Chapter 2

Design and Fabrication of Nonwoven Fabrics for Oil/Water Separation Applications

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ABSTRACT

Considering that oil spills and oil-containing wastewater seriously damaged to marine life ecosystems, sustainable, environmentally friendly, and efficient oil/water separation technology has become an inevitable trend for environmental pollution prevention. Fabric materials are accepted in the class of environmentally friendly materials for water treatment processes due to their low cost and good degradability. fabrics with natural hydrophilic/oleophilic properties provide super-wettable surfaces in the separation of oil/water mixtures and complex emulsions consisting of different components. Among the fabric types, nonwoven fabrics form a basis to produce superhydrophobic nonwoven fabrics due to their short production processes, low costs, high mechanical properties, and three-dimensional porous structures. Superhydrophobic nonwoven fabrics are among the functional nonwovens researches due to their selective properties against water and oil, as well as their resistance to water and pollution, antibacterial activities, and self-cleaning properties. In this review, firstly the theories of wettability and separation mechanisms depending on specific wettability are explained, and the effects of nonwoven fabric structure on superhydrophobicity are presented. Subsequently, nonwoven-based functionalized surfaces for oil-water separation applications reported in the literature are explained in detail and lastly, in the conclusion section the challenges and expected future research directions are indicated. Considering the recent interest in studies in which nonwoven fabrics are used as a base surface and the increase in publications, it is thought that this review will benefit researchers as a guide that systematically presents the current situation to guide the development of functional structures that separate oil/water.

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1. INTRODUCTION

In recent years, as the increase in oil production due to the rapid development of industries using oil resources and the accidents in the transportation process cause oil spills, and in addition, industrial wastewater poses threats to the ecosystem, all these situations threaten the sustainable development of humanity[1-10]. Oil spilled into the sea causes environmental and ecological damage by threatening marine life, biodiversity and affects human health due to decomposition of oil into harmful chemicals[11-14]. Accordingly, the selection of the method to be used to separate the oil from the water is very important. Various approaches have been developed and used for oil/water separation, such as gravity separation, in situ incineration, electrolysis, chemical dispersion flotation, and centrifugation. However, in general, disadvantages such as non-recyclability in general, high cost and environmental pollution, and low efficiency emerge with the use of these methods[10],[15]. Porous materials, which have recently been preferred in both commercial applications and research studies, are frequently used in the fields of self-cleaning, stain sensor, tissue scaffolds and oil/water separation[16–20]. For these reasons, there is a need for the development of sustainable, environmentally friendly, new materials that are efficient for water/oil separation. Especially in the last two decades, wettability materials with superhydrophobic (SHB) surface properties have been proposed as functional materials for the separation of oil/water mixtures and emulsions, and many scientific articles have been published in this context(Fig.1)[15]. There is a need for higher requirements on separation techniques for the purification of oils in emulsified form. Therefore, it is important to search for high-efficiency, low-cost and environmentally friendly separation techniques[1]. Particularly recently, the focus has been on bio-inspired SHB materials with high selectivity for oil separation from water[16,17].

The functional surfaces of materials with high absorbency are expected to effectively filter oily wastewater[16,17,21]. SHB surfaces can be obtained by modifying the surface chemical composition and surface microstructure. Foams, polymeric membranes, sponges, metal nets, and woven/non-woven fabrics are the preferred SHB porous materials to be used for oil/water separation[12,15,22] and a schematic representation of the typical material used for oil/water separation was given in Fig.2. Among them, membrane separation is considered the most efficient base substrate based on its unique advantages such as environmentally friendly, energy saving, simple process, easy operation, and low maintenance cost[4,23–25].

Fabrics, which are widely used as porous materials, have flexible, rough structure and hydrophilic/oleophilic properties. Therefore, these properties facilitate the creation of efficient wetting surfaces for the controlled and effective separation of complex emulsions consisting of oil/water mixtures and other different components. Differentiating from other porous materials with these properties, fabrics serve as promising efficient, environmentally friendly and comprehensive platforms for water treatment due to the many advantages they offer: (1) fabrics in woven structure improve mechanical properties; (2) fabric structures can offer micro/nano level roughness structure; (3) fabrics have a porous structure to facilitate the movement of liquids; and (4) and in terms of sustainability, the fabrics are degradable, accessible and inexpensive. On the other hand, fabrics show low resistance to heat, chemicals, and wrinkling. Because of these properties, fabrics present both opportunities and challenges, for the design and manufacture of superimpregnated fabrics in the preparation of oil/water separation systems[1].

Showing hydrophobic and oleophilic properties as soft and porous materials, non-woven fabrics (NWFs) have become an economical and readily available material for oil separation from oil/water mixtures and emulsions[26,27]. NWFs are considered as random sheets/webs of fibers which can be composed of single or different fiber compositions produced by bonding or clamping fibers together by chemical, mechanical, thermal or solvent methods[15]. NWFs are widely used in industrial, agricultural, medical and decoration fields thanks to their environmental friendliness, highly flexible structures, affordable costs and low-density properties. In addition, NWFs have enough space to grow micro and nanostructures and contact water and oil drops from different angles, due to their irregular and multi-layered network structure[28].

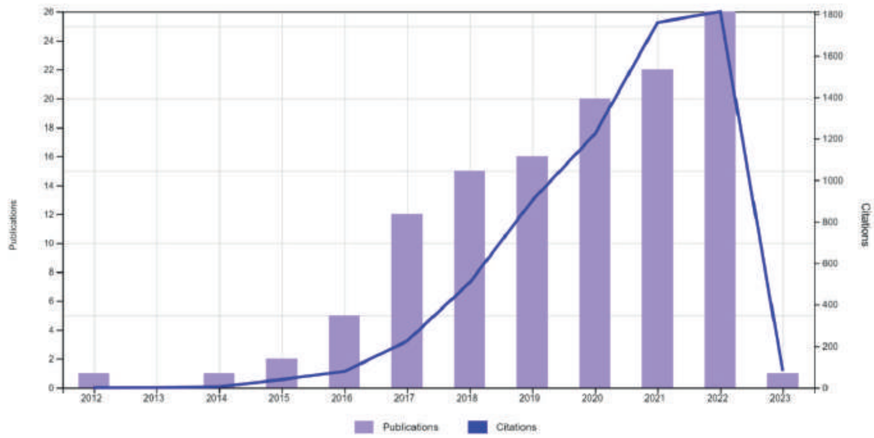


Figure 1. Numbers of articles and citations of oil/water separation indexed in Web of Science by the title of "oil/water separation".

