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Facile approach to develop durable and reusable superhydrophobic/superoleophilic coatings for steel mesh surfaces

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HIGHLIGHTS

- Superhydrophobic coating by chemical etching and HDTMS.
- Characterization of coating by surface morphology and water contact angle.
- Examine the wetting stability of coatings under perturbation conditions.
- Coating exhibits excellent separation efficiency for oil-water separation of kerosene-water and hexane-water system.

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ABSTRACT

Oil spills on the surfaces of bodies of water are now a great concern as they affect marine creatures and the ecosystem. Therefore, a facile technique for removal of oil from water is of much interest. In this study, superhydrophobic and superoleophilic coating is synthesized on stainless steel mesh via chemical etching using a mixture of FeCl_3 and HCl followed by treatment with hexadecyltrimethoxysilane. Surface morphology analysis shows the presence of flower like microstructures on the surface after treatment. Superhydrophobic and superoleophilic properties have been achieved on stainless steel mesh with water static contact angle of $167^\circ \pm 3^\circ$, water tilting angle of $6^\circ \pm 1^\circ$, and oil static contact angle of nearly 0° . The coating shows excellent thermal, chemical and mechanical stability. Kerosene-water and hexane-water mixtures were successfully separated via a simple filtering process using coated steel mesh with a separation efficiency of more than 98%. This approach can be implemented on any shape or size of stainless steel mesh and will have industrial applications.

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1. Introduction

Oil spills, oil handling and transportation accidents are the leading causes of pollution in bodies of water. Pollution has an adverse effect on the eco-system and marine animals. There is a need for effective and low cost methods for separation of spilled oil from water [1]. Various conventional methods such as filtration [2], oil-skimmers [3], magnetic separation [4], and floatation techniques [5] are used for removing oil spills from bodies of water. Disadvantages of these conventional techniques are expensive equipment, high processing cost, long processing time, low separation efficiency, poor recyclability, and generation of secondary pollutants. It is, therefore, essential to develop surfaces which can

easily separate oil spills from the surface of water that are fast and have minimum environmental impact.

Oil spills can be removed from the surface of water based upon the differing wetting properties of oil and water. A surface can be modified to be both superhydrophobic and superoleophilic. This type of surface repels water and allows oil to flow through it, thereby separating oil from an oil-water mixture [6–8]. Superhydrophobic and superoleophilic surfaces have water static contact angle $>150^\circ$ and static oil contact angle less than 10° . Superhydrophobic surfaces are inspired from nature such as lotus leaf [9], rice leaf and butterfly wings [10,11]. Recently, for oil water separation, superhydrophobic coatings have been created on different porous substrates such as mesh [6–8], fabrics [12,13], filter paper [14,15] and sponges [16]. The use of these superhydrophobic surfaces also has certain problems like poor reusability and low separation efficiency. After adsorbing of certain amount of oil, the separation efficiency of these surfaces reduces as the surface

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and the bulk gradually retains more oil. Therefore, continuous removal of oil from an oil-water mixture by mechanical means is required.

Stainless steel mesh is used for oil water separation because of its porous property and also for its superior physical, chemical and mechanical properties, wide availability, and relatively low cost. Many studies have been reported using different coating techniques for synthesis of superliquiphobic/philic coating on steel mesh. For instance, Brown and Bhushan [6,7] synthesized superoleophobic/superoleophilic coating using poly(diallyldimethylammonium chloride) (PDDA) binder, SiO₂ nanoparticles and fluorosurfactant for oil-water separation by layer-by-layer technique. The prepared samples were mechanically stable to wear and anti-smudge tests. Lee et al. [17] fabricated superhydrophobic/superoleophilic coating on mesh, using electro-spinning of polystyrene, for separation of diesel oil from water. Varshney et al. [18] coated the surface of the aqua regia etched steel mesh with lauric acid to achieve a superhydrophobic/superoleophilic property with thermal, chemical and mechanical stability. Hexadecyltrimethoxysilane (HDTMS) is used as a low surface energy material to create superhydrophobicity because it forms a self-assembled monolayer (SAM) on the surface of the substrate [19]. In this process, the hydrophilic head (-SiOMe) reacts with surface -OH bonds and thus forms a chemical bond with the surface which becomes covered with the molecules of HDTMS and forms a hydrophobic layer. Although several works have been published on superhydrophobic coatings for stainless steel mesh using various low surface energy materials, no detailed study has been done using HDTMS as a low surface energy material. Recently, Yu et al. [20] fabricated superhydrophobic coatings on cotton, stainless steel, glass and paper by drop-casting HDTMS modified raspberry-like silica coated PS microspheres for oil-water separation. Yeom and Kim [21] coated HDTMS modified SiO₂ nanoparticles on steel mesh and sponge to fabricate a superhydrophobic surface for oil-water separation. Wang et al. [22] synthesized a dual-scale hierarchical structure on steel mesh to create a superhydrophobic coating sol-gel of HDTMS and silica coated PS particles. Unfortunately, in these studies, the stability of the coating under harsh conditions was not studied which is an important factor for industrial applications.

