alkyl silane (FAS), and amino and methacrylate functionalized silica precursors are illustrated in *Figure 4*.

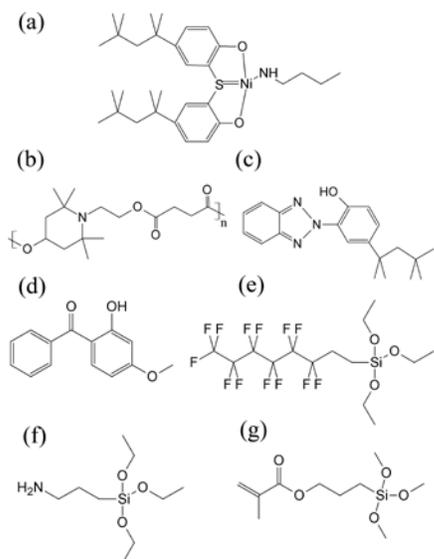


FIGURE 4. The chemical structures of UV stabilizers: (a) UVQ, (b) HALS, (c) BTMBP, (d) HMBP, as well as (e) FAS, (f) APS, and (g) MPS.

Photochromic Properties

The lifetime of the photochromic effect was measured by exposing the coated fabric to strong UV light for periods of 3.2 hours. The optical absorption was measured at the beginning of the experiment and after each exposure period. The test was continued until a photochromic peak was no longer distinguishable in the absorption spectrum. The response and fading times were calculated from the strips in *Figure 3b*. The photostability and response/fading data are plotted in *Figure 5*. All three types of treatments improved photostability, without significantly affecting coloration/decoloration times. The incorporation of UVQ was the most efficient treatment in terms of increasing photostability. Its performance was followed by that of double-coating with APS sol-gel. The enrichment of the pores with fluorine however was the least effective treatment, although it also slightly increased photostability.

It is difficult to judge the performance of the UV absorbers in comparison with the free radical scavenger, as BTMBP performed better than HALS in terms of improving photostability, while HMBP performed poorer.

Figure 5b shows the coloration and fading half-life times for the untreated (control) and treated samples. It was observed that the incorporation of UV stabilizers, FAS coating, and extra protective coating increased the response and fading times, while extra coating layer from APS sol-gel improved both these rates and MPS enhanced fading rates only. However, the impact of the treatments on coloration and de-coloration rates was overall insignificant.

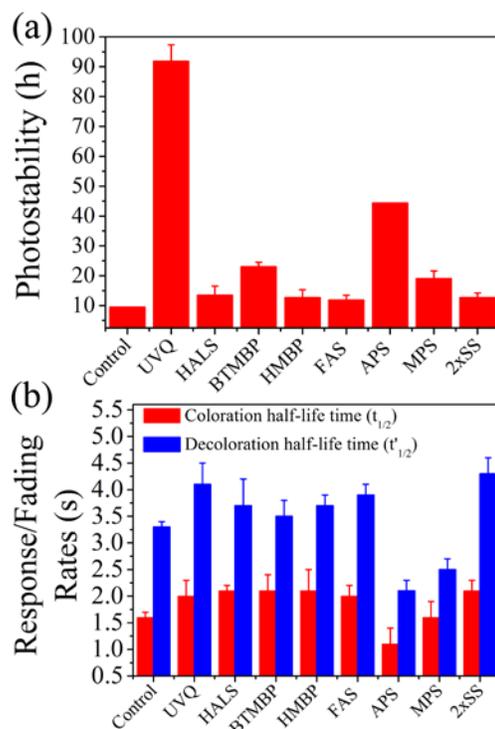


FIGURE 5. (a) Photostability and (b) photochromic response and fading half-life times of the coatings.

The UV absorbers and extra APS silica coating reduced the maximum absorption while HALS exhibited a smaller impact and UVQ displayed none (*Figure 6*). Since UV absorbers also absorbed UV light, the addition of the UV absorbers lowered the photochromic optical absorption.

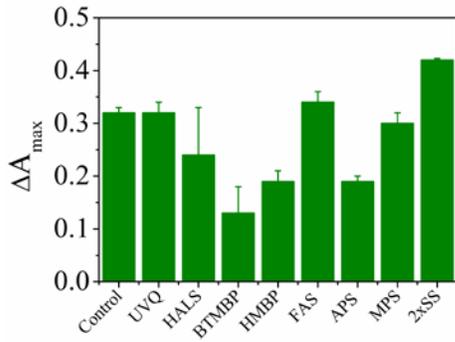


FIGURE 6. The differential absorption at a wavelength of 627 nm.

