Provisional chapter

Hydrophobic Surface Modification of Silk Fabric Using Plasma-Polymerized HMDSO

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Additional information is available at the end of the chapter

Abstract

In this work, we study the wetting properties of silk fabrics by deposition of plasma-polymerized (PP) hexamethyldisiloxane (HMDSO) using low-pressure plasma-enhanced chemical vapor deposition (PECVD). Recently hydrophobic properties are under active research in textile industry. The effect of exposure time and power on the HMDSO-coated silk fabrics has been investigated. Water contact angle of PP-HMDSO-coated silk fabric surface has been measured as the function of power and coating time. Fabric surface has shown enhancement in hydrophobicity after coating. Attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR) reveals the surface chemistry, and scanning electron microscopy (SEM) shows the surface morphology of the untreated and HMDSO-coated fabrics, respectively. In the case of untreated fabric, water droplet absorbs swiftly, whereas, in the case of HMDSO-coated fabric, water droplet remains on the fabric surface with a maximum contact angle of 140°. The HMDSO-deposited silk surface is found to be durable after detergent washing. Common stains like ink, tea, milk, turmeric, and orange juice are tested on the surface of both fabrics. In HMDSO-coated fabrics, all the stains are bedded like ball droplet. The fabric is tilted to 45° angle; stain droplets easily roll off from the fabric.

Keywords: plasma polymerization, HMDSO, silk fabrics, hydrophobic, PECVD

1. Introduction

Silk is a natural protein fiber from silk cocoon. It is highly praised as the queen of textiles because of its properties such as softness, glossy appearance, wearer comfort, warmth, and biodegradability. Silk fibers have large numbers of polar groups such as -OH, -COOH, and -NH_2 which are the backbone and side chains of polypeptide molecules. These hydrophilic structures provide a great atmosphere for growth of bacteria and fungi [1]. Silk fibers are susceptible to environmental circumstances, such as sunlight, staining from dirt, and debris. It is essential to extend silk fabrics with water-repellent functional properties having great potential in stain-free textile products. It prevents from accidental staining or water damage. The hydrophobicity of the fabric surface depends on its chemical functional groups, surface energy, and physical geometry [2]. There are some wet chemical methods available to change the surface properties of the fabrics, based on solvent-borne treatment with alkyl or partially fluorinated alkyl components [3, 4]. Several studies showed that properties of fabrics could be altered through surface modification. Alternating the surface properties of natural silk fibers by deposition of fluorinated polymers on the surface of the fabrics, it is becoming hydrophobic in nature [5]. Iriyama and Yasuda et al. reported that plasma treatment of CF_4 and C_2F_6 did not give good durability on the surface of polymer [6–8]. The tensile strength and hydrophobicity of Muga silk fiber have been reported by using RF argon (Ar) plasma treatment [9]. Li and Jinjin increased the contact angle of silk fabric up to 120° by C_3F_6 plasma treatment [10, 11]. Silk fabrics treated with SF_6 plasma showed that F replaced H and fluorination improved the hydrophobic property of the samples [7, 12–14]. Teli et al. improved the hydrophobic property of silk and cotton fabric using atmospheric pressure plasma in the presence of helium-fluorocarbon gasses, He/1,3-butadiene, and He/dodecyl acrylate [15–17]. The hydrophobicity has been achieved by plasma sputtering of polytetrafluoroethylene (PTFE) [18]. Fluorine-based polymers have hydrocarbons that break up into toxic compounds of perfluorooctane sulfonic acid (PFOS) and perfluorooctanoic acid (PFOA). The US Environmental Protection Agency (EPA) banned the long-chain fluorocarbon materials (PFOS/PFOA) due to their toxicity to human and environment [19–23]. However, the use of fluorocarbon and partially fluorinated alkyl compounds is undesirable due to the potential risks of the degradation by-products to human health and the environment, exceptionally high greenhouse effect compared to CO_2 [24, 25]. Many researchers' industrialized nanocoatings, like ZnO_2, Cu, TiO_2, DLC, etc., on the fabrics to increase the hydrophobic properties are still under active research [26–32].

