Hydrophobically modified cotton fabric assisted separation of oil-water mixture

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ABSTRACT

Superhydrophobic-superoleophilic fabrics were prepared and evaluated for oil–water mixture separation efficiencies. The nano-TiO2 and nano-SiO2 based coatings were done on the surface of the cotton fabric to create nanoscale roughness over the surface which was further modified by low energy material 1, 1, 3, 3-Hexamethyldisilazane (HMDS) and, polydimethylsiloxane (PDMS). Particle size and stability of prepared sol were characterized by particle size analysis and zeta potential. Coated cotton fabric samples were characterized by contact angle, contact angle hysteresis and surface free energy for its hydrophobic nature. Surface morphology was studied by scanning electron microscopy (SEM). The coated fabrics were found to be hydrophobic with low surface free energy values. The maximum contact angle was found to be 133° and lowest contact angle hysteresis was 5°. SEM confirmed the appearance of nanoscale surface roughness after coating of sols on cotton fabric. The average particle size and zeta potential values of silica sol was 61 nm and 137 mv whereas for titania sol it was found 344 nm and 200 mv, respectively. The oil–water separation efficiency of coated fabric was also observed by a different oil–water mixture. The coatings were found to be hydrophobic in nature and seem to be very useful for oil–water mixture separation.

Key words: coating, contact angle, separation, superhydrophobic

HIGHLIGHTS

- Nanosilica (SiO2) and nanotitania (TiO2) were used for hydrophobic-oleophilic coating.
- Sols were hydrophobically modified with HMDS and PDMS.
- Sols were coated on fabric, to create roughness over the surface.
- Coated fabrics were characterized by SEM and contact angle measurements.
- Application of coated surface for oil–water separation was performed.
- More than 80% of efficiency towards different oil–water mixture.
INTRODUCTION

Most of the industries such as textiles, foods, petrochemicals, mining, etc. drain their waste in the form of oil-water emulsion. It has become a very common pollutant and very serious environmental concern. It can also damage machinery and can be dangerous for the environment and animal life. There are several methods used to separate oil and water including using oil spill kits, centrifugation, distillation, industrial filtration, settling tanks, magnetic separation, skimmers, flotation technology, etc. These conventional methods are not very efficient and the remaining oil in the mixture can cause blockage of pipes in the treatment plant which further require frequent cleaning of these pipes or sometimes replacement is also required (Mueller et al. 2003). Hence, due to low efficiency of conventional methods the oil–water separation from the effluent become costlier.

There is need of further development in traditional methods for oil–water separation so that they can fulfil recent and future demand, can purify large volumes of mixture, low in cost, environmentally friendly, easily recyclable/reusable, with high efficacy (Yu et al. 2016, Gupta et al. 2017; Bhushan 2019).

To combat with the problems associated with conventional methods of oil–water separation, recently superhydrophobic, super-oleophilic coatings have drawn interest towards oil–water separation. The most recent study for oil–water separation is based on superwettability: either it is superhydrophobic, superhydrophilic, superoleophilic, or superoleophobic (Cortese et al. 2014; Gupta et al. 2017; Cheng et al. 2018). These types of coatings can be done on different types of fabrics, plastic or metal mesh which can be further used to separate oil–water mixture as due to hydrophobic nature of coated material the water remain upside and due to hydrophilic nature the oil flow down from the coated fabric or mesh. This type of oil–water separation strategy also provide a simple and hassel free operation compared to conventional oil–water separation methods.

Various materials modification methods used for the oil–water separation technologies are summarized in Figure 1.

Cheng et al. prepared a bio-based superhydrophobic coating on cellulosic fabric, which provides the oil–water separation efficiency around 97.5% (Cheng et al. 2018). Cortese et al. prepared superhydrophobic, super-oleophilic fabric by deposition of diamond-like carbons by plasma enhanced chemical vapour deposition (PECVD) method showed high efficiency for oil separation (Cortese et al. 2014). Shang et al. fabricated a superhydrophobic, super-oleophilic cotton fabric using silica nanoparticles, showing oil–water separation efficiency up to 98.2%. Also, the coated fabric was found to be reusable up to more than 30 separation cycles (Shang et al. 2018).
Apart from oil–water separation, the superhydrophobic textiles also have multifunctioning such as self-cleaning, anti-fogging (Park et al. 2015), anti-bacterial (Xing et al. 2007; Singh & Singh 2017), UV resistant, antifouling (Pan et al. 2019), photo-catalyst, (Xing & Ding 2007; Park et al. 2015), self-healing (Xue et al. 2016), flame-retardant (Mahltig & Böttcher 2003; Brancatelli et al. 2011; Ismail 2016), etc. which make them an efficient and long lasting material for oil–water separation applications.

