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Synthesis of core-shell fluorine-silicon containing polyacrylate latexes for water and oil repellent finishing of cotton fabric

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A novel core-shell fluorine-silicon containing polyacrylate emulsion has been synthesized via semi-continuous seeded emulsion polymerization technique and then used to treat cotton fabric to achieve water and oil repellent properties in the latex coated fabrics. Structure of the resultant core-shell polyacrylate latexes has been characterized and the water and oil repellent properties of its coated fabrics are studied. Improved hydrophobicity and oleophobicity of the treated cotton fabrics are observed. The contact angle of a water droplet on the treated cotton fabrics is found up to 143.7°, and the rating of oil repellency and anti-soil properties are determined as 4.5 and 5 respectively. The changes in water and oil repellent properties of the coated fabrics after 20 cycles of standard washing are inconspicuous, while their physical and mechanical properties show a slight decrease.

Keywords: Core-shell copolymers, Cotton fabric, Fluorine-silicon containing polyacrylate, Oil repellent property, Water repellent property

1 Introduction

Hydrophobic and oleophobic surface finishes of different substrates, especially cellulose-based cotton fabric, are currently a flourishing research field, because cotton fabrics having water- and oil-repellency properties have wide applications in garments and industrial textiles for their desirable stain repellency and stain release as well as self-cleaning functions. For example, treated cotton fabrics were used to repel water but absorb oil in the process of oil-spill clean-up[1-5]. The extensively used approaches to obtain waterproof and oilproof fabric are based on the treatment of cotton fabric with functional substances of low surface energy, such as polysiloxanes, organosilanes, polytetrafluoroethylene (PTFE), fluorinated polyacrylates, and fluorosilicone[6-11], by offering a low-energy surface to make water and oil tend to form spherical droplets, thus refusing to wet the surface of textiles. Among which, fluorinated acrylic copolymer emulsions are the most favorable and widely used for the water and oil repellent finishing of fabric. However, these treatments have the disadvantages of poor durability, harsh handle, low breathability and adverse environmental impact. It is evident that these fluorocarbon chemicals lead to bioaccumulation and toxicity in the environment[12,13].

The common method adopted to enhance the washing durability and other easy care performances of the treated cotton fabrics is to include different cross-linking agents in coating recipe to strengthen the interactions between functional coating agents and cellulosic substrates. However, many of the cross-linking agents used contain toxic formaldehyde[14-16]. Water and oil repellency properties of treated textiles can also be facilitated by finishing with fluorinated polyacrylate latexes which were modified with silane coupling agents and silicone oil containing highly reactive groups in their molecular structures[17,18]. The washing durability of the treated fabrics is improved due to the formation of covalent bonds between fluorine-silicon containing polyacrylate and fibres, making the fabric feels relatively softer, smoother and more flexible.

Emulsion particles, composed of fluorine-containing acrylic shell and fluorine-free acrylic core formed via core-shell emulsion polymerization technique, were used in the process of water and oil repellent finishing; the shells of latex particles have the advantage of, preferentially, migrating towards the fabric surface in comparison with general fluorine emulsion particles. Cotton surface modifications

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based on fluorocarbon copolymers or core-shell fluorine-containing polyacrylate latexes have been reported[19-21]. However, the synthesis of core-shell fluorine-silicon polyacrylate latexes for water and oil finishing of cotton fabrics is relatively infrequent. Preparation of such latexes was given priority owing to their significant advantages in further promoting water and oil repellent properties (fluorine-silicon containing acrylic shell preferentially migrates towards the surface during film-forming process), strengthening washing durability of treated cotton fabric (covalent bond could be formed between siloxane groups of fluorine-silicon copolymer units and hydroxyl groups from cotton fabric), and reducing production cost (less fluorine-containing monomers were used by employing core-shell technique)[11].