For these reasons, different approaches have been adopted for the design and fabrication of superwetting NWFs to achieve efficient oil/water separation performance[15] and in literature there are limited studies related to NWFs design for oil/water separation applications. Table 1 provides the materials and methods used to fabricate the superhydrophobic NWFs. The use of environmentally unfriendly chemicals and high costs in these complex and expensive manufacturing processes indicate that NWFs may not be practical for large-scale applications. For this reason, studies on the use of sustainable materials and low-cost production methods are being conducted. Hydrophobic and oleophilic NWFs have superior properties such as random three-dimensional networks of fibers forming the fabric structure, good wetting behavior and flexibility. However, the fibers in the structure of non-woven fabrics produced by conventional methods have smooth surfaces that result in low wettability and oil absorption. Therefore, the development of superior micro/nano fabric structures for oil/water separation has become a prominent issue in fibrous materials research. Since the surface roughness of the fibers is a determining feature of the wettability capacity of the materials, there are many studies in the literature about the topographic structure of the surface and the application of micro/nano fabrics. Inspired by porous materials found in nature, such as very fine-pored meteorites and micro-grooved cactus, fibers with a rough surface have recently been studied to further improve liquid wettability. It has been found that the nanopores formed on the surface of the fibers greatly increase the fiber surface roughness, resulting in a superior level of oil adsorption efficiency[26].

Within the scope of the studies carried out, important research data were obtained by combining the advantages of membrane separation and special superwetting fabrics for the central optimization experimental design. Fabrics with high separation capacity and SHB/ superoleophilic (SOL) and superhydrophilic (SHL)/superoleophobic (SOB) surface properties are designed by producing fabrics with micro/nano structure surface and adjusting the surface energy of these materials. Fabrics with SHB/SOL properties that separate oil from oil/water mixtures or emulsions are defined as "oil-removing" materials (Fig. 3)[1,29]. Fabrics with SHL/SOB properties on the surface are defined as "water-removing" materials. On the other hand, fabrics with SHL/underwater super oleophobic (SHL/UWSOB) and UWSOB/underoil super oleophobic (UWSOB/UOSHB) properties are also being developed. In particular, janus and smart fabrics have the potential to be used in the separation of oily wastewater and oil spills, as they have a switchable wettability and can reverse switch between the "oil-removing" and the "water-removing" state. Janus fabrics have asymmetrical wettability with unique unidirectional and non-uniform properties. They can exhibit self-cleaning, self-healing and photocatalytic properties with special applications, while providing a sustainable separation of heavy oil/water mixtures in smart fabrics[30]. In addition, some fabrication methods like sol-gel approach, chemical etching method, polymer grafting and plasma assisted vapor deposition are important to facilitate the development of superwetting fabrics[1].

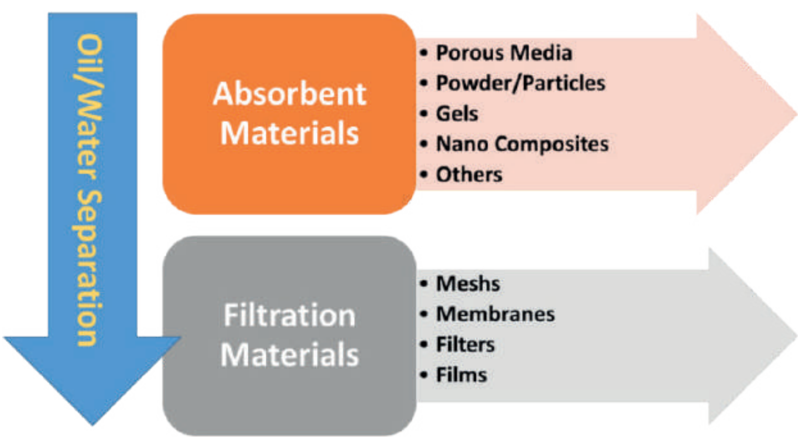


Figure 2. Schematic representation of the typical material used for oil/water separation[31].

Although progress has been made in recent years with varying degrees of success, practical application of oil/water separation has limited the efficiency of the structure due to oil contamination on the membrane surface. Because of this problem, a membrane with a low probability of contamination is desired from the membrane to be used. In particular, the superwetting membrane with SOB and SHL properties exhibits high affinity and selectivity behavior. While the hydration layer formed on the SHL surface passes water, it prevents the passage of oil content. SHB, on the other hand, may cause the opposite effect of oil accumulation on the membrane surface and preventing the passage of water. For these reasons, superwetting materials separate oil/water mixtures more efficiently and separation problem with surface contamination problem may not arise. In this case, the SHL membrane surface structure is suitable for gravity-driven oil/water separation than SHB membrane[32,33].

In a wettability-related approach, Tsuteja et al. described the operation of oil/water separation membranes with moisture sensitive surfaces using fluorodecyl polyhedral oligomeric silsesquioxane (F-POSS), which performs both SHL and SOB in air and underwater. This ‘water-removing’ material enables selective separation because the SOB surface structure allows only water to pass through in the mixture, so it is not affected by oil contamination and an interface is not formed that inhibits or slows the passage of water[34]. However, despite the design and development of water removing materials, the negative effects of fluorinated chemicals on the environment and complex synthesis steps limit their application areas and prevent their widespread use[33]. In recent studies, to overcome these problems, the structure of fish scales has been studied and other types of wettability surfaces that exhibit both SHL and UWSOB have been designed as water-removing, oil/water-separating materials[35]. These special surfaces do not create oil pollution and do not form a barrier to prevent water passage due to their UWSOB and SHL properties. However, although the use of these surfaces is advantageous, there are some difficulties because these surfaces are easily chemically attacked and degraded by aqueous environments with high or low pH in salty sea water as well as variable temperatures[33].