Surface roughness and low surface energy are the key factors which are responsible for the superhydrophobicity of any surface. The surface roughness can be created by nano-dimensional coating over the surface. Generally, SiO₂, TiO₂, ZrO₂, Fe₂O₃, Al₂O₃ nanoparticles are used to coat the surface with desired roughness. The second-factor low surface energy can be achieved by modifying the coating by low surface energy material. These materials tend to decrease the surface free energy of the solid surface. Hence, by coating a mixture of nanoparticles and low surface energy material leads to modification of fabric surface to give superhydrophobic and super-oleophilic properties.

The sol-gel method was initially used to prepare inorganic–organic polymers but nowadays it has become a very successful and most used method in coating technology. Textiles are also coated by the sol-gel method because of easy processibility, low temperature, and cost-effective, high homogeneity with various functionality (Aslanidou & Karapanagiotis 2018; Cheng et al. 2018). For textile applications, the sol can be deposited onto textile fabrics in different ways, it may be by dip-coating, rolling, padding, spin coating or spray coating. By using this technology, fabrics can provide multi-functional characteristics with flame retardant, anti-mosquito, water-repellent, wrinkle resistant, anti-bacterial, ultraviolet (UV) resistant, and self-cleaning properties. Generally, artificially prepared superhydrophobic surfaces possess less stability and low mechanical durability (Zhang et al. 2013).

Most of the researchers utilized multistep processes involving specialized equipment and use of costly chemicals for fabric coating. The main objective of the present study is to prepare superhydrophobic–super-oleophilic cotton fabric by simple coating for the separation of oil–water mixture. These types of coatings repel water and absorb oil, so oil can be filtered from coated material and water can be separated from oil and related wastes. Also, hydrophobicity will be evaluated by contact angle, surface free energy and contact angle hysteresis. Oil–water separation efficiency of the coated cotton fabric will be evaluated for various oil water mixtures.

**MATERIALS AND METHODS**

**Materials used**

Titanium isopropoxide, tetra ethoxy orthosilicate, ammonium hydroxide, ethyl alcohol, hydrochloric acid (HCl), acetic acid, hexamethyldisilazane, polydimethylsilazane, and cotton fabric. All reagents used were of laboratory-grade reagents and doubly distilled water was used throughout the experiments.

**Preparation of Sol**

The titania sol (TiO₂) was prepared by hydrolysis of titanium isopropoxide in the presence of an acidic medium (HCl and CH₃COOH) and water was taken as solvent. Acidic solution was prepared by diluting glacial acetic acid (CH₃COOH)
and concentrated hydrochloric acid (HCl) with distilled water to attain 6% and 1.6% concentration, respectively. 250 mL of acid solution was taken in a conical flask and heated to 60°. Further 15 mL of titanium isopropoxide solution was added dropwise with constant stirring which was continued for 2 h at 800 rpm on magnetic stirrer at room temperature. After constant stirring for 2 h at 60 °C, the nano dispersed titania sol was prepared. The sol was kept for four days at room temperature to complete the hydrolysis and condensation reaction. The pH of prepared sol was found to be 2.5.

The silica (SiO₂) sol was prepared from hydrolysis of tetraethylorthosilicate (TEOS) in the presence of ammonium hydroxide, which was used as a catalyst for the reaction. 20 mL TEOS precursor was taken and diluted with 20 mL of ethyl alcohol. 1 mM NH₄OH was added dropwise with constant stirring which was continued for 2 h at 800 rpm on magnetic stirrer at room temperature. For the completion of the reaction, the sol was kept for four days at room temperature. The final pH of prepared sol was found to be 9.8.

Coating of fabric

Small pieces (3 cm × 3 cm) of cotton fabric were taken, washed with detergent, and dried in an oven. After that, the sol was coated on pre-cleaned and dried cotton fabric by the dip-coating method. The fabric pieces were dipped in prepared titania and silica sol 3 times at intervals of 5 min. Finally, the coated fabric pieces were dried in oven for 2 h at 70 °C.

