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In-situ preparation of superhydrophobic and fire-retardant finish for polyester-cotton blend

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Abstract

Pyrovatex ACS is a popular commercial fire-retardant which is mainly applied for cotton fibers. However, Pyrovatex ACS has the concern of releasing poisonous formaldehyde, and it requires formaldehyde-based cross-linkers to improve the fire-retardant effect and durability on fabric. In addition, it can be applied efficiently only on 100% cotton fabrics as its efficiency decreases with textiles other than pure cotton. Herein, we introduce an easy to apply technique to produce a water-repellent and fire-retardant polyester-cotton blend fabric. A room temperature vulcanized silicone was applied as a formaldehyde free cross-linker to allow using Pyrovatex ACS at lower concentration compared to commercial cross-linking agents. The fire-retardant effect of the polyester-cotton blend fabric was improved as only 300 g/L of Pyrovatex ACS with silicon rubber silicone compared to the commercial amounts used at about 500 g/L. Moreover, silicon rubber silicone further enhanced the superhydrophobic effect on the polyester-cotton blend fabric surface. The surface morphology, stiffness and comfort properties of the treated polyester-cotton blend were explored. The washing performance of the treated polyester-cotton blend fabrics demonstrated good durability.

Keywords: Silicone; Pyrovatex ACS, Superhydrophobic, Flame-retardant, Cotton-polyester blend.

1. Introduction

Technical protective textile industry mainly focuses on non-aesthetic textile merchandises for certain functions to improve public safety, such as fire-retardant, medical and water repellent garments [1, 2]. Cotton-based textiles, such as pure cotton and polyester-cotton blend are very flammable, which may lead to severe damaging to the property, such as furnishings, house, vehicle, as well as human life [3]. Consequently, fire-retardant curing for cotton-based textiles is highly beneficial and can be accomplished [4, 5]. There are a range of commercial fire-retardant chemical treatments, particularly for textile clothing and soft furnishing, which are extremely flammable. Surface curing of textiles is an appropriate proficient method to impart fire-retardant feature to flammable substrates [6, 7]. The assembly of a fire-retardant thin efficient layer on a textile surface does not alter the mechanical characteristics of the treated high performance technical clothing [8]. Additionally, a fire-retardant layer on the fabric surface can be on purpose integrated with other functional materials to achieve multiple functions, such as superhydrophobicity, ultraviolet shielding and antimicrobial properties. Both hydrophobicity and fire-retardancy considerably raise the application range of technical garments in many fields, such as transport, craft and packaging [9]. A water-repellent surface, provoked by lotus phenomena has been a significant research field for latest decades [10].

Distinctive fire-retardant materials applied for cotton-based clothing are based on organophosphorus compounds which can afford a protective char layer on fabric surface in order to stop further burning [11, 12]. Both Pyrovatex CP and Pyrovatex ACS are the most commercially proficient and durable fire-retardant materials for cotton-based textiles. Though, they are lesser eco-friendly and they require trimethylol-melamine cross-linker to improve the fire-retardancy and durability as well as increasing the bonding of Pyrovatex to cellulosic polymer chains [13]. Furthermore, trimethylol-melamine, during the treatment process, produces high levels of formaldehyde as a by-product, which is highly poisonous because formaldehyde is carcinogenic, and may lead to skin and eye irritation [14]. Hence, the development of formaldehyde-free, inexpensive and durable cross-linking agent without affecting the physic-mechanical properties of treated fabric is very significant.

Room temperature vulcanized silicone is resistant to temperature, acids, alkaline materials, ageing, chemicals, as well as good mechanical stress and easy-to-operate. It has been applied in a range of applications, such as ink-jet printing and electronic materials [15, 16]. Recently, there has been a great concern to endorse an eco-friendly curing procedure toward fire-retardant for cotton-based textiles; particularly cotton-polyester blends. Herein, we developed an inexpensive and formaldehyde-free silicone rubber cross-linker to introduce water-repellent and fire-retardant cotton-polyester blend.

2. Experimental details

2.1. Chemicals and substrates

Mercerized plain weave cotton-polyester blend was obtained from **Misr Spinning** and **Weaving** El-Mahalla El-Kobra Company, Egypt. The fabric was 86×82 plain-woven cotton-polyester blend fabric (~110 gm/m^2 varieties); warp and weft yarns were 34^S and 32^S respectively; tensile strength warp 55 kgf; bend-length warp 3 cm and weft 2 cm; DCRA warp 55^o and weft 60^o; CRA warp 56^o and weft 54^o. Pyrovatex ACS was obtained from Huntsman chemicals. Decoseal silicone rubber (2540) was obtained from ADMICO Egypt. Toluene (anhydrous, 99.8%) was obtained from Sigma-Aldrich.