In our investigation, a novel core-shell fluorine-silicon containing polyacrylate emulsion has been made via semi-continuous seeded emulsion polymerization by utilizing fluorinated acrylate monomer [1H, 1H,7H-Dodecafluoroheptyl methacrylate (G-04)], organofunctional alkoxysilane monomers [vinyl trimethoxy silane (KH-151)] and methacryloxy propyl trimethoxyl silane (KH-570). Methyl methacrylate (MMA), butyl acrylate (BA) and hydroxyethyl acrylate (HEA), were also used to treat cotton fabrics. Structure of the resultant core-shell latexes are characterized by employing Fourier Transform Infrared (FTIR) spectrometry, Transmission Electron Microscopy (TEM), Thermogravimetry Analysis (TGA) and Scanning Electron Microscopy (SEM). The water and oil repellent properties of coated fabrics were examined by using contact angle goniometer. The changes in water and oil repellent properties of the coated fabrics after 20 cycles of standard washing are also examined.

2 Materials and Methods

2.1 Materials

1H,1H,7H-Dodecafluoroheptyl methacrylate (G-04), vinyl trimethoxy silane (KH-151), methacryloxy propyl trimethoxyl silane (KH-570) and fluorocarbon surfactant (FS-200), supplied by Harbin XEOGIA Fluorosilicone Chemical Co. Ltd, China, were used as hydrophobic monomers and emulsifiers. Methyl methacrylate (MMA), butyl acrylate (BA) and hydroxyethyl acrylate (HEA), obtained from Tianjin Kemiou Chemical Reagent Co., China, were used as acrylate monomers. The chemical structures of these monomers are shown in Fig. 1. Fatty alcohol-polyoxyethylene ether (AEO-15), sodium dodecyl

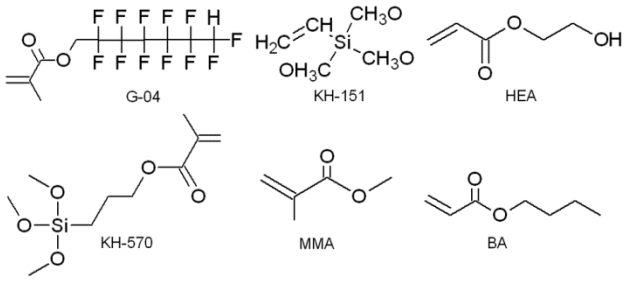

Fig. 1 — Chemical structure of monomers

benzene sulfonate (LAS) and potassium persulfate (KPS), provided by Wuxi Weier Chemical Co. Ltd, China, were used as emulsifiers and initiator. All the chemicals were used as received without further purification. Scoured and undyed 100% cotton fabric was coated using the conventional coating procedures.

2.2 Synthesis of Core-shell Fluorine-silicon containing Polyacrylate Latex

The hydrophobic functional monomers (G-04, KH-151 & KH-570), acrylic monomers (MMA & BA) and reactive monomer (HEA) were employed to prepare copolymer emulsions via semi-continuous seeded emulsion polymerization technique in two stages namely core phase and shell phase construction.

Emulsification of core and shell parts– Core phase (containing MMA 5.36 g, BA 13.39 g, KPS 0.15 g and deionized water 50.0 g) and shell phase (containing G-04 6.0 g, KH-151 1.0 g, KH-570 1.0 g, BA 3.0 g, KPS 0.15 g and deionized water 80.0g) were placed in two poly-tetrafluoroethylene beakers of 200 mL separately. The pre-emulsions of core phase and shell phase were homogenized intensively for 30 min using a FLUKO FM30-D high-shear dispersion homogenizer at a speed of 10,000 rpm respectively.

Preparation of core– 1/3 of core phase pre-emulsions and 1/3 of KPS solution (8% on the weight of core phase monomers) were added into a four-neck glass reactor equipped with a reflux condenser, a tap funnel (used for feeding the pre-emulsions) and a mechanical stirrer under N_2 purging. The mixed solution was stabilized in a constant temperature water bath at 78°C after the emulsion polymerization took place; another 2/3 of core phase pre-emulsions and the rest of the KPS aqueous solution were fed drop-wise in about 30 min, and the reaction temperature was raised to 80°C and maintained for 60 min.