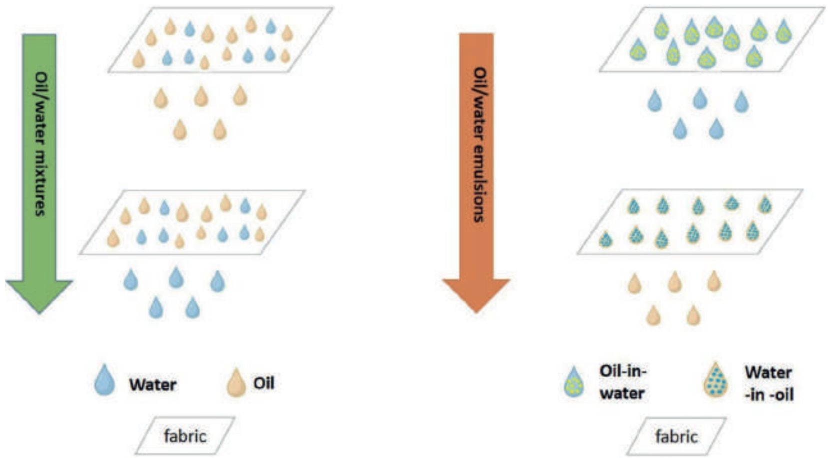


Figure 3. Schematic representation of SHB/SOL behavior for oil/water separation.

Microfiltration and ultrafiltration membranes developed from polyethersulfone (PES), polypropylene (PP), polysulfone (PSF) and poly(vinylidene fluoride) (PVDF) materials for separation oil/water mixtures or emulsions have been extensively investigated for their superwetability surface behavior[36]. However, most membrane separation materials are complex, costly, unsustainable, petroleum-based, do not degrade and are difficult to recycle therefore leading to environmental pollution[4]. Generally, contaminated separation materials are directly disposed of or incinerated after use, causing secondary pollution to the environment. Therefore, it is very important to use environmentally friendly, sustainable green materials in the production of membranes used for oil/water separation [37]. For these reasons, it is very important to be able to produce membrane materials that are easy, low cost and environmentally safe[4]. In recent years, polylactic acid (PLA), which is obtained from plant sources such as corn and cassava, has been approved for use in human biomedical and pharmaceutical applications by the Food and Drug Administration as an environmentally friendly polyester material due to its biocompatibility, high mechanical properties, and compostable properties. In this direction, in recent years, researchers have studied the suitability of PLA fiber-containing NWFs produced from PLA polymer for oil/water separation applications[36–38]. PLA materials have a hydrophilic structure due to the ester group, hydroxyl group and carboxyl hydrophilic groups in their structures. Therefore, it is very difficult to create a superhydrophobic surface by modifying PLA-based materials. It has been reported that a superhydrophobic PLA surface is obtained for oil/water separation by using many methods such as three-dimensional (3D) printing, layer-by-layer coating, electrospinning and phase separation[38]. However, a versatile and easy approach is still lacking, which can be seen as a systematic descriptive and technical basis for the preparation of PLA-based separation material exhibiting effective and efficient surface wettability and high mechanical stability for use in large-volume practical applications[37].

In this review, it was focused on the recent trends on NWF designed for oil/water separation applications. Within this scope, firstly, the fundamental theory of wettability is discussed then, the effects of structure control, interface fabrication, and nonwoven's self-structure on superhydrophobicity are prospected. These were followed by recent studies in literature related to nonwoven-based functionalized surfaces for oil-water separation application.

2.MODELS OF WETTING BEHAVIOR

Surfaces with SHB properties are defined by their high water contact angles (WCA> 150°)[39] and low roll-off water sliding angles (WSA < 10°)[15,24,39,40]. The amount of oil in emulsions with high water content is more effectively separated by SHB and superoleophilic (SOL) surfaces that repel water and absorb oil content. In this context, methods such as chemical vapor deposition, dip coating, so-gel, spray coating and polymer grafting are used in the development of SHB and superoleophilic (SOL) surfaces. Surfaces with a water contact angle of less than 90° or nearly 0° are called superhydrophilic (SHL) and were first reported in 1995. As explained by Young's equation, the surface tension of these surfaces is higher than that of water. However, it is very difficult to develop super hydrophilic surfaces with this property, since there is no special material that has both a surface energy higher than water and lower than oil droplets. The structure of these special surfaces was solved by examining fish scales, which have the characteristics of SHL in air and SOB in water. Since the fish have a SHL interface, they show the feature of being hydrophobic under water. A three-phase oil/solid/water system Where γ_{WA}, γ_{OA}, and γ_{OW} explain the interface tension of water-air, oil-air, and oil-water while θ_{WA}, θ_{OA}, and θ_{OW} shows the water-air, oil in the air, and oil in water contact angles in Young's equation was used to explain underwater oleophobicity (Equation (1)). The other equation (2) is to explain the oil contact angle in air, and according to this equation, it states that hydrophilic surfaces in air can behave oleophobic in water, which can improve the increase in surface roughness. According to these explanations, the main parameters of underwater oleophobic surfaces are rough surface and chemically hydrophilic composition. Fig. 4 shows the schematic representation of hydrophilic and hydrophobic surfaces[24].

$$\cos\theta_{OW} = Y_{OA}\cos\theta_{OA} - Y_{WA}\cos\theta_{WA} / Y_{OW} \tag{1}$$

$$\cos\theta_{OA} < (Y_{WA}/Y_{OA})\cos\theta_{WA} \tag{2}$$

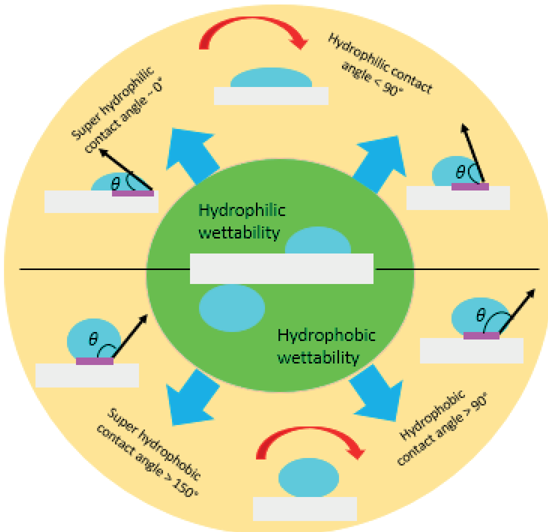


Fig. 4*: Schematic illustration of superhydrophic and superhydrophiic surfaces, adapted from Ali et al.,* [24]*.*