2.2. Treatment of cotton-polyester blend fabric

Silicone was dissolved in anhydrous toluene at 10% weight of silicone to volume of toluene. Pyrovatex ACS was added (100, 200, 300, 400 to 500 g/L) to the solution with vigorous stirring. The cotton-polyester blend fabrics were padded in each solution prepared above followed by air-drying. The cotton-polyester treated fabrics were then cured with ammonia gas in an oven at 75^{o}C, and finally rinsed with running water.

2.3. Characterization and testing

The flammability exam was performed using standard BS5438:1989. The bend-length (an average of five readings) was reported on a Shirley-Stiffness-Tester using ASTM-D-1388. The air permeability (an average of five readings) was recorded using ASTM-D-737 on Textest-FX-3300 at pressure of 100 Pa. Wascator-FOM-71-MP was employed to perform the laundry test using AATCC detergent (66 g), cycle number 5-MSA at 40°C for 1 hour. The whiteness of the treated samples was measured by exploring the color coordinates (L^*, a^*, b^*) on Ultra Scan Pro, Hunter Lab, United States. The surface of the treated cotton-polyester blend fabrics was analyzed by scanning electron microscope (Quanta FEG-250 with field emission gun, Czech Republic). The elemental content was explored on Energy dispersive X-ray spectroscopy (TEAM-EDX). The water contact angle was measured on OCA-15EC (Dataphysics GmbH, Germany) with 10   L water drop.

3. Results and discussion

3.1. Flame testing and durability

There are two key fire-retardant curing for industrial textile finishing, known as Proban and Pyrovatex. Proban technique is an easy to apply approach because it generates insoluble high molecular weight polymeric materials held physically within the cotton-polyester fibers and yarns within the fiber voids and gaps of the cotton-polyester blend yarn without forming actual chemical bond to the surface of cotton-polyester blend. Consequently, it affords, to some extent, harsh handle and as a result requires softener, such as TURPEX ACN. It also requires an oxidizing agent, such as hydrogen peroxide to decrease the quantity of formaldehyde and/or wetting agent, such as INVADINE PBN to improve the treatment process. Proban method is phosphorus-based substance previously treated with urea to afford phosphine oxide form while discharge toxic formaldehyde. Following to padding, the somewhat wet fabric was then cured with dry ammonia gas, and then oxidized by hydrogen peroxide. Uncoated cotton-polyester could not pass the fire test and was completely burnt to indicate poor fire-retardant effect. Pyrovatex ACS standard treatment at 500 g/L to cotton-polyester blend afforded good fire-retardant property as the fabric burn progress was stopped instantaneously by removing the flame source showing char length at 71mm. The cotton-polyester blend fabrics were treated using the Proban approach with Pyrovatex ACS at the concentrations (100, 200, 300, 400, and 500 g/L), while keeping silicone concentration constant at 10%. This introduced char lengths at 76, 65, 60, 52, and 48mm, respectively as shown in **Table 1**. The performance of the fire-retardant effect was improved by using silicone as a cross-linker in the treatment process. Treating the cotton-polyester blend with silicone in absence of Pyrovatex resulted in a complete burning of treated fabric during fire testing because silicone rubber itself cannot introduce any fire-retardant effect. Combining silicone with only 300 g/L of Pyrovatex in toluene led to improve the fire-retardant property of cotton-polyester blend. The char length was reduced from 71mm to 55mm compared to cotton-polyester fabrics previously exposed to standard Pyrovatex curing (only 300 g/L) and in absence of silicone cross-linker. Pyrovatex content of 300 g/L in combination with silicone cross-linker achieved better fire-retardant property as compared to standard Pyrovatex treatment at 5000 g/L without silicone. This is advantageous in reducing the quantity of the costly and less eco-friendly pyrovatex fire-retardant and the released formaldehyde during treatment process.

Table 1. Effect of silicone content on the fire-retardancy of cotton-polyester blend.