Shell polymerization– The shell pre-emulsion and KPS solution (8% on the weight of shell phase monomers) were put into the tap funnel and dropped into the four-neck glass reactor in 90 min, and then

the emulsion polymerization was carried out at 80°C for another 60 min. At the same time, fluorine-free acrylate polymer emulsion was also prepared through copolymerization of MMA and BA, referring to the aforementioned preparing procedure of core phase.

It is worth noting that a common mixed emulsifier system was adopted in the above two procedures. The identical amount of AEO-15, LAS and FS-200 (at a weight ratio of AEO-15/LAS/FS-200=1:2:1) was used in both core phase and shell phase pre-emulsions.

2.3 Characterization of Copolymers

2.3.1 Determination of Solid Content of Copolymer Emulsion

The solid content (SC %) of copolymer latex was calculated gravimetrically using the following relationship [22]:

$$SC\% = (M / N) * 100$$

where M is the weight of dry, solid core-shell copolymer contained from N gram of copolymer emulsion.

2.3.2 Characterization of Copolymers by FTIR Spectroscopy

FTIR spectra of dry copolymer latex (free of moisture) were obtained in the range between 4000 cm^{-1} and 400 cm^{-1} on a Nicolet-5700 infrared spectrometer employing KBr pellet technique. The resolution ratio was 16 cm^{-1} and the number of scans was 100.

2.3.3 Characterization of Copolymers by SEM and TEM

Transmission electron microscopic (TEM) study of copolymer particles was carried out using JEOL JEM-2100 with an acceleration voltage of 80 kV. The surface morphology of the latex copolymer films formed on the surface of coated cotton fabric was examined in KYKY-2800B scanning electron microscope (SEM) (KYKY Technology Co., Ltd., China) operating at 20 KV.

2.3.4 Characterization of Copolymers by TGA

Thermal properties of copolymer film were studied using Thermogravimetric analysis (TGA) on a TGA/SDTA851^e thermogravimeter (METTLER TOLEDO, Switzerland) under nitrogen atmosphere in the 25° - 800°C temperature range at the heating rate of 10°C /min.

2.4 Treatment of Cotton Fabric with Copolymer Emulsion and its characterization

Eight per cent (8%) solution was prepared with resultant polymer in distilled water. The scoured white cotton fabric samples were immersed in the aforesaid solution for 20 min and padded through a laboratory padding mingle to have a wet pick-up of 75－85%. The wet cotton fabrics were dried at 105°C for 5 min and cured at 170°C for 3 min.

2.4.1 Evaluation of Hydrophobicity and Oleophobicity

Water contact angle shows the hydrophobicity of the surface of cotton fabric modified by copolymer emulsion. Sessile drop method was used to exhibit the static contact angle on a JC2000C3 contact angle goniometer (Shanghai zhongchen digital technic apparatus co., Ltd, China) at ambient temperature. The volume of the injected liquid was 5μL and the final contact angle of the treated samples was an average of five readings.

Hydrocarbon resistance test (AATCC-118) was used to study the oil repellency of the treated fabric. Droplets of eight standard fluids from a series of eight hydrocarbons with decreasing surface tensions were placed onto the surface of treated cotton fabrics. The rating of oil repellency on the scale of 0 - 8 was given based on the highest numbered fluid that did not wet the fabric.

2.4.2 Evaluation of Moisture Resistance and Soil Resistance

Moisture resistance of the finishes on the cotton fabric was performed on a LFY-214 fabric surface moisture resistance (wet) tester (Shandong Science Textile Research Institute, China), according to the test method specified in Chinese Standard (GB/T4745-1997). Soil resistance of the treated cotton fabric was examined based on AATCC Test Method 130-2000.

2.4.3 Evaluation of Durability of Finishing

The washing durability of the treated cotton fabric was also studied. The fabrics were washed in a standard washing machine based on the test method specified in Chinese Standard (GB/T 12490-2007) briefed below.