Surface phenomena related to the wetting behavior of solid and liquid materials in each other are explained within the scope of surface chemistry. For example, these wetting behaviors are used to effectively separate hydrophobic and oleophilic (oil-loving), hydrophilic and oleophobic (water-loving) oil/water emulsions. In emulsions rich in water content, effective separation of water can be achieved by hydrophilic/oleophobic materials. In the membrane separation process, the materials that allow the oil to pass through by blocking the water are defined as hydrophobic/oleophilic, on the other hand, the materials that block the oil and allow the water to pass through are defined as hydrophilic/oleophobic. Therefore, efficient separation of oil/water emulsions can be achieved with smart surfaces and using special materials with high wetting performance. Due to these needs, material designs and productions with super-wetting properties are important in terms of achieving the desired efficiency in oil/water separation processes[41]. Accordingly, in this section, theories of wetting and mechanisms of oil/water separation based on special wettability were given. Wettability is a property of the surface that depends on both the chemical composition of the material and the surface morphology. The internal character of the material is the chemical polar/nonpolar composition that determines the free energy of the surface. If the wetting surface is a rigid, chemically insoluble and non-reactive smooth solid surface, Young's equation (Equation (3)) is used to describe the typical wetting mechanism[19].

$$\cos\theta_Y = \frac{\gamma_{SV}-\gamma_{SL}}{\gamma_{LV}} \tag{3}$$

In this equation, surface tension or free energy determines the wetting behavior[19]. Briefly, Young's angle (θ_Y) is a result of the thermodynamic balance of free energy at the solid-liquid-vapor interface and is related to the interface energies between solid-vapor (γ_{SV}), solid-liquid (γ_{SL}) and liquid-vapor (γ_{LV}) (Fig. 5a). In the past decades, researchers have focused on understanding of the "lotus effect" with their experimental and theoretical studies on superhydrophobic surfaces. Wenzel and Cassie models proposed in the 1840s are generally used to explain the relationship between surface roughness and contact angle[27,42].

In the Wenzel model, it is stated that the increase in surface roughness increases the surface area of the solid material. In the case of Wenzel, the water forms a continuous contact with the solid surface it comes into contact with, and the contact angle changes depending on the surface roughness. Cassiee Baxter model suggested that small air pockets form at the bottom of water droplets on rough surfaces, thus providing superhydrophobicity by forming a composite surface. In this case, it can be said that hydrophilic materials can exhibit hydrophobic behavior with appropriate roughness. According to both Wenzel and Cassie models, water droplets can form high contact angles on rough surfaces, but because the adhesion properties of water droplets are different, it determines the superhydrophobic properties of the surface together with its topographic feature and chemical component. At the same time, Cassie found that the porous material structure had an effect on the hydrophobicity, which in turn increased the porosity and thus the hydrophobicity[27]. In the Wenzel model, the liquid droplet completely fills the surface as in Fig. 5b and the associated Wenzel contact angle is explained by Equation (4)[19]. The "r" factor, which is the ratio of the actual surface area to the apparent area of the rough surface, is used to describe the wettability of a solid rough material surface.

$$\cos\theta_W = r\cos\theta_Y = \tau\frac{\gamma_{SV}-\gamma_{SL}}{\gamma_{LV}} \tag{4}$$

Hydrophobicity ($\theta_Y > 90°$) or hydrophilicity ($\theta_Y < 90°$) is related to the degree of surface roughness as specified in Equation (2). Increasing the surface roughness in this way leads to wetting and non-wetting conditions of the surface. When solid surfaces are rough and porous, the Wenzel model was limited in explaining this, and the Cassie model was proposed, in which the heterogeneities of surface chemistry were explained. As shown in Fig. 5c, air pockets are assumed to form under the liquid droplet and the apparent contact angle is explained by Equation (5)[19].

$$\cos\theta_C = -1 + f_S(\cos\theta_Y + 1) = -1 + f_S\left(r\,\frac{\gamma_{SV} - \gamma_{SL}}{\gamma_{LV}} + 1\right) \quad (5)$$

The f_S and f_V specified in Equation (5) represent the solid and vapor field ($f_S + f_V = 1$). Also, the r factor, which is the degree of roughness of the solid surface areas in contact with the liquid, can be explained by giving Equation (6) to indicate the contact angle of the solid surface[19].

$$\cos\theta_C = -1 + f_S(r\cos\theta_Y + 1) = -1 + f_S\left(r\,\frac{\gamma_{SV} - \gamma_{SL}}{\gamma_{LV}} + 1\right) \quad (6)$$

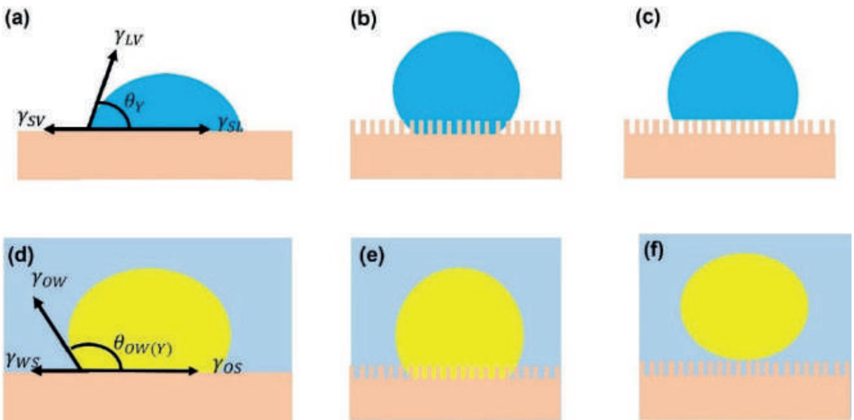


Figure 5. Schematic representation of the wetting models for water droplets (a-c) and oil droplets (d-f) on solid surfaces, adapted from Lin et al, with permission from John Wiley and Sons Publishing Group [19].