Pyrovatex content (g/L)	char length (mm)
100	76
200	65
300	60
400	52
500	48

In order to investigate the durability of the coated cotton-polyester blended fabrics with water-repellent and fire-retardant effects, we

The main purpose silicone cross-linker was to improve the washing durability of the fire-retardant coating finish at lower pyrovatex concentration compared to standard Pyrovatex commercial approach. The wash durability was described depending on the char length of the coated cotton-polyester blended samples at different numbers of wash cycles as shown in **Figure 1**. Compared to standard commercial treatment, it was found that the wash durability of the fire-retardant effect after ten wash cycles was enhanced when the cotton-polyester fabric was coated with a mixture of silicone and Pyrovatex. This can be assigned to the raised number of bonded sites between fire-retardant Pyrovatex and cotton-polyester blend fibers. The higher content of Pyrovatex in recipe enhanced the wash durability at 10 g/L concentration of silicone cross-linker. This suggested the effectiveness of silicone cross-linker to physically bond Pyrovatex to cotton-polyester fibers. As a result, the existence of silicone in the fire-retardant bath enabled the fire-retarding system to be more proficient and durable to wash.

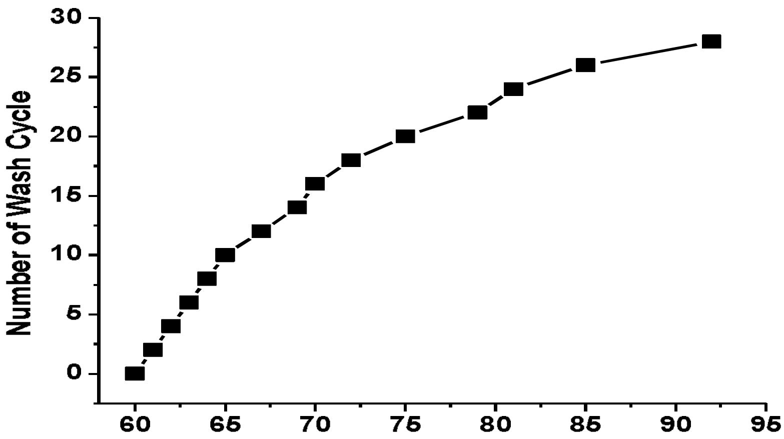

Figure 1. Effect of different wash cycles on treated fabrics; silicone content at 10% and pyrovatex concentration at 300 g/L.

3.2. Surface morphology of treated cotton-polyester blend fabric

The morphology of the cotton-polyester blend fabrics treated with silicone/pyrovatex was studied by scanning electron microscope and energy dispersive X-ray analysis as shown in **Figure 2**. It was found that the silicone/pyrovatex layer on cotton-polyester blend at different concentrations of the pyrovatex (100, 200, 300, 400 and 500%), while the concentration of silicone was constant at 10 g/L. No significant differences were monitored on the surface of the treated fibers upon increasing the pyrovatex concentration. However, the nanoparticles deposited on the fibrous surface of cotton-polyester blend proved the existence of the lotus effect to confirm the hydrophobicity. The composition in weight% of the treated cotton-polyester blend fabrics at two different positions for each treated sample, were displayed in **Table 2**. The energy-dispersive X-ray is shown in **Figure 3** proving the homogeneous distribution of silicone/pyrovatex on the cotton-polyester blend fibrous surface.