Plasma-enhanced chemical vapor deposition (PECVD) of silicon compounds is an eco-friendly process and has been used to deposit ultrathin layer on the fabrics at a lower temperature without causing any thermal damages [33]. PECVD employs the conversion of monomer into reactive radicals, ions, and neutral molecules. Plasma polymerization creates a polymer film of organic compounds that do not polymerize under normal chemical polymerization process because it involves electron impact dissociation and ionization for chemical reactions [34]. Moreover, to reduce waste, pollution, water, energy, and time, plasma polymerization technology is employed, and it is a dry clean process which does not affect the environment. Silicon containing precursors like tetramethylsilane (TMS), tetraethoxysilane (TEOS), and hexamethyldisiloxane (HMDSO) is used for surface modification in textile industries. Among these, HMDSO precursor is nontoxic and nonexplosive and has a high vapor pressure at room temperature than TEOS [35–38]. Due to the presence of methyl groups; Si, H, and C atoms; and oxygen bond on the HMDSO, it changes the surface of the fabric into hydrophobic [35–39]. PP-HMDSO along with inert gasses in various natural as well as synthetic and blended textile substrates such as cotton, polyester wool, polypropylene, etc. has been studied by various researchers. Riccardi et al. observed that HMDSO-air was deposited by using dielectric barrier discharge plasma on silk surface by atmospheric pressure plasma to obtain a water-repellent silk fiber [40]. Hocker performed a deposition of HMDSO on oxygen-treated cotton fabric and achieved a maximum contact angle of 130° [41]. Ji et al. obtained the hydrophobic properties of polyester fiber by making use of an in-line atmospheric RF glow discharge plasma in a mixture of Ar and HMDSO [42, 43]. Palaskar et al. reported that HMDSO was coated with the mixture of helium and argon carrier gas for generating dielectric barrier discharge plasma on the polyester-/cotton-blended fabrics and improved the wetting properties of the fabrics [44]. Kale and Palaskar examined that deposition of TESO and HMDSO precursors by using PECVD was carried out on nylon 66 fabrics and HMDSO deposition rendered more hydrophobicity than TEOS [33]. Plasma polymer thin layers were deposited from pure HMDSO on polyimide substrate for water-repellent property enhancement and charge storage stability [45]. Shahidi et al. observed that HMDSO/N_2 plasma polymerization of wool fabrics improves anti-felting properties and dyeing behavior [39]. Plasma polymerization of organosilicon compounds is used in textile materials to increase functional properties of the materials. A lower surface energy indicates higher contact angle and greater hydrophobicity. A deposition of pure HMDSO by PECVD at low pressure on the silk fabric has not been reported yet.

In this study, the plasma polymerization of HMDSO on silk fabric has been performed by using the PECVD method at low pressure without causing thermal damage to the fabrics. PECVD coating technology has many advantages over conventional wet chemical methods. It has been used in a variety of deposition applications at a lower temperature. PP-HMDSO coating gives the possibility to obtain durable water-repellent surface due to retention of methyl groups. This coating makes the silk fabric surface water resistant, preventing it from accidental staining or water damage.

2. Materials and methods

2.1. Materials

A pure degummed silk fabric is purchased from a silk center with a warp count of 38 per/cm and a weft count of 38 per/cm 100% pure hexamethyldisiloxane (HMDSO), and the molecular formula is $C_6H_{18}OSi_2$. It is a colorless and highly volatile liquid, and the chemical structure is shown in **Figure 1**. It is obtained from Sigma-Aldrich.

2.2. Experimental

Plasma polymerization of HMDSO coating has been deposited onto silk fabric, and the experiment is carried out in a capacitively coupled plasma-enhanced chemical vapor deposition technique (PECVD) using pure HMDSO as a liquid precursor. PP-HMDSO depends on system pressure, discharge power, and coating time. The plasma reactor composed of stainless steel process chamber (24 cm height and 60 cm diameter) powered by a radio frequency generator (13.56 MHz) at room temperature. The chamber is evacuated to a base pressure of 1×10^{-5} mbar using rotary and diffusion pump. Plasma reactor consists of a pair of parallel symmetrical electrodes (35 cm diameter) separated by a distance of 3.5 cm. The schematic experimental setup is shown in **Figure 2**. The upper electrode has multipoint gas feeding

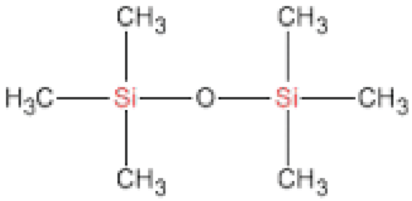

Figure 1. Chemical structure of hexamethyldisiloxane.