The treated cotton fabric samples were put into a soaping machine (concentration of soap flake was 2 g/L and liquor ratio was maintained at 1:30) to wash at 30°C for 5 min each time, and the fabrics could be washed for different number of times. Each of the washed fabrics was further subjected to rinsing in a beaker for three times with occasional stirring and hand squeezing. Then rinsed specimens were dried at 100°C for 5 min and transferred into a desiccator kept for 60 min before being evaluated.

2.5 Evaluation of Comfort Properties of Treated Fabric

2.5.1 Air Permeability

The air permeability test of modified cotton fabric was performed on an YG461E Digital Fabric

Permeability Instrument (Nantong Hongda Experiment Instruments Co., Ltd., China) according to the Chinese Standard GB/T 5453-1997. Nozzle 2 was selected for testing with a differential pressure of 100 Pa/mmH_2O, and the average of five readings was recorded.

2.5.2 Wrinkle Recovery and Whiteness

Wrinkle recovery and whiteness of the treated fabric were measured with a YG541L Digital Fabric Crease Elasticity Measure Instrument (Nantong Hongda Experiment Instruments Co., Ltd., China) and a Digital Fluorescent Whiteness Tester (DSBD-1, Wenzhou, China), according to AATCC Test Method 66-2003 and AATCC Test Method 110-2005 respectively.

2.5.3 Easy Clean Property

The oleophobicity of both untreated and treated cotton fabric were studied. The oil release test was performed according to the Chinese Standard GB/T 30159.1-2003. The images of the remaining oil stain on the fabrics after testing was taken for visual comparison.

3 Results and Discussion

3.1 Structure Characterization of Polymers

The structure of fluorine-silicon containing polyacrylate latex and fluorine-silicon free polymer emulsion was confirmed by FTIR spectra analysis (Fig. 2). For both the fluorine-silicon containing and fluorine-silicon free polymers, the characteristic absorption band at 2959 cm^{-1} attributed to C-H (CH_2), characteristic peaks at 1741 cm^{-1} attributed to stretching vibration of C=O, and distortion vibration of CH_2 at 1455 and 1383 cm^{-1} are observed. The absorption peaks at 1246 cm^{-1} and 1169 cm^{-1} have been assigned to asymmetrical and symmetrical stretching vibration of C-O, and the absorption at 973 cm^{-1} results from characteristic of BA. In the spectra for the fluorine-silicon-containing polymers, new absorption peaks at 686 and 1109 cm^{-1}, assigned to the stretching vibration of -CF and -CF_3 groups and the wagging vibration of -CF groups [23,24], and the characteristic peaks at 1026 and 803 cm^{-1} identified as stretching vibration of Si-O-C and Si-Me respectively, are found. In addition, the characteristic peak at 1641 cm^{-1} caused by the C=C bonds of the monomers disappears. This means that all the employed free monomers have participated in polymerization reaction.

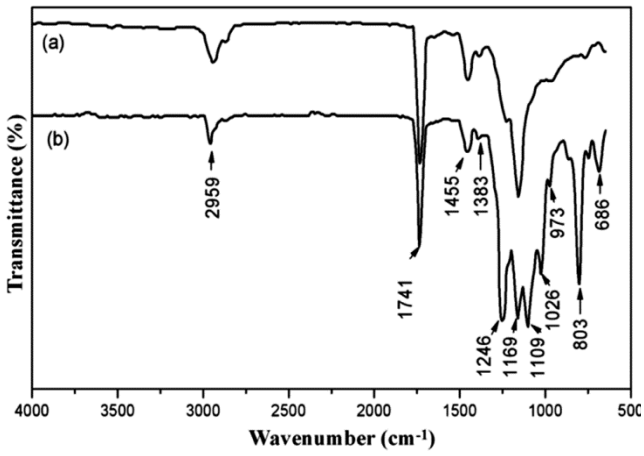

Fig. 2 — FTIR spectra of (a) fluorine-silicon free and (b) fluorine-silicon containing polymers

3.2 Morphology of the Resultants

3.2.1 SEM Analysis of Coated Fabric

SEM technique was employed to reveal the surface morphology of the cotton fabric untreated and treated with resultant fluorine-silicon-containing polymers. While untreated cotton fabric surface is found uneven as shown in Fig. 3(a), the surface of the fibres on the treated cotton fabric seems to be smooth and cylindrical; some local edges of untreated fibres become blunt, and a uniform resin film is deposited on the treated fiber surface [Fig. 3(b)].