2.1. Basic theories of oil–water separation

Superwetting surfaces have been developed in different combinations in air, under water and under oil such as SHB-SHL, SOL-SOB and SOL-SOB, respectively. Surfaces showing SHB and SHL properties according to the contact behavior of water droplets on solid surfaces correspond to oil-based SOL and UWSOL properties, respectively, and surfaces with these properties were used for the design of super-wetting membranes, which are aimed to use oil-water separation. Normally a SHL surface in air shows SOB surface behavior underwater. In addition, extended wettability under water or oil depends on the micro/nano scale structure and chemical composition of the surface. In particular, the different anti-wetting properties of oil droplets on fish scales compared to water droplets led to research on UWSOB surfaces. surface[19,27].

$$\cos\theta_{OS(Y)} = \frac{\gamma_{OV}\cos\theta_{OV} - \gamma_{WV}\cos\theta_{WV}}{\gamma_{OW}} \tag{7}$$

The underwater oil contact angle ($\theta_{OW(Y)}$) in the Young's model is also related to the oil-vapor (γ_{OV}), water-vapor (γ_{WV}), and oil-water (γ_{OW}) interfaces interfacial tension or energies. θ_{OV} and θ_{WV} represent the contact angles of oil and water droplets in the air, respectively. From Equation (7) subtract UWSOB ($\theta_{OW(Y)} > 90°$ or $\cos\theta_{OW}(Y)\gamma_{OW} < 0$) in Equation (8).

$$\cos\theta_{OV} < \gamma_{WV}\cos\theta_{WV} \tag{8}$$

If rough solid or porous membrane surfaces with micro/nano-scale structural features are included in an oil-water-solid system, the modified Cassie state in Fig. 5e is obtained and this model is described by Equation (9).

$$\cos\theta_{OW(C)} = f\cos\theta_{OW(Y)} + f - 1 \tag{9}$$

Here, the underwater oil contact angle in the Cassie model is denoted by $\theta_{OW(C)}$, while f is the area fraction of the rough solid surface. Here, if the surface roughness f approaches 0, $\theta_{OW(C)}$ will be closer to 180° (superoleophobicity), which is shown in Fig. 5f [19].

2.2. Effects of nonwoven fabric structure on superhydrophobicity

In recent years, researchers have investigated the theory of superhydrophobic surfaces and carried out theoretical and experimental studies in this direction, and they have carried out the design, simulation, and fabrication of superhydrophobic nonwovens by adding superhydrophobic structures to nonwoven fabrics by chemical or physical processes. Compared to woven and knitted fabrics, non-woven fabrics are 3-dimensional structures due to their superior porosity of 90-95% and the positions of randomly placed fibers. This 3D structure not only keeps the air on the surface, but also stores the materials. Superhydrophobic fabric surfaces can be created by creating nanoscale holes and particles on the surfaces of the fibers that make up nonwoven fabrics. In this direction, researchers have carried out theoretical studies to examine the fabric structure by using different types of non-woven fabrics as base material. The structural design of these fabrics can be accomplished by introducing fiber fineness and micro-nano structure. It is known that fiber fineness affects the superhydrophobicity of nonwoven fabrics. Research results showed that fibers with smaller fiber fineness produced higher apparent contact angle and thus superhydrophobic fabrics could be produced. On the other hand, with chemical applications, in recent years, many researchers have reported micro-nano hierarchical structure on the surface of nonwoven substrate to obtain superhydrophobic nonwoven fabrics by impregnation method, spraying method, layer-layer self-assembly and other methods [27].

3. NONWOVEN-BASED FUNCTIONALIZED SURFACES FOR OIL-WATER SEPARATION APPLICATIONS

Non-woven fabrics are a kind of fabric consisting of regular or randomly arranged fibers with high porosity, excellent oil absorption performance. Superhydrophobic nonwovens find use in filtration, corrosion resistance, self-cleaning and oil/water separation systems. Non-woven fabrics have attracted attention in the development of oil/water separation filters, especially with their three-dimensional fiber structure. However, unmodified nonwoven fabrics do not show much selectivity in oil/water separation processes. Therefore, in order to improve the web/water selectivity of these fabrics, superhydrophobic structures have been placed on their surfaces and high oil/water selectivity surfaces with high repulsion against water have been developed to some extent. The structure of superhydrophobic fabrics must meet a combination of surface chemistry and surface roughness. Domestic and foreign researchers are working on combination of surface chemistry and surface roughness at the micro and/or nanoscale. The first is to replace the low surface energy material, and the other is to create a rough structure on the low surface energy surface. According to the Wenzel and Cassie Baxter model, the rougher a surface, the more effective superhydrophobic behavior. The preparation methods of these superhydrophobic fabrics include dip coating method, electrospinning method, deposition method, spraying method, sol-gel method, etc[27,42].

An overview of nonwoven-based oil/water separation application is given in Table I, and several examples of their structures are displayed in the Fig. 6-7. There are limited studies conducted so far to design nonwoven-based functional surfaces for oil/water separation applications[2,3,4,5,7,12,14–17,18–26]. Post processing of functional superhydrophobic materials developed for effective oil/water separation is problematic as they are not biodegradable and easily contribute to secondary contamination. For example, in one study, Chen et al., developed an ecofriendly biodegradable superhydrophobic PLA nonwoven fabric using a one-step spraying method. The as-prepared PLA fabric showed superoleophilic and superhydrophobic surface behavior with water contact angle of $161 \pm 2^{\circ}$ and an oil contact angle of 0° and it was obtained efficient oil separation capacity (>97.5%) as well as high oil flux ranging from 2239 $L \cdot m^{-2} \cdot h^{-1}$ to 59.713 $L \cdot m^{-2} \cdot h^{-1}$. In this study, an environmentally friendly method is proposed for the development of biodegradable separation materials[38]. In another study, Gu et al. reported a biodegradable PLA nonwoven fabric with rough and porous structure that can be used efficiently for oil/water separation (Fig. 6(I)). In this study, dopamine, a self-polymerizing biomimetic molecule in an alkaline environment, was first used as a modifying agent for the modification of PLA nonwoven fabric. Hydrophobic polystyrene (PS) microspheres and silica oxide (SiO_2) nanoparticles were deposited on the polydopamine (PDA) modified PLA NWF surface to impart superoleophilic and superhydrophobic surface behavior for a gravity driven oil−water separation (Fig.6(II)). The modified PLA NWF exhibited a permeation flux of ≈6.000−12.000 $Lm^{-2}h^{-1}$. The SiO_2/PS/PLA non-woven fabric prepared within the scope of the study both separated the oil/water mixtures efficiently and preserved its superhydrophobicity after friction and stretching tests, so it has been shown to be a suitable candidate for separation processes[37].