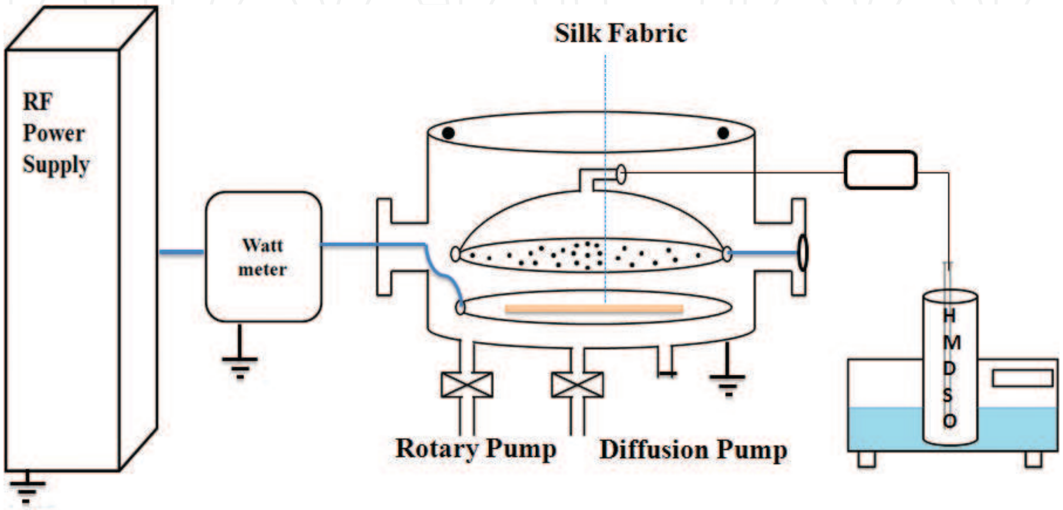

Figure 2. Experimental setup.

Sample code	Operating power (W)	Exposure time (min)	Pressure (mbar)
a	—	—	—
b	100	7	1.5×10^{-1}
c	100	15	1.5×10^{-1}
d	150	7	1.5×10^{-1}

Table 1. Operating parameters of PP-HMDSO.

shower head which is electrically grounded along with the chamber wall. The HMDSO is heated to 60°C using a constant temperature water bath. The working pressure is maintained at 0.15 mbar, and vaporized HMDSO liquid precursor is injected into the reactor using a needle valve without any carrier gas. The samples are placed on the surface of the lower electrode. The deposition is carried out for 7 min at 100 W and 150 W power and for 15 min at only 100 W RF power. The detailed experimental parameters of PP-HMDSO are reported in **Table 1**.

2.3. Instrumentations

FTIR spectra of the untreated and HMDSO-coated silk fabric are recorded by ATR-FTIR (Nicolet 6700, Thermo Scientific, USA). Morphologies of silk fabric, HMDSO-deposited fabrics, are examined with a scanning electron microscope (LEO s440i) at 10 kV and a magnification of 500×, 5000×, and 10,000×. Prior to SEM examination, the samples are pre-coated with gold sputtering to prevent charging of the samples by the electron beam. The static water contact angles on the untreated silk fabric and HMDSO-coated fabrics are measured using video contact angle optima (AST Products, Inc.) goniometer. A 5 μL drop of deionized water is placed on the substrate. The values of the static contact angles reported are the average of three measured values. Wet-out time is measured according to AATCC Test 79-1995. A 0.1 ml distilled water droplet is allowed to fall from a height of 5 cm onto the surface, and the time required for the droplet to be fully absorbed by the fabric is taken as the wet-out time. To study the durability of the HMDSO-coated fabric, washing test has been conducted. Based on the home laundering procedure, the HMDSO-coated fabrics are soaked in 60 ml of a 5.0% aqueous home laundering Surf Excel detergent (sodium carbonate, sodium aluminosilicate, alcohol ethoxylate, and sodium perborate monohydrate). The fabric is soaked in the detergent solution for 30 min at room temperature and rinsed with distilled water for several times and dried at room temperature overnight. Water repellency spray tester of AATCC Test method 22-2005 is modified to test the self-cleaning ability. In order to study the aging effect of HMDSO-coated silk fabrics, it has been left at room temperature for 100 days. Photographic images of different stains like ink, tea, milk, turmeric, and orange juice droplets on silk fabrics with and without HMDSO coating were measured by a digital camera after 100 days.