3.2.2 TEM Analysis of Fluorine-silicon containing Polymers

The morphological structure of as-synthesized copolymer emulsion is studied in transmission

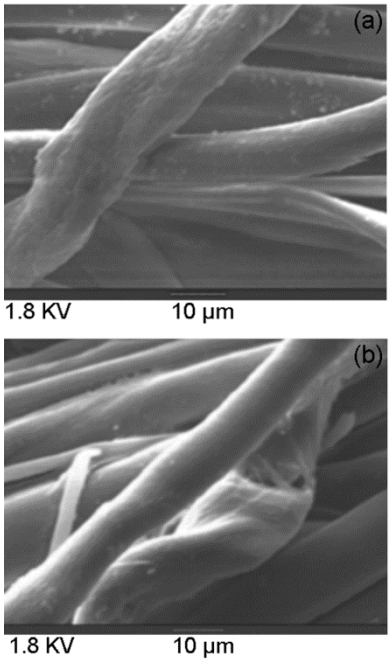

Fig. 3 — SEM micrographs of (a) untreated cotton and (b) cotton fabric treated with fluorine-silicon containing polymers

electron microscope (TEM). As shown in Fig. 4 (a), the latex particles show a relatively smooth spherical surface, and the average particle diameter is found about 100 nm. The core and shell structures of the latex particles could be observed clearly in Fig. 4 (b),

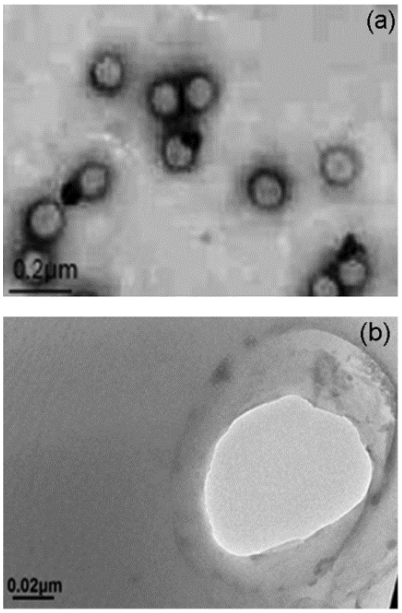

Fig. 4 — TEM micrographs of polymer emulsion (a), and the core-shell structure of particle (b)

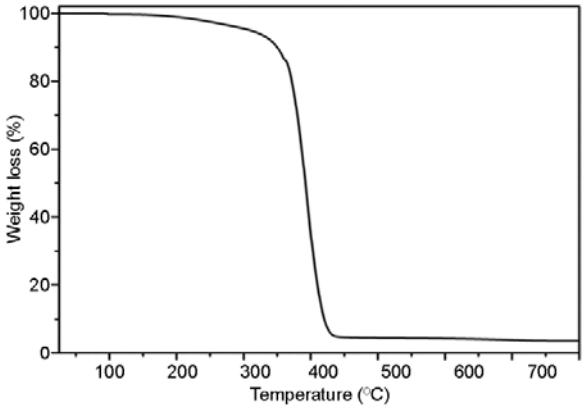

Fig. 5 — TGA curves of copolymer film

which may be attributed to the difference in electron penetrability of the core and shell[24,25].

3.3 TGA Analysis of Copolymer Film

Thermogravimetry was used to analyze the thermal behavior of fluorine-silicon containing copolymer film. As illustrated in Fig. 5, the initial and final decomposing temperatures of fluorine-silicon containing copolymer film are 347°C and 471°C respectively, and the maximum thermal decomposition rate is appeared at about 398°C. When heated to 800°C, organic parts are completely decomposed. The remaining residue is possibly from silicon and residual carbon.