Hydrophobic modification of coated fabric

Titania and silica sol-coated fabric pieces were separately dipped in previously prepared 2% polydimethylsiloxane (PDMS) in toluene solution and 1:1 (v/v) HMDS in ethanol solution. Hydrophobically modified fabrics were dried in the oven for 70 °C for 2 h. Drying at high temperature results in more hydrophobic behaviour due to transition from Wenzel state to Cassie-Baxter state which is having more air in micronanostructure to hold the liquid drop. In the present study we found the maximum bearable temperature for drying of modified fabric was 70 °C; after that the coated fabric started burning. Titania sol coated fabrics modified with PDMS and HMDS are coded as TP and TH, respectively. Similarly, silica sol coated fabrics modified with PDMS and HMDS are coded as SP and SH, respectively. The overall method of fabrication of fabric is illustrated in Figure 2.

CHARACTERIZATION

Contact angle and contact angle hysteresis

Contact angle is the measurement of contact of any liquid with the solid surface. More will the contact angle, more will be hydrophobicity. The hydrophobicity of coated fabric surfaces were observed by a drop shape analyzer DSA25, Kruss, Germany. The 5 μL water droplet was poured on the coated surface by an automatic dispensing unit and the contact angle was observed.

The contact angle hysteresis

Contact angle hysteresis defines the homogeneity of the surface. Homogeneously rough surfaces make lower contact angle hysteresis. Contact angle hysteresis is the difference between the advancing angle and receding angle. It was measured by the volume change method. The volume was increased by pouring liquid to the surface until the maximum angle (advancing angle) was found and poured liquid is sucked back until the minimum angle (receding angle) was found. The difference between these two is called contact angle hysteresis. Advancing angle and receding angle was also observed by using drop shape analyzer DSA25, Kruss, Germany.
Surface free energy

Surface free energy (SFE) is the measurement of intermolecular forces acting over the solid surface. A large amount of attraction means it will attract the liquid drop towards it and the surface free energy will be higher. If the surface has lower attractive forces it tends to repel liquid droplet and lower will be surface free energy.

SFE is the amount of extra energy the surface has compared to its bulk. Lower surface energy refers to more hydrophobicity. The surface free energy of prepared coating was also observed by using drop shape analyzer DSA25, Kruss, Germany. For the observation of surface free energy the two kinds of liquid, one polar and one non-polar were poured on the surface one by one. The surface free energy was automatically calculated by the software utilizing different contact angle values by polar and non-polar liquids.

Scanning electron microscopy (SEM)

SEM was used to observe the morphology of the coated fabric surface. The analysis was done by using field emission scanning electron microscopy (FESEM) MIRA3b TESCAN, USA.

Particle size analysis and zeta potential

Particle size distribution and zeta potential are very important parameters to analyze particle size distribution, stability, and surface charge distribution of nanoparticles in colloidal suspension. Particle size analysis is performed by the dynamic light scattering (DLS) method. When a beam of laser light is scattered by a group of particles present in the solution, the angle is formed by scattered light. This angle is inversely proportional to particle size. Zeta potential is the potential, which is observed over the surface of sol and arises between nanoparticle charge and thin layer of charge over the surface. The magnitude of zeta potential is responsible for its stability. Zeta potential is defined as the potential difference between the dispersion medium and the stationary medium. The particle size and zeta potential of prepared sols were analyzed by Microtrac Nanotrac, wave II.

Water and oil separation efficiency

A specific amount of oil and distilled water (pH 7) was taken in 1:1(w/w) ratio and mixed together. To differentiate colourless liquid like chloroform, and hexane, toluene malachite green dye was added in water, and for coloured liquids like mustard oil, petrol, and diesel, no dye was added. The coated fabric was placed in a filtration assembly. The mixer of liquid and oil was poured in Buchner funnel, where the coated fabric was placed for separation. Dose size of oil–water mixture for analyzing separation efficiency was 100 gm having 50 gm distilled water and 50 gm oil and the separation was done at room temperature. The oil was get filtered from coated fabric due to its oleophilicity. The mixed solution was separated and after separation weight of separated oil was noted. The procedure of separation is shown in Figure 3.

The percentage oil–water separation efficiency (X) was calculated by using the following formula:

\[ \% \text{oil water separation efficiency} (X) = \left( \frac{M_1}{M_2} \right) \times 100 \]

where M1 is the weight of oil separated and M2 is the weight of oil taken. Water–oil separation efficiency of coated fabric was determined by using different types of oil phase such as chloroform, hexane, toluene, mustard oil, petrol, and diesel and compared. To know the reusability of fabric, the contact angle of coated fabric was again measured after one-time separation.