The changes of UV absorption with time for the two most photostable coatings, measured in a UV light box, are shown in *Figure 7a*. Absorption dropped with a rate of 0.01 and 0.03 for UVQ and APS, respectively, in the first cycle (3.2 hours). This was followed by a 0.04 and 0.01 drop in the following cycle for UVQ and APS, respectively. Generally, photo-degradation was most rapid in the first few cycles of irradiation, but slowed down after approximately ten hours of UV exposure. This can be attributed to a screening effect in which the photochromic dyes in the uppermost layers of the coating are the first to become degraded from UV irradiation, and their degraded products screen the inner layers of dye from UV exposure.

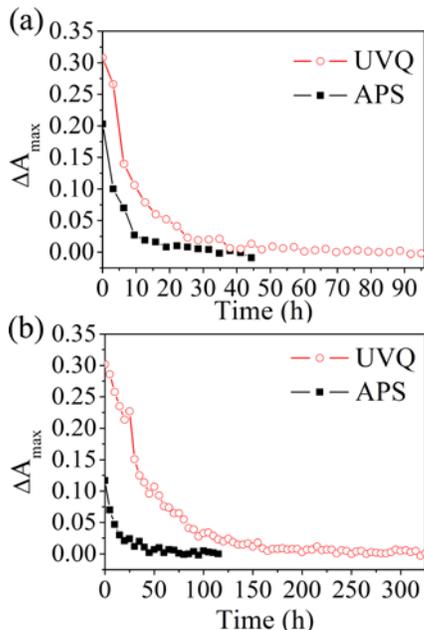


FIGURE 7. Change of photochromic absorption with UV irradiation time, irradiated by (a) Hg metal halide lamp, and (b) Suntest.

Figure 7b shows the UV absorption change for the UVQ and APS coatings during irradiation with Suntest. The slope of the fall was 0.003 for UVQ and 0.009 for APS in the first exposure period (5 hours). This was followed by a 0.006 and 0.005 decline for UVQ and APS, respectively, in the second cycle of irradiation. The rate of photochromic loss in the Suntest, which simulated solar radiation, was an order of magnitude slower, and the total photochromic lifetime longer compared to the UV light box, which used a Hg metal halide lamp as the light source. This is due to the lower intensity of UV irradiation in the Suntest compared to that in the Hg metal halide lamp. It is noteworthy that the Suntest provides a closer simulation of the real-life application of photochromic fabrics. However, the UV light box was selected as the main apparatus for photostability testing in this work because its higher UV intensity helped expedite the measurements.

The higher photostability which resulted from the incorporation of UVQ compared to that of APS can be attributed to the mechanism by which UVQ works and the method by which APS was applied to the fabric. As mentioned earlier, UVQ converts light into thermal energy or other forms of emission. The amino group in APS protects the dye from decomposition in the presence of light and oxygen. However, APS was applied to the fabric as an additional layer of silica.

The photostability of the control, UVQ, APS, and FAS samples, measured using the Suntest, were 45 ± 0 , 328.3 ± 2.9 , 113.3 ± 5.8 , and 36.7 ± 2.9 hours, respectively.

Washing and Abrasion Durability

The durability of the coated fabrics against abrasion was assessed by monitoring the changes in absorption after 1000 abrasion cycles (*Figure 8a*). Although some of the coatings reduced the abrasion durability, the overall level of abrasion fastness was satisfactory. The incorporation of UV absorbers BTMBP and HMBP merely reduced the durability by approximately 2%, while MPS and FAS caused a 9% and 11% fall, respectively. The double coating with same silica and APS treatments shifted durability by 11% and 13%, respectively. None of the treatments seemed to lead to a significantly large change in abrasion durability.