3.4 Properties of Cotton Fabric Treated with Copolymer Emulsion

The untreated cotton fabric exhibits no water and oil repellency. The hydrophobicity, oleophobicity, moisture resistance and soil resistance of the treated cotton fabrics are evaluated.

3.4.1 Evaluation of Hydrophobicity and Moisture Resistance

Figure 6 shows the water contact angle of the cotton fabric treated with fluorine-silicon free acrylate polymer [Fig. 6(a)] as well as fluorine-silicon containing polymer [Fig. 6(b)]. Fabrics coated with fluorine-silicon-free finishing agent show a contact angle of 123.5°. The water droplet as shown in [Fig. 6(b)] has a larger contact angle of 143.7°. The larger the contact angle, the better is the hydrophobicity. The water contact angle results show that the surface hydrophobicity of cotton fabric treated with fluorine-silicon containing agent is clearly improved; this is attributed to the hydrophobic surface formed by fluorine-silicon compounds.

Surface moisture resistance of the treated cotton fabric is also investigated (Fig. 6). The surface of the cotton fabric with fluorine-silicon free acrylate polymer is wetted by large and connected droplets over a large area [Fig. 6(c)]. In contrast, the water droplet on the surface of the fabric treated with the

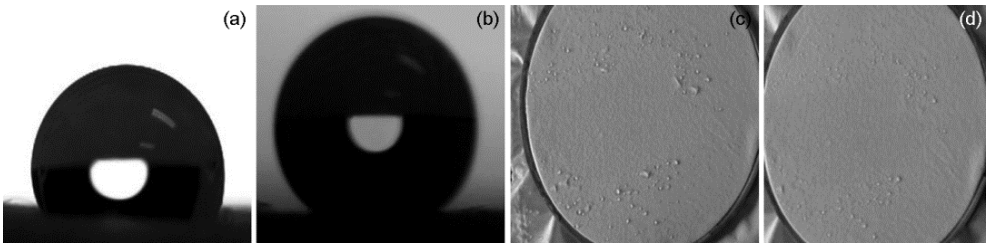

Fig. 6 — Static contact angle on cotton fabric surface treated with fluorine-silicon-free acrylate polymer (a), and fluorine-silicon-containing polymer (b); and surface moisture resistance of cotton fabric treated with fluorine-silicon free acrylate polymer (c), and fluorine-silicon containing polymer (d)

fluorine-silicon containing polymer is apparently smaller [Fig. 6(d)] as compared to the untreated one [Fig. 6(c)], which can be attributed to the superior hydrophobicity offered by fluorine-silicon containing polyacrylate latexes.

3.4.2 Evaluation of Oleophobicity and Anti-soil Properties

Oil-repellent rating on the scale of 0—8 has been provided according to drops of eight standard fluids, drop-wise added on the surface of cotton fabrics, which can be accurate to 0.5 according to the morphological characteristics of standard fluids. As demonstrated in Fig. 7 (a), standard fluids exhibit spherical and no surface wetting phenomena appear. Moreover, standard fluids are found partially wet [Fig. 7 (b)], yet still presented ellipsoidal. However, the surface of treated cotton fabric is entirely wetted by standard fluids as shown in Fig. 7 (c). Therefore, the rating of oil repellency is identified as 4.5.

The oil removal effects of the untreated and treated cotton fabrics are illustrated in Fig. 7. Apparent residual stains of peanut oils are observed on the surface of untreated cotton fabrics after being absorbed with tissues as showed in Fig. 7 (d). This indicates that the untreated fabric has no resistance to oil drops, and the resistance grade is confirmed as 1. Hardly any visible staining is shown on the surface of the treated fabric [Fig. 7 (e)] after being wiped out with tissues, its resistance grade is determined as 5.

3.4.3 Physical and Mechanical Properties

Physical and mechanical properties of treated and untreated cotton fabrics are shown in Table 1. It is observed that the breathability, whiteness, breaking strength as well as wrinkle recovery angle of the fluorine-silicon treated cotton fabrics are decreased to some extent. This means that the treated cotton fabric has become relatively stiffer and harsher in fabric handle. This effect may be because of the fact that a thin high molecular polymer **s**urface layer is formed on the finished cotton fabric in this process and then decreases the original performance of untreated cotton fabric.