2.4. Results and discussion

2.4.1. ATR-FTIR

Attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR) is used to examine functional groups of the untreated and HMDSO-coated silk fabrics and is shown in **Figure 3**. From **Figure 3a**, it is seen that the ATR-FTIR spectra of untreated silk fabric, the IR spectral region from 1700 to 1500 cm^{-1}, are due to peptide backbone, and the characteristic bands at 1621 cm^{-1} (amide I) are due to β-sheet confirmation of C═O stretching vibrations,

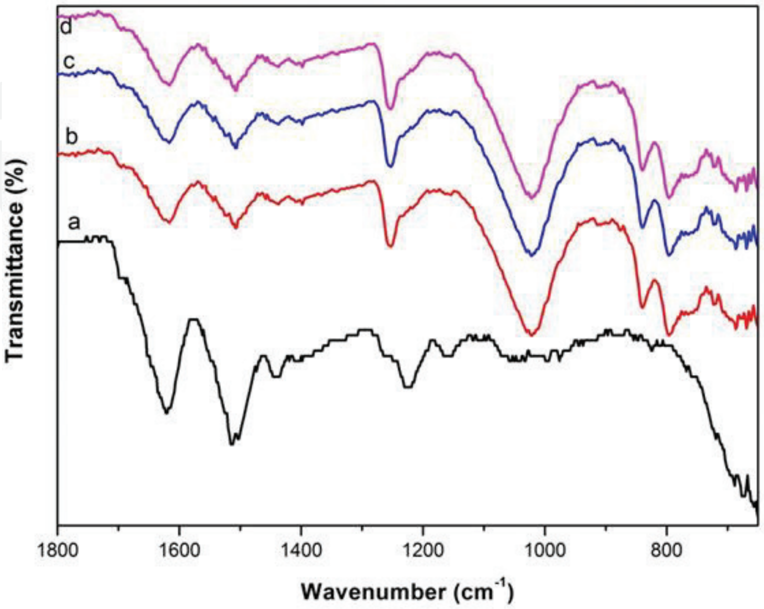

Figure 3. ATR-FTIR of (a) uncoated, (b) 100 W_7 min, (c) 100 W_15 min, (d) 150 W_7 min.

while band at 1514 cm^{-1} (amide II) is assigned due to the random coil conformation of fibroin molecules. The peak that appeared at 1226 cm^{-1} (amide III) is due to C—N stretching vibrations of the secondary structure of β-sheet [46]. **Figure 3b–d** shows the HMDSO-coated silk fabric; it is very clear that additional peak at 1218 cm^{-1} is due to symmetric bending of Si-CH$_3$. A band at 1041 cm^{-1} is exhibiting due to Si—O—Si stretching vibrations, whereas the peaks appeared at 970 and 763 cm^{-1} are due to Si-C rocking vibrations in the Si—CH$_3$ groups. The change in the peak intensities of the region from 1400 to 1800 cm^{-1} is attributed to the deposition of HMDSO [44].

2.4.2. SEM

SEM images of untreated and HMDSO-coated silk fibers are shown in **Figure 4**. **Figure 4a1–a3** shows SEM images of the untreated silk fabric at a magnification of 500×, 5000×, and 10,000×, respectively. It is clearly observed that the untreated silk fiber has a tranquil surface and is free from harshness. Since the surface of the silk is smooth, it has high polar groups which absorb the polar group immediately. **Figure 4b1–b3** and **c1–c3**shows the HMDSO deposition at operating power 100 W and exposure time of 7 and 15 min with a magnification of 500×, 5000×, and 10,000×, respectively. It is seen in the HMDSO-coated silk fabric SEM images (**Figure 4b1** that the presence of some flakes like structure and roughness of the silk surface (**Figure 4b2**) is altered in the case of pop-HMDSO silk compared to untreated silk surface.