RESULTS AND DISCUSSION

Contact angle

Contact angle is the angle formed between liquid–air interphase and solid–air interphase, when the liquid droplet is poured on the surface. The lower the contact of any liquid with solid makes a higher contact angle resulting in a higher degree of hydrophobicity. The coating, contact angle, and contact angle hysteresis of coated cotton fabrics are given in Table 1.

Droplet behaviour on various surface are shown in Figure 4. Images of contact angles are shown in Figure 5. HMDS based coatings (SH and TH) show high contact angles 133° and 125° for silica and titania, respectively. High contact angle in the case of HMDS based coatings attributed to the fact that HMDS has a low surface tension value and hence it decrease the surface energy of the coated surface. PDMS-based silica and titania coatings, SP and TP were also hydrophobic, showing contact
angle 118° and 110°, respectively, but the values are lesser than HMDS based coatings. PDMS is hydrophobic material showing contact angle 100° but it will not decrease surface energy in large extent as in case of HMDS treated coating.

Particle size analysis and zeta potential
The particle size distribution of silica nanoparticles in silica sol was found in the range of 30–243 nm and the average value of silica nanoparticles observed was 61 nm. A narrow distribution of silica nanoparticles was observed in prepared silica sol and represents the more homogenous nature of silica sol. The particle size distribution of titania nanoparticles in titania sol was found to be in the range of 9.03 to 1944 nm and the average size of particles are 18 and 344 nm. The distribution of titania nanoparticles is not very good distribution as compared to silica nanoparticles, which can be seen in Figure 6. But the stability
of both sols is very high, which can be analyzed by zeta potential values. Zeta potential of silica sol and titania sol were observed at 137 mV and 200 mV, respectively. The prepared sols are of higher stability with nano-sized particles. The nanometric range of sol is responsible for nanometric rough surface and more hydrophobicity of surface. The particle size distribution curve is shown in Figure 6.

**Scanning electron microscopy (SEM)**

SEM of uncoated fabric, silica-coated fabric, and titania-coated fabric were done to analyze the surface morphology of surfaces. The SEM images of surfaces are shown in Figure 7.

The difference in the morphology of coated and uncoated fabric can be seen easily, as uncoated fabric is smooth and no coating appeared in fabric. In coated fabrics, a layer of coating with aggregated particles of SiO$_2$ and TiO$_2$ appears. The coatings can be seen in every thread of the fabric. The coating distribution is more uniform in case of silica coatings compared to titania coatings, this is due to the narrow particle size distribution of silica nanoparticles in silica sol. Appearance of rough
surface due to presence of silica and titania nanoparticles over the coated fabric surface is responsible for the formation of rough and nanostructured surface which leads to hydrophobic behaviour of the coated fabric.

Surface free energy
A low surface free energy is responsible for hydrophobicity. All coatings are hydrophobic in nature showing low surface free energy. Hexamethyldisilazane (HMDS) with silica and titania in SH and TH fabric, showing less surface free energy. HMDS decreases the surface energy to a greater extent due to its smaller molecular size and low surface tension and leads to more hydrophobicity. While PDMS with silica and titania in SP and TP fabric shows surface energy more than HMDS coated because of its polymeric nature, so PDMS coated fabric is less hydrophobic than HMDS coated. Surface free energy of all coated surfaces is given in Table 1.

Separation efficiency
The observed oil–water percentage (%) separation efficiency is shown in Table 2. All coated fabric showed significant separation efficiency for oil–water separation due to the hydrophobic and oleophillic nature of the coated cotton fabric. In case of mustard oil–water less, separation was observed compared to all other oil–water mixtures which is due to the more partial miscibility of mustard oil–water. Separation efficiencies are higher for chloroform–water, hexane–water, toluene–water because their miscibilities are almost negligible. In the case of mustard oil, petrol,

![Silica Particle on fabric fiber](image1)

![Titania Particle on fabric fiber](image2)

**Figure 7** | Scanning electron microscopy images of (a) uncoated fabric, (b) SH fabric, (c) SP coated fabric, (d) TH fabric, and (e) TP fabric.

| S. No. | Coating code | Chloroform | Hexane | Toluene | Mustard oil | Petrol | Diesel |
|-------|--------------|------------|--------|---------|-------------|--------|--------|
| 1.    | SH           | 93         | 90     | 93      | 84          | 89     | 88     |
| 2.    | SP           | 92         | 91     | 94      | 86          | 90     | 87     |
| 3.    | TH           | 91         | 93     | 95      | 87          | 88     | 89     |
| 4.    | TP           | 94         | 94     | 94      | 86          | 89     | 88     |

**Table 2** | Oil–water separation efficiency by differently coated surface
diesel, they showed less separation efficiencies compared to chloroform–water, hexane–water, toluene–water, because they show some miscible or partial miscible behaviour with water. The graphical representation of the separation of different oil–water mixture is shown in Figure 8.