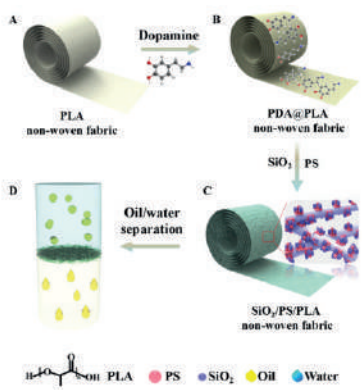
Ong et al. used the block copolymer strategy to prepare nonwoven fabric and polyurethane sponge surfaces that exhibit super-oleophilic and super-oleophobic behavior in aqueous media, and for this purpose, a block copolymer containing pH-sensitive poly (2-vinyl-pyridine) and oleophilic/hydrophobic polydimethylsiloxane (i.e., P2VP-b-PDMS) blocks is preferred. The P2VP block provides controllable and modifiable access of oil by the oleophilic PDMS block by changing the wettability and conformation through protonation and deprotonation according to changing pH values in the aqueous medium, and this results in a smart surface with varying oil wettability behavior[54]. Zhu et al. modified PLA NWFs with stereocomplex crystals using the solvent-free phase separation method (Fig. 6(III)). The modified PLA NWFs showed inlet pressure of 11.78 kPa and were found to be suitable for separation of water/oil mixtures with separation efficiencies and permeability fluxes of ≈10^4 L m^{-2} h^{-1} and >97%, respectively[43]. In another study, NWF embedded with PLA microparticles was prepared by Yuan et al. using a multi-step procedure (Fig. 6(IV)), and fabrics modified by immersion in super hydrophobic polyester Triphasic solution for water/oil separation. The prepared NWF showed high permeability flux in the range of 20,000−30,000 $Lm^{-2}h^{-1}$, a water contact angle of 153°(Fig.6(V)), >97% separation efficiency, and poor inlet pressure of 0.216 kPa. Moreover, the fluxes of different organic solvents were also tested, which were distributed in the range from 17 to 27 $m^3m^{-2}h^{-1}$ [4].

Abu-Thabit et al. presented a simple, efficient, fast and green two-step production method for preparing superhydrophobic/superoleophilic polyacrylonitrile (PAN) NWF for oil/water separation applications. In the first step, hydrophilic PAN NWF converted to the hydrophobic fabric structure by in situ deposition of iron hydroxide nanoparticles (NPs) on the fabric surface, then iron/palmitic acid micro/NPs were deposited on the surface to reduce the surface energy (Fig.6(VI)). The resulting PAN NWF showed good oil absorption capacity, high permeability flow and high separation efficiency. It also exhibited stable performance for 20 separation cycles and superhydrophobic behavior with WCA >150° and WSA <5° (Fig.6(VII))[15]. In another study, Zhang et al., developed groove-like PP/polyester (PP/PET) micro/nanofiber surface with a three-step tailored process of blending, drawing, and etching. Additionally, nonwovens including the groove-like fiber showed efficient oil wetting capability for separating oil and water mixtures and hydrophobic behavior with high water contact angles of 150.45° and a separation efficiency of 99% was obtained[26]. In another study, Yuan et al. used a thermally induced phase separation method to develop polyurethane (TPU) microspheres for use in superhydrophobic dip coating of nonwoven fabric for oil/water separation. The solution-coated nonwoven fabrics containing the microspheres showed superhydrophobicity with a water contact angle of 153°[16]. Superhydrophobic nonwoven fabric with hydrothermal and modification process was designed by Zhang et al. for oily wastewater treatment. The developed non-woven fabric provided a high separation efficiency of over 95.0%, showed effective performance after 10 cycles in strong acids and bases environments and retained its superhydrophobic properties throughout the entire pH range[44]. Zeng et al. proposed an easy and effective method for developing superhydrophobic and magnetic poly (lactic acid) (SMPLA) nonwoven fabric, consisting of three stages of preparation (Fig. 7(I)). First, polydopamine was formed on the fabric surface by dopamine polymerization, then iron oxide (Fe3O4) particles were immobilized on the polydopamine to provide surface roughness and give the fabric a magnetic feature. In the last step, coating with poly (vinylidene fluoride-co-hexafluoropropylene) was applied to reduce the fabric surface energy. A water contact angle of 151.7° and oil-water separation efficiency reaching 99.5% were obtained in the resulting SMPLA fabric99.5%[45].

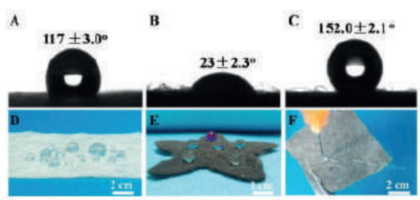
In another study, a nonwoven fabric produced from PP/low melting point polyester (LPET) fibers with hydrophilic-underwater superoleophobic behavior for oil/water separation was successfully produced by Sun et al. The fabrics were dip coated with poly(N-isopropylacrylamide) (PNIPAM) to obtain hydrophilic-underwater superoleophobic property. Underwater super oleophobicity was achieved with a high oil contact angle of 150°, while the water contact angle of the PNIPAM coated fabric was between 80° and 0° (within 8 s). Moreover, modified nonwove fabrics showed high penetration flow (~21850 $Lm^{-2}h^{-1}$) and high separation efficiency (~99%)[46].

Ahmad et al. have produced hydrophilic chitosan-graphene oxide-modified sustainable non-woven fabric-based sieve membranes (NWF@ Cs/Gx) and the performances of these fabrics have been studied in terms of protein rejection, antifouling and oil-water emulsion separation studies. The hydrophilicity of these membranes was investigated by examining water uptake studies, which shows water flow up to 165 $Lm^{-2}h^{-1}$. Separation of multigrade oil/water emulsions with fabrics produced was achieved with an efficiency of 98%, and these studies have shown that formulated membranes can find applications in the environment and petroleum-based industries[47]. A surface functionalization method for nonwoven fabrics with no selectivity of water/oil mixture reported by Xiong et al. Nonwoven fabrics membrane was produced via hydro-thermal growth of ZnO hierarchical nanorods on the ultrathin layer. The functionalized nonwoven membranes were obtained with both underwater oleophobicity and under oil hydrophobicity surface behavior. Nonwoven membranes efficiently separated water/oil mixtures and it was shown that these functionalized nonwoven membranes may find application against separation systems including complex components[48]. In another study, Brindha et al., reported the effects of needling density, blend proportion and fabric weight of nettle and polypropylene (PP) based nonwoven fabrics by using Box and Behnken experimental design to determine the performance of fabrics on diesel engine oil and crude oil sorption. From this study, it is concluded that nettle/PP (30/70) needle-ounched nonwoven fabrics can be efficiently used for separation oil/water systems[49]. Sharma et al. investigated and reported the oil-water separation of diesel soot (Ds) coated nonwoven fabrics, as well as the adsorption of dyes, detergents, and pharmaceuticals. In this context, separation efficiency of over 95% was achieved with coated nonwoven fabrics, while it was found to be hydrophobic with a contact angle of 140° (Figure 7 (II))[50].