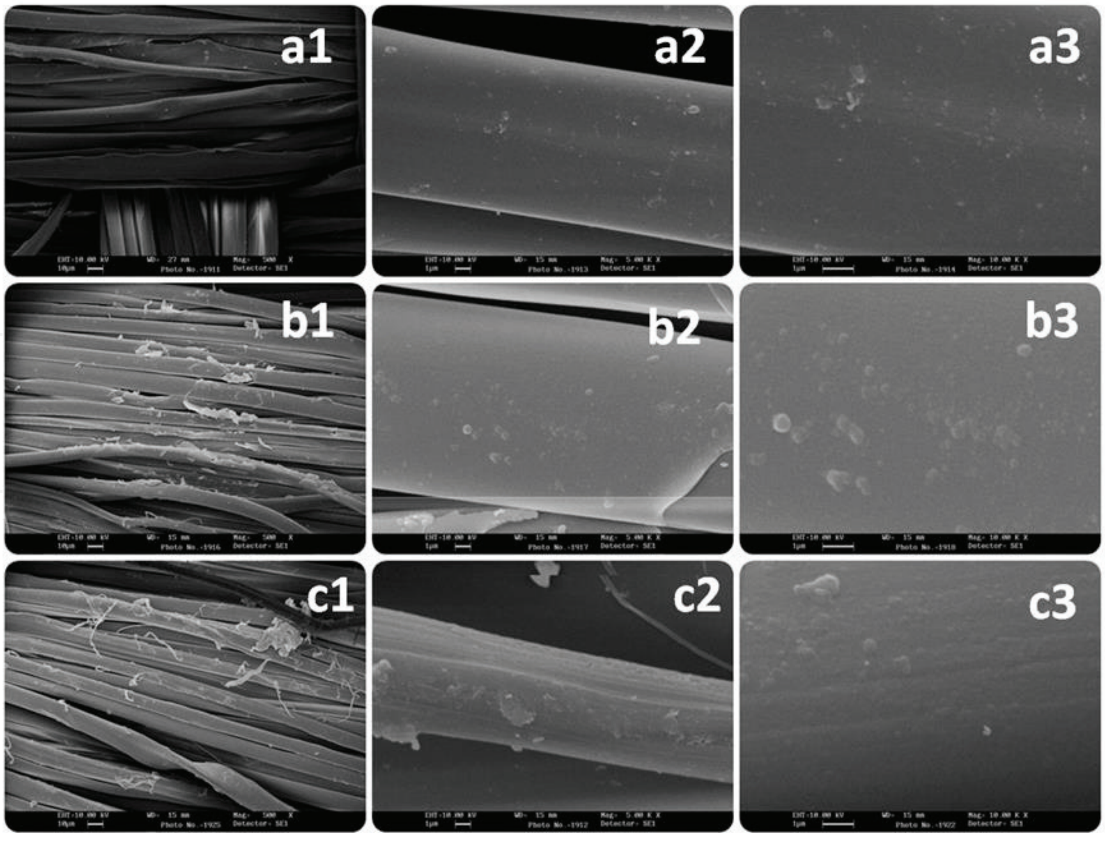

Figure 4. SEM images of (a) uncoated, (b) 100 W_7 min, (c) 100 W_15 min.

Similarly **Figure 4c1** shows the presence of a thin layer on the surface of the fabrics which would render the attachment of HMDSO, and **Figure 4c2** shows the higher magnification of fabrics with increased surface roughness. However, by increasing the deposition time at constant power, the deposition of HMDSO increases the surface roughness and promotes the hydrophobicity properties.

2.4.3. Contact angle

Contact angle measurements are carried out using 5 μl water as the polar liquid. The untreated silk fabric has rich hydroxyl, carboxyl, and amine groups on its surface which exhibit a hydrophilic nature. The contact angles and wet-out time results are listed in **Table 2**.