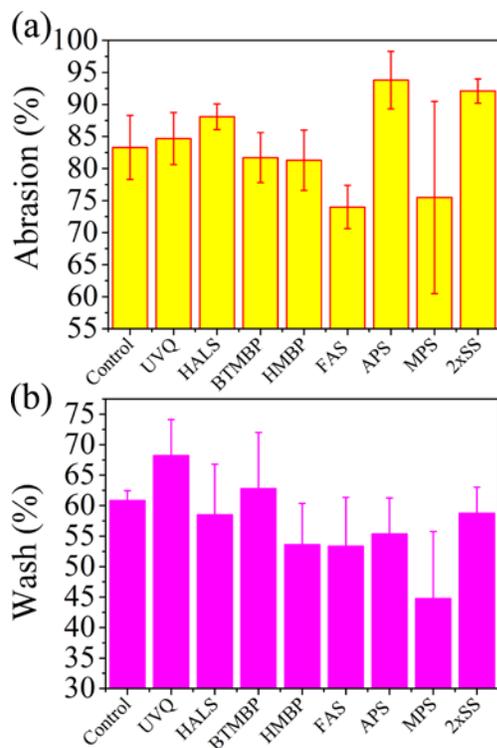


FIGURE 8. Durability to (a) abrasion and (b) washing.

The washing durability was measured by recording the changes in absorption after a wash cycle (Figure 8b). The incorporation of UVQ and BTMBP were found to improve the washing durability, while the other treatments showed reduced durability compared to the control. The wash durability of the MPS double coating was the lowest among all treatments.

Fabric Flexural Rigidity

The handle of the coated fabrics was assessed based on their flexural rigidity (mg·cm) and bending modulus (kg/cm²). The results indicated a notable increase in stiffness caused by APS sol-gel treatment (Figure 9). The other treatments did not cause a significant increase in flexural rigidity and bending modulus. All the treatments showed a higher flexural rigidity and bending modulus in the warp direction in comparison with the control, while this trend was not observed for all coatings in the weft direction.

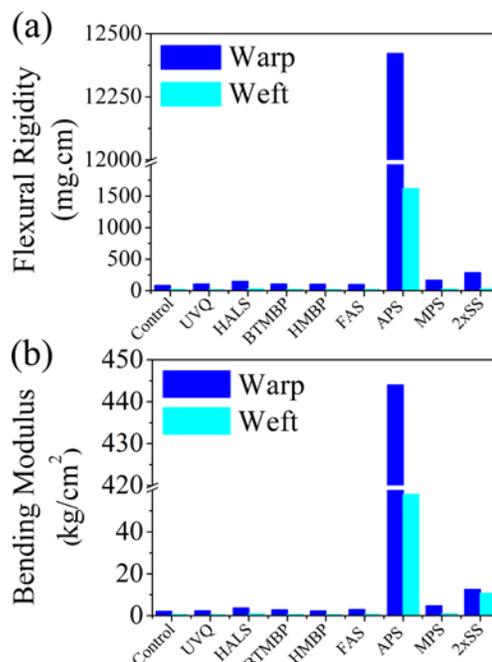


FIGURE 9. (a) Flexural rigidity and (b) bending modulus.

Wettability

The water contact angle of the coated fabrics was also measured. All the treatments caused a slight increase in the surface hydrophobicity, as evidenced by the slightly increased contact angle after the coating treatment. The largest increase in contact angle was caused by HALS (4.5%), followed by BTMBP (3.9%) and UVQ (3.9%). Double coating with the same silica caused the smallest change (0.3%), followed by double coating with APS (0.9%) and HMBP (1.1%). The more hydrophilic nature of the APS and HMBP coatings compared to the other treatments could be attributed to the amino group and hydroxyl group present in their chemical structures, respectively.

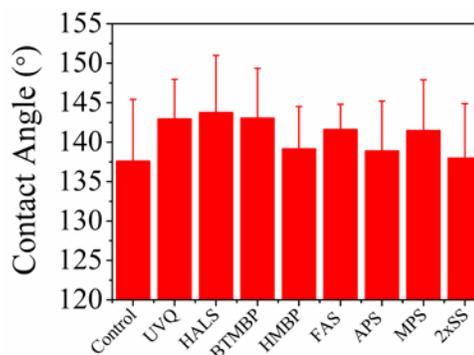


FIGURE 10. Water contact angle of the coated fabrics.

CONCLUSION

A photochromic silica coating on fabric was produced. The effects of incorporating UV stabilizers, modifying the surface wettability, and adding an extra silica layer on the coating properties were examined. All the treatments improved photostability to different extents, but did not affect the other photochromic properties significantly. The highest photostability was obtained by incorporation of a quencher UV stabilizer. The addition of an extra layer of APS sol-gel was the next most effective treatment in terms of photostability, although it lowered maximum absorption and increased coating rigidity. All coatings exhibited high abrasion durability and acceptable wash fastness.

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