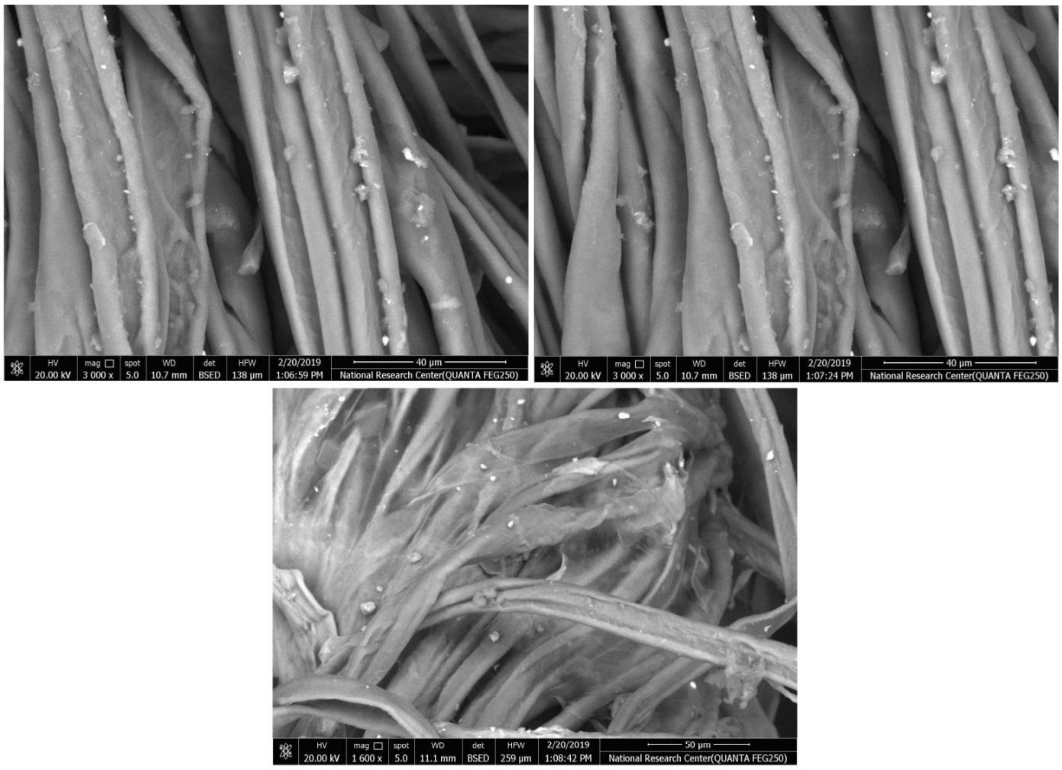

Figure 2. SEM images of cured cotton-polyester blend fabric (pyrovatex conc. at 300% and silicone conc. at 10%).

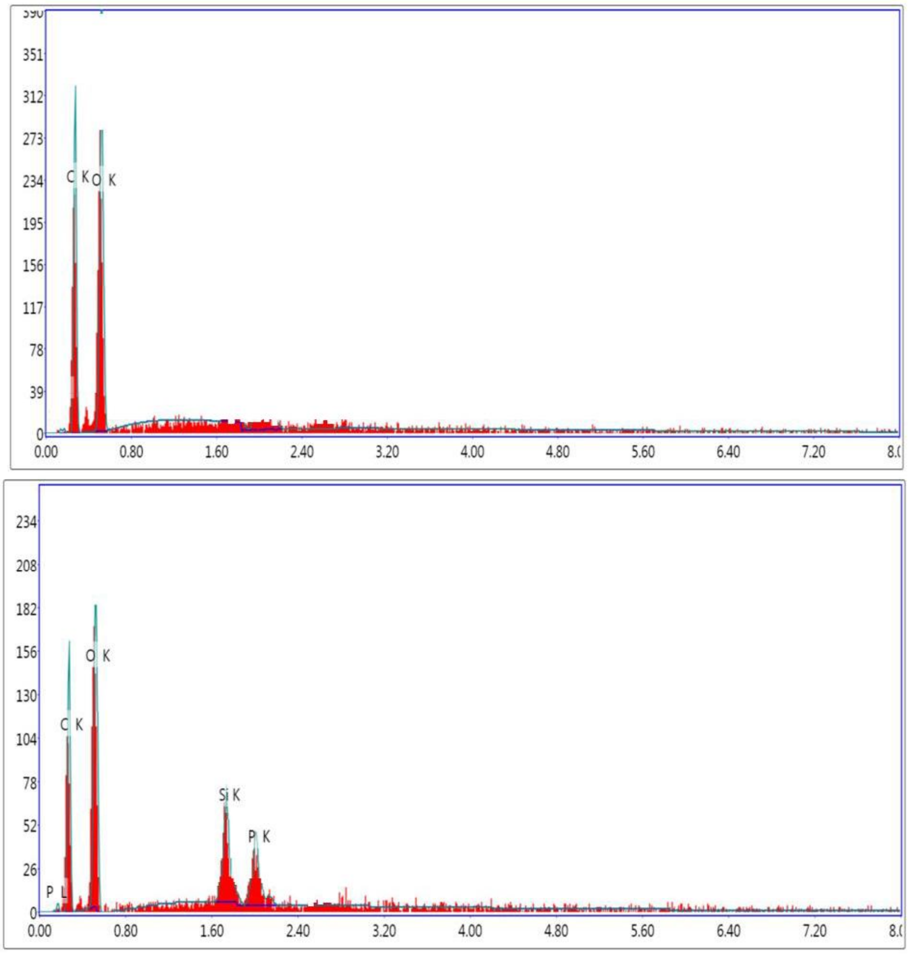

Figure 3. Energy-dispersive X-ray diagram of pristine (*top*) and cured (*bottom*) cotton-polyester blend fabric (pyrovatex conc. at 300% and silicone conc. at 10%).

Table 2. The elemental content in weight % at two different locations on the cured cotton-polyester blend fabric (silicone conc. at 10% and pyrovatex conc. at 100, 200, 300, 400 and 500%).