When a thin layer of HMDSO is coated on the surface of the fabric, the fabric becomes hydrophobic due to the presence of $(Si{-}CH_3)_3$ rocking, $Si{-}O{-}Si$, and $Si{-}CH_3$. **Figure 5** shows

Operating power (W)	Exposure time (min)	Wet-out time (s)	Contact angle (°)
—	0 (Untreated)	44	—
100	7	>3600	134
100	15	>3600	135
150	7	>3600	140

Table 2. Contact angle and wet-out time of silk fabrics coated by HMDSO.

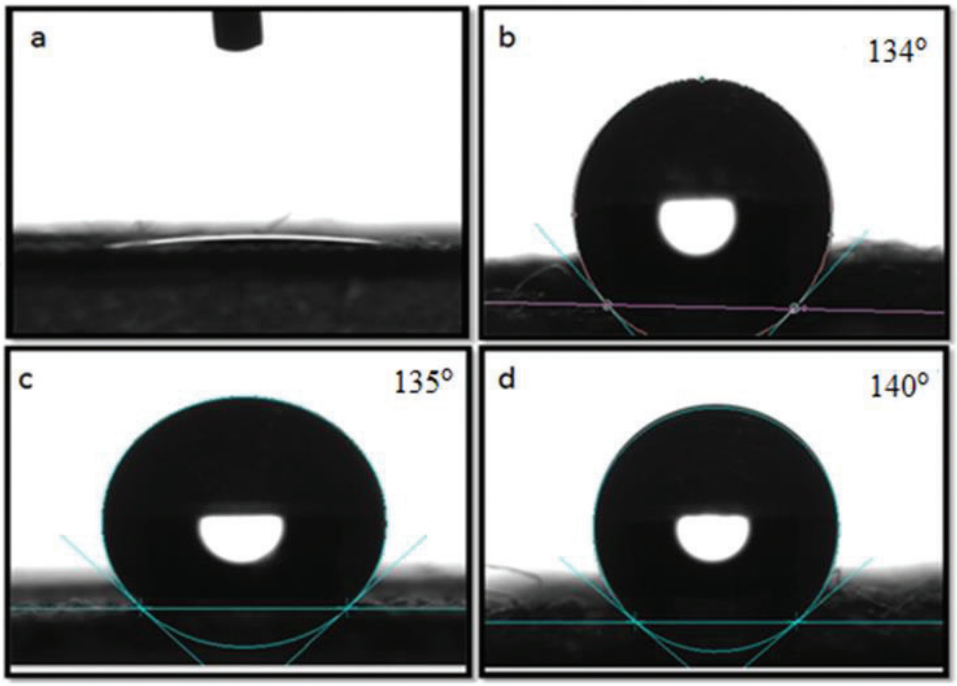

Figure 5. Static water contact angles of (a) uncoated, (b) 100 W_7 min, (c) 100 W_15 min, (d) 150 W_7 min.

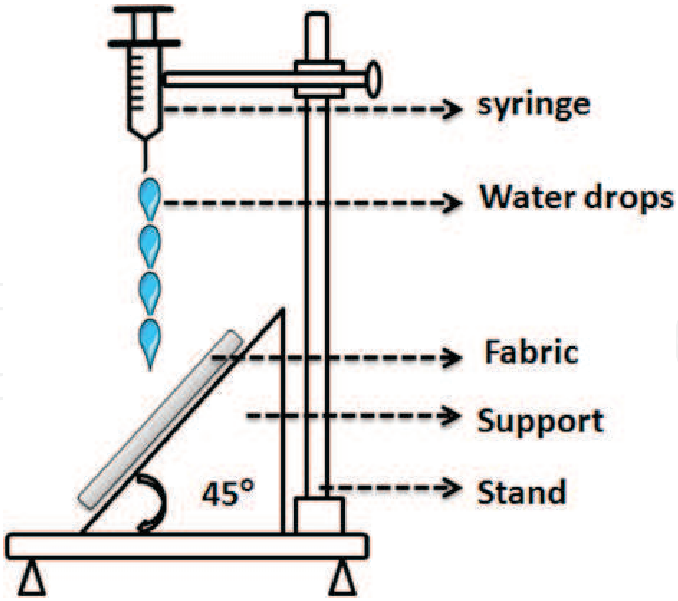

Figure 6. Experimental setup of the self-cleaning test.

a 5 μl water droplet on the silk fabric. It is seen that there is a drastic increase in the surface contact angle from 0° to 140° for untreated and HMDSO-coated silk fabric. **Table 2** represents the contact angle of HMDSO-coated samples for an exposure time of 7 min with the effect of discharge power 100 W (b) and 150 W (d), respectively. It is evident from the contact angle that more deposition is observed in samples treated with 150 W and its contact angle is 140°. From the above discussion, it shows that lower treatment time (7 min) and higher discharge power (150 W) result in more deposition on the fabric. However, there are no significant changes in the wet-out time of all HMDSO-coated fabrics.