The washing durability of the treated cotton fabric has been investigated using multiple washing cycles as seen in Table 2. Seven samples of the treated cotton fabric are loaded in a soaping machine at 40 °C and washed for 30 min in 150 mL water as well as detergent (2 g/L). Finally the fabric was washed for 5, 10, 15, 20, 25 and 30 times respectively. Table 2 shows that the durability of finish is slightly decreased with the increase of the washing cycles upto 20, but the durability declines obviously after 20 times of washing. It can be concluded that the fluorine-silicon agent molecules are probably firmly covalently bonded to the surface and the inner structure of treated cotton fabric, that is the reaction occurs between siloxane groups of fluorine-silicon copolymer units and hydroxyl groups from cotton fabric. The slight decrease in the durability during the initial washing might be attributed to the slight removal of a very small amount of copolymer molecules attached to the surface of cotton fabric due to abrasion effect between fabric surfaces. The sharp decrease of washing durability after 20 washing

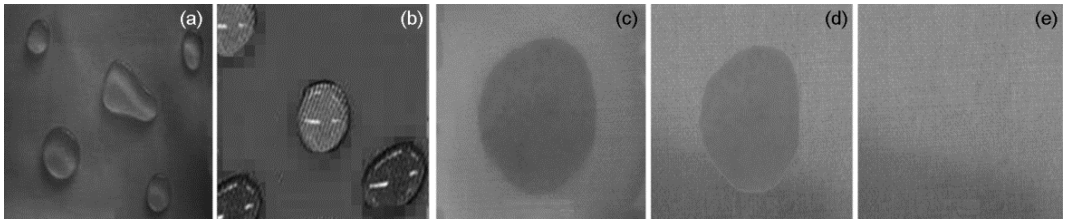

Fig. 7 — Oleophobicity of treated cotton fabric finished by n-**hexadecane** (a), n-**tetradecane** (b), and n-**dodecane** (c) fluids; Peanut oil stains on the untreated (d) and treated (e) cotton fabric surface

Table 1 — Physical and mechanical properties of pre-and post-treated fabric

Sample	Breathability, mm/s	Whiteness, %	Breaking strength, N	WRA (T+W), %			
				Immediate elastic recovery		Delayed elastic recovery	
				Warp	Weft	Warp	Weft
Untreated	107.8	73.2	468.6	94.97	67.21	107.86	81.63
Treated	106.4	71.4	459.7	88.98	65.82	93.78	78.53

WRA is wrinkle recovery angle.

Table 2 — Durability of finish on cotton fabric after finishing

Wash cycles	Water contact angle, deg	Hydrostatic pressure, kPa	Moisture resistance rating	Oil-repellent rating
0	143.7	1.85	5	4.5
5	142.8	1.83	5	4.5
10	138.9	1.78	5	4
15	134.2	1.75	4	3
20	132.7	1.73	4	3
25	130.1	1.68	3	2
30	121.6	1.36	3	1.5

cycles might be ascribed to the partial removal and shedding of uneven fluorine-silicon copolymer coating layer, indicating that the reduced mechanical strength caused by repeatedly washing could be the main factors affecting the washing durability.

4 Conclusion

A novel fluorosilicone-acrylate copolymer latex with core-shell structure has been successfully synthesized and characterized; structure and performance of the cotton fabrics treated with it are also examined. The emulsion particles has uniform spherical core-shell structure with an average diameter of about 100 nm as shown in TEM micrographs, and a uniform resin film is formed on the treated cotton fabric surface according to SEM. Hydrophobicity and oleophobicity of the cotton fabric treated with the functional latex perform better in comparison with that of the fabric treated with fluorine-silicon free polymer emulsion. The water droplet on the treated cotton fabric shows a contact angle of 143.7°, and the rating of the oil repellency and anti-soil properties is determined as 4.5 and 5 respectively. Washing durability of the treated fabrics decreases inconspicuously within 20 washing cycles.

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