After one-time filtration, the coated fabrics were again dried in an oven at 70 °C and contact angles were observed and shown in Table 3. SH and TH coating showed a small decrease in contact angle, but the hydrophobicity was not vanished, while the contact angle of SP and TP coatings remains the same. So it can be concluded from this, that the PDMS-based coatings provide more stability to coatings and can be reusable for further separation. No change in contact angle after separation

![Figure 8](image)

**Figure 8** | Separation efficiency toward different of (a) SP coated fabric, (b) SH coated fabric, (c) TH coated fabric, and (d) TP coated fabric.

| Table 3 | Contact angle values of coated surface before and after separation |
| S. no. | Coating type | Contact angle before separation | Contact angle after separation |
|-------|---------------|---------------------------------|-------------------------------|
| 1.    | SH            | 133°                            | 119°                          |
| 2.    | SP            | 118°                            | 118°                          |
| 3.    | TH            | 125°                            | 111°                          |
| 4.    | TP            | 110°                            | 109°                          |
in case SP and TP coating can be attributed to the fact that PDMS being a polymer is more adhesive to the surface while HMDS is not a polymer so it is less adhesive to the surface. Scheme of modification of silica coating by HMDS and PDMS are shown in Figure 9. This scheme is also applied for modification of titania coating.

Oil–water separation mechanism
The oil–water separation phenomenon can be explained by capillary action on the basis of observed contact angle formed by liquid on surface. When there is no coating on fabric surface, no capillary action is there, because of the big size of pores on the surface. The movement of droplet on surface is result of gravitational force. Capillary action is present when the capillary pores are smaller in size compared to droplet size. After coating, the pore size decreases and capillary action starts. According to Young-Laplace theory (Kim & Harriott 1987):

\[ \Delta P = \frac{4 \gamma_{SL} \cos \theta_{liq}}{D_{pore}} \]

where \( \Delta P \) is Laplace pressure, \( \gamma_{SL} \) is surface tension of liquid droplet against solid surface, \( \theta \) is the contact angle made by liquid droplet on the surface and \( D_{pore} \) is the pore diameter of solid surface.

By taking \( D_{pore} \) the same for all, when water is taken as poured liquid, and \( \theta \) is more than 90°, it means \( \Delta P \) is negative. Hence there is need of external pressure to penetrate through the surface. So it is not easy for a water droplet to penetrate coated fibre pore. When oil is poured at surface, the surface is oleophilic, and the formed contact angle is less than 90°. In this case \( \Delta P \) value will be positive, which shows there is no need of external pressure to penetrate the liquid though fabric pore diameter. The diagrammatic representation is shown in Figure 10.

Figure 9 | Scheme of modification of silica coating by HMDS and PDMS.

Figure 10 | Schematic diagram of oil–water behavior in uncoated and coated fabric.
Oil–water separation through superhydrophobic-super-oleophilic coating can be also explained on the basis of surface free energy of surface and surface tension of liquid. Oil can easily penetrate the coated fabric but water cannot. Oils have lower surface tension than water. The prepared surfaces also possess low surface energy. Therefore, the designed surfaces repel water but attract oil and allow it to go through.

CONCLUSIONS

Silica and titania nanoparticle-containing sols were prepared for coatings and further modified with hydrophobic modifiers. All prepared coatings showed hydrophobic nature which possesses low surface energy and contact angle hysteresis. HMDS modified silica and titania coatings were more hydrophobic and showed high contact angle while PDMS modified silica and titania coatings showed comparatively low contact angle. These coatings are very useful for oil–water separation. Prepared sol are found to be very stable as confirmed by zeta potential values. SEM images confirmed the presence of silica and titania nanoparticles on the fabric surface which impart nanoscale surface roughness. Various oil–water mixtures were separated by using coated cotton fabric and in most of the cases about 90% of separation efficiency was achieved. In the case of mustard oil–water mixture the separation efficiency was found lower with all coated cotton fabrics which showed that with high viscous oil separation efficiency is lower. Hence, separation of high viscous oil–water mixture is quite challenging and more architectural alteration on the surface is required for better separation efficiency.

DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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