In another study, Peng et al. developed a sandwich structured filter using non-woven fabrics for oil/water separation application. Spunbond-meltblown-spunbond (SMS) nonwoven fabric, which is coated using hydrophobic silica and fluorine resin, is used as the top layer of this structure. Citrate impregnated spunlaced viscose (VS) was used for the interface and spunbond PP nonwoven fabric was used for the lower surface. This three-layer laminated structure was strengthened in an ultrasonic sealing machine and tested for oil-water separation. After 15 recycling of the prepared laminated three-layer nonwoven structure, the water contact angle decreased from 150° to 135°. Thanks to the SMS layer of the filter structure, a connection was ensured with the water in the mixture, a water channel was created on the substrate by gravity and capillary action, and new information was obtained on oil/water separation systems with this study[51]. PP non-woven fabric/ZIF-8 composite film was prepared by Yang et al. ZIF-8 is a metal organic framework (MOFs) formed by the complexing of zinc ions with imidazolium salts. In the PP nonwoven fabric/ZIF-8 composite film, the contact angle was obtained up to 137.45° when the ZIF-8 loading amount was 0.85 g·g−1. The gasoline adsorption capacity of the resulting composite was up to 3612% by mass and retained a high oil absorption capacity even after 43 reuses. In this study, it has been shown that a good recyclable oil/water separation material can be obtained with woven fabric/ZIF-8 composite film[52]. In another study, Pakdel et al. used carbon fiber waste to produce a functional nonwoven fabric for use in oil/water separation applications. Within the scope of this study, waste carbon fibers were formed into fabrics by carding and needling nonwoven production method, and surface modification was achieved by covering them with fluorine-free coatings containing polydimethylsiloxane (PDMS) and zeolite imidazole frame-8 (ZIF-8) particles. The resulting fabric was investigated for the absorption of organic solvents/oils and oil-water separation. The results showed that PDMS application initially increased the contact angle, but excessive coating application reduced the superhydrophobicity and absorbent capacity of the fabrics. The sample coated with PDMS/ZIF-8 exhibited superhydrophobicity with a water contact angle of 153.5°. This research presented a sustainable approach to the development of carbon fiber nonwoven fabric with oil-water separation capability from waste materials[53].



(I)



(II)

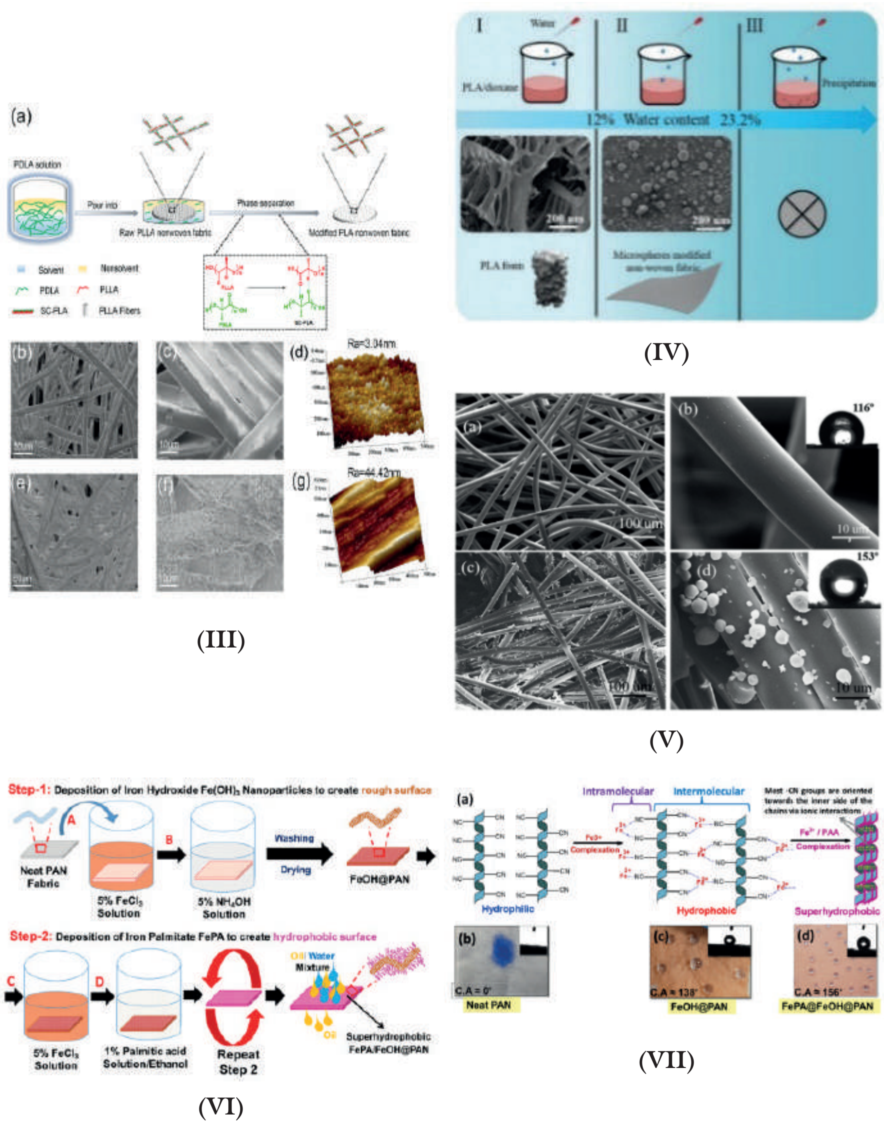
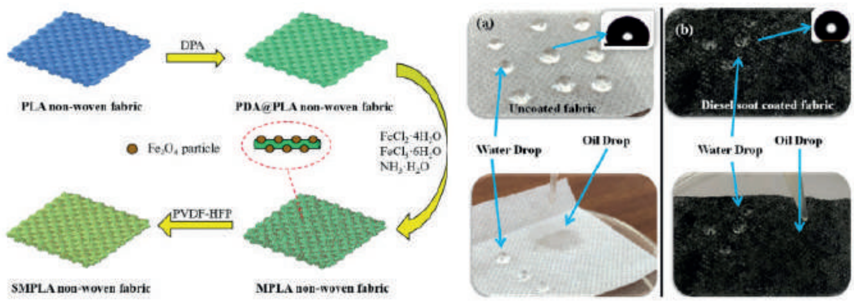