Pyrovatex Conc.		C	O	Si	P
		Wt. %	Wt. %	Wt. %	Wt. %
Blank	area 1	48.04	51.96	0	0
100%	area 1	44.14	41.99	11.04	2.83
	area 2	44.70	41.85	10.67	2.78
200%	area 1	43.17	41.75	11.63	3.45
	area 2	43.02	41.90	11.68	3.40
300%	area 1	43.59	41.39	11.07	3.95
	area 2	43.70	41.85	11.66	2.79
400%	area 1	43.14	41.99	11.57	3.30
	area 2	43.22	41.37	11.46	3.95
500%	area 1	42.08	41.98	11.62	4.32
	area 2	42.02	42.45	11.05	4.48

Surface roughness was fabricated by padding cotton-polyester blended fabric in silicone/pyrovatex solution. The water contact angle was found to be almost similar in the range of 144.7^o, 143.1^o, 142.9^o, 144.8^o, to 144.3^o for the treated cotton-polyester fabrics at different concentrations of pyrovatex in the range of 100, 200, 300, 400 and 500, respectively. There have been many studies that described the formation of water-repellent surface using expensive substances, which were then applied in time-consuming and complicated techniques [9, 10]. Therefore, it is a technological challenge to establish a water-repellent surface using the current simple and inexpensive approach that can be applied for potential large-scale manufacture of water-repellent substrates, such as water-repellent and fire-retardant tents.

3.3. Comfort and colorimetry

The major reason of using padding procedure was to introduce a smooth water-repellent and fire-retardant film with lower optimized

comfortability and physic-mechanical properties, such as breathability and flexibility. We assessed the flexibility of the treated cotton-polyester blended fabrics by determining the bend-length. The results of both bend-length and air permeability testing for coated and uncoated cotton-polyester blends are shown in **Table 3**. It was found that the treating process did not significantly influence the air permeability but a little effect was monitored as the fabrics rigidity slightly increased by increasing silicone content. Likewise, no considerable influence was detected in air permeability but a little increment in fabric rigidity was monitored by increasing the concentration of pyrovatex.

Table 3. Effect of padding on the blend stiffness and air permeability at different concentrations of pyrovatex (silicone conc. was 10%).

Pyrovatex Conc.	Bend-length (cm)		Air permeability ($cm^3\ cm^{-2}\ s^{-1}$)
	weft	wrap	
Blank	3.18	4.03	55.72
100 g/L	3.28	4.22	55.09
200 g/L	3.47	4.43	54.42
300 g/L	3.64	4.56	53.81
400 g/L	3.85	4.69	53.07
500 g/L	3.97	4.88	52.35

In order to evaluate the color alterations owing to silicone/pyrovatex curing, the three CIE LAB color coordinates (L^*, a^*, b^*) were recorded and summarized in Table **4**. As anticipated, no significant color alterations were detected upon curing of fabrics. This was confirmed by the very close values of the CIE LAB color coordinate values before and after curing of cotton-polyester blend fabric using silicone/pyrovatex formula.

Table 4. Color space values of coated fabrics at different concentrations of pyrovatex (silicone conc. was 10%).

Pyrovatex Conc.	L^*	a^*	b^*
Blank	95.56	0.02	2.46
100 g/L	94.02	- 0.18	2.17
200 g/L	94.37	-0.26	1.85
300 g/L	93.80	0.64	1.55
400 g/L	93.01	0.82	1.19
500 g/L	92.35	0.97	1.02

4. Conclusion

Cotton-polyester blend fabric was coated with an eco-friendly benign silicone rubber togeher with Pyrovatex ACS to impart fire-retardant and superhydrophobic effects. Cotton-polyester blend fabric was coated employing the pad-dry-cure method in an attempt to improve their fire-retardant performance employing silicone as a formaldehyde-free cross-linker at lower Pyrovatex ACS concentration at 300 g/L, compared to the commercial approach at 500 g/L. The results were confirmed by exploring the surface morphology of the coated cotton-polyester blended fabric on scanning electron microscope, energy-dispersive X-ray and elemental mapping. The comfort properties of treated cotton-polyester blended fibers were also evaluated to demonstrate an acceptable stiffness and air permeability. This approach was characterized by a simple bath assembly, stability to wash, simple to handle, no necessary for softener or wetting agent, and negligible loss in tensile strength and air permeability. Also, superhydrophobic finishing added an extra value to the treated cotton-polyester fabric.

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