2.4.4. Self-cleaning ability test

Water repellency spray tester of AATCC Test method 22-2005 is modified to test the self-cleaning ability [47]. **Figure 6** shows the experimental setup of the self-cleaning ability of the fabric. Graphite powder is spread on the HMDSO-coated fabrics. The fabric is tilted and placed at the center of the tester on a 45°-angle slope, and by using the syringe, water drop is allowed to fall on the fabric surface from a distance of 150 mm. The water droplets easily roll off along with contaminated surface and remove contamination from the fabric.

2.4.5. Home laundering and aging effect

To investigate the durability of HMDSO-coated silk fabric, the fabrics are washed with detergents. After detergent washing, there is a slight decrease in the contact angle, which will not affect the hydrophobic properties of the fabric. The aging effect of HMDSO-coated fabrics reveals that there are no significant visual changes in the wettability of silk fabrics after 100 days of deposition of the coating, which suggests good durability of the treatment. **Figure 7a** shows the common stains like ink, tea, milk, turmeric, and orange juice tested on the surface of both fabrics. In HMDSO-coated fabrics, all the stains are bedded like ball droplet.

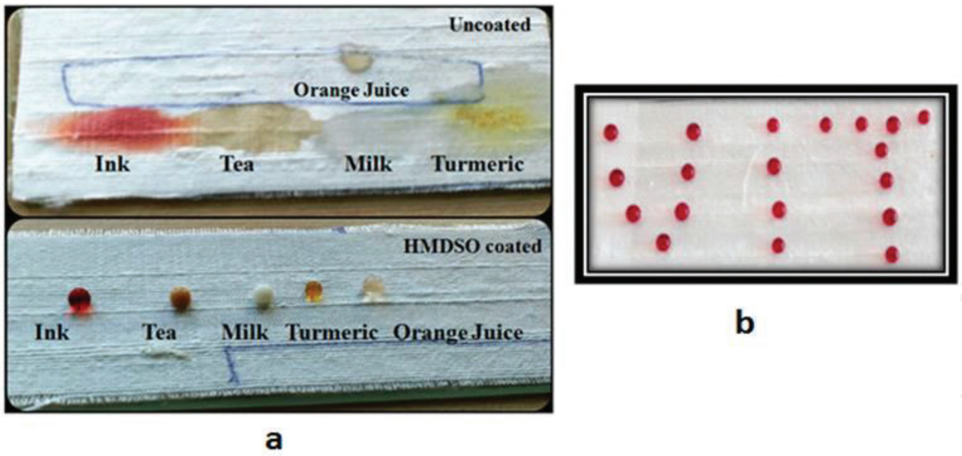

Figure 7. Photograph of stain with different kinds of liquids on the uncoated and HMDSO-coated fabric after 100 days.

In the case of untreated fabric, it's fully observed and stains the silk fabrics. **Figure 7b** shows an aging effect after 100 days, the ink droplet on the fabric bedded on the surface of the fabric.

3. Conclusion

The enhancement of hydrophobicity of the silk fabric has been achieved by deposition of pure HMDSO using plasma-enhanced chemical vapor deposition technique. The hydrophobic property has a great potential in the textile industry for stain-free fabrics. It is evident from the contact angle that more deposition is observed for a shorter exposure time along with higher discharge power (150 W) and its contact angle is 140°. Functional groups of HMDSO coating have been confirmed by ATR-FTIR spectroscopy; SEM images show the altered surface morphology of PP-HMDSO-coated silk fabric. Contact angle measurements reveal that the surface of silk fabric becomes more hydrophobic after deposition of PP-HMDSO. Coated fabrics are capable of repelling most aqueous liquids and dirt particles.

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