In this study, we simplify the coating technique for stainless steel mesh by avoiding the use of any functionalized particles. Chemical etching with a mixture of ferric chloride (FeCl₃) and hydrochloric acid (HCl) followed by immersing the etched sample in HDTMS was carried out to achieve the superhydrophobic and superoleophilic properties. To make the coating industrially applicable, the mechanical, chemical and thermal stability of the coating was also investigated. Further, gravitational oil-water separation application and the separation efficiency of the mesh with different oil-water systems were also studied.

2. Experimental details

2.1. Materials

Commercially available stainless steel meshes with opening sizes of $634.2 \pm 15 \mu\text{m}$ ($4 \text{ cm} \times 4 \text{ cm} \times 0.5 \text{ mm}$) and $239 \pm 10 \mu\text{m}$ wire width were used as substrates. FeCl₃ and HCl (35%) were purchased from Himedia Laboratories Pvt. Ltd., India. Toluene (Merck Specialties Private Limited, India) was used as the solvent for the experiment. Hexadecyltrimethoxysilane (HDTMS, CH₃(CH₂)₁₅Si(OCH₃)₃, $\geq 85\%$ GC) from Sigma-Aldrich Co., USA was used for the fabrication of superhydrophobic and superoleophilic coating. Millipore water of $18.2 \text{ m}\Omega\text{ cm}$ resistivity was used throughout the experiment.

2.2. Chemical etching of the steel mesh

Steel mesh was first washed thoroughly with normal water followed by Millipore water. The substrate was also cleaned in a water bath ultrasonicator for 10 min. After cleaning, the substrate was dried for 2 h at 60 °C in a hot air oven. For etching, 50 mL solution of FeCl₃ (2 mol/L) and HCl (4:1 vol%) was prepared. The cleaned mesh was then immersed in the solution for 7 min. After that, the substrate was washed with Millipore water and dried at 60 °C for 12 h.

2.3. Synthesis of superhydrophobic coating

A sol-gel HDTMS solution was prepared by adding 2% (v/v) of HDTMS in 40 mL toluene at ambient temperature. The sol-gel was stirred for 15 min before coating. Dried etched mesh was immersed for 10 min inside the sol-gel. To remove the solvents, samples were oven dried at 120 °C for 1 h. After drying, the samples were used for different characterizations.

2.4. Physical and chemical characterization

Contact angle measurement was carried out by Drop Shape Analyzer (25, Kruss, Germany) with a droplet of distilled water having a drop volume range of 4–6 μL . The experiments were carried out in ambient temperature and were repeated at five different places on each sample, and their average with standard deviation was calculated. Surface morphology of the coated sample was studied by scanning electron microscopy (SEM) (Nova Nano SEM FEI). Energy-dispersive X-ray spectroscopy (EDS) was carried out for elemental analysis on the coated and uncoated surfaces. The presence of a functional group on the surface was examined by Fourier Transformation Infrared Spectroscopy (FTIR) (Thermo Fisher Nicolet iS10). Optical images were taken from Nikon DSLR Camera (50 frames per second).

2.5. Thermal, chemical and mechanical durability tests

To test the thermal stability, the coated sample was annealed for 1 h in a hot air oven at different temperatures (120–480 °C). The sample was then taken out and cooled for 1 h at ambient temperature, and water contact angle was measured. To test the chemical stability, the coated sample was immersed in an aqueous solution of HCl and sodium hydroxide (NaOH) with pH ranging from (2 to 14). The contact angle of the sample was measured at 1 h regular intervals of immersion. Additionally, the sample was also immersed in hexane and kerosene to examine the stability of coating.

Mechanical stability of the surface was examined by abrasion, surface bending and twisting tests. The abrasion test was carried out by rubbing the sample on sand paper over a distance of 6 cm in a single direction with 0.5 N weight (pressure of $\sim 1590 \text{ Pa}$) over an area of about 3.14 cm^2 . The effect of mechanical stress on the wettability of the coating was studied by bending and twisting the samples into different shapes and measuring the water contact angle of the surface in the bend or kink region.

2.6. Oil-water separation studies

Oil-water separation experiments were performed by using different ratios of oil (hexane ($\gamma = 18 \text{ mN/m}$) [23] and kerosene ($\gamma = 30 \text{ mN/m}$) [24]) and water mixtures (1:1, 4:1 and 1:4). The prepared solutions were then poured on the coated mesh. Oil passed through the mesh whereas the water remained on the surface and was later separated out into another beaker. The separa-

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