Figure 6. (I) Schematic illustration of the preparation of SiO_2/PS/PLA nonwoven fabric, (II): Water contact angles of A) PLA nonwoven fabric, (B) PDA@PLA nonwoven fabric, and (C) SiO_2/PS/PLA nonwoven fabric. (D) A PLA nonwoven fabric with water droplets on the surface. (E) SiO_2/PS/PLA nonwoven fabric with water droplets on the surface (dyed with $KMnO_4$, $CuSO_4$, and $FeCl_3$, respectively). (F) Image of a water column looks on the SiO2/PS/PLA nonwoven fabric, adapted from Gu et al., with permission from ACS Publishing Group[37]. (III): (a) Image of PLLA nonwoven fabric with the SC crystal. ESEM and AFM images of the raw and modified PLLA nonwoven fabric (b–d) and (e–g), respectively adapted from Zhu et al., with permission from ACS Publishing Group [43]. (IV): Schematic figure of PLA with different water contents, (V): SEM images of a, b) NWF and c, d) PLA/NWF adapted from Yuan et al., with permission from John Wiley and Sons Publishing Group[4]. (VI): Schematic illustration for the fabrication of NWF, (VII): (a) Schematic illustration for the re-orientation of the PAN chains; (b–d) corresponding digital images for the neat and modified PAN fabrics and water contact angles, adapted from Thabit et al., with permission from ACS Publishing Group [15].



(I) (II)

Figure 7. (I): Schematic fabrication image of SMPLA nonwoven fabric, adapted from Zeng et al., with permission from ACS Publishing Group[45], (II): Hydrophobic and oleophilic nature of (a) uncoated, and (b) DS coated non-woven fabrics, adapted from Sharma et al., with permission from Springer Nature [50]

4. CONCLUSIONS AND FUTURE PERSPECTIVES

As a result of years of studies, researchers have created advanced functional fabrics that reveal the advantages and application potentials of textile surfaces. However, in recent years, although working on simple, environmentally friendly, highly efficient textile surfaces to achieve oil/water separation, many difficulties remain in practical applications. In the last decade, studies on superhydrophobic non-woven fabrics, especially developed using biomaterials, have increased. This review examines the latest developments in the application of superhydrophobic nonwoven fabrics. The preparation of these fabrics includes either a two-stage (to make hydrophobic after pre-etching) or a single-stage coating to hydrophobic fabrics. Among the production methods, dip coating and electrospinning method attract attention in terms of being easy and common. The common difficulty is the low interface strength between the coating material and the nonwovens. To obtain effective results, adding micro-nano particles, adjusting fiber morphology, and combining fibers of different fineness in fabric structure to achieve a greater WCA can lead to positive results. The in-situ growth of nanoparticles on the fiber surface is expected to be effective in obtaining superhydrophobic nonwovens in the future. In current studies, the interfacial strength between coating materials and non-woven fabrics is increased in two ways: 1) achieving high adhesion between non-woven fabrics and coating by using bio-environmentally friendly adhesives, 2) creating strong chemical bonds or cross-linked structures between fabric and coating. These methods will change the SHB structure of the fabric surface. Apart from these, it is possible to obtain high strength superhydrophobic non-woven fabrics with the use of micro/nano fine fibers that exhibit hydrophobic behavior. In the current state of the research, polymers containing fluorine-silicone continue to be used for superhydrophobic behavior. However, these polymers are not economical and cause environmental pollution due to their toxic properties. Nowadays, inorganic nanoparticles, metal organic framework, two-dimensional carbon nanomaterials, etc. are used to modify fabric surfaces but the strength properties of these materials are low. Therefore, high performance superhydrophobic nonwoven fabrics with modified eco-friendly biomaterials will be one of the future development directions. Despite the limitations in their application, it is a fact that the development prospects of fabrics in the field of oil/water separation are bright and promising, and the trend in future work is expected to be to develop green and sustainable oil/water separation materials.

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Table 1: Nonwoven Fabrics for Oil-Water Separation Applications.

Substrate	Materials	Fabrication Technologies	CA	SA	Separation type	Separation Efficiency	Separation Flux (L m^{-2} h^{-1})	Feature	Refs.
Nonwoven Fabric	Iron palmitate complex nano/ microparticles	Via an in situ deposition	WCA: 150°	5°	Oil/water mixtures	99.9%	23,935 ±380 and 20,900±266	SHB	15
Nonwoven Fabric	PLA micro-particles-triphasic solution	Chemical bath deposition	WCA: 153°		Oil/water mixtures	97%	20,000–30,000	SHB	4
Nonwoven Fabric	(PP/PET) micro/ nanofiber	A three-step tailored process	WCA: 150.45°	-	Oil/water mixtures	99%	-	SHB	26
Nonwoven Fabric	poly(2-vinylpyridine) and polydimethylsiloxane	Block copolymer grafting	WCA: 165.3°	-	Oil/water mixtures	-	-	SOL and SOB	35
Nonwoven Fabric	PS and SiO_2 nanoparticles	Chemical bath deposition	WCA: 152°	-	Oil/water mixtures	-	≈6000–12.000	SHB	37
Nonwoven Fabric	PLA particles and MWCNTs perfluorinated silane	One-step spraying method	WCA: 161 ± 2°	-	Oil/water mixtures	97.5%	2239-59.713	SHB/SOL	38
Nonwoven Fabric	Stereocomplex crystals	Non-solvent-induced phase separation	WCA: 130.8°	-	Oil/water mixtures	97%	≈10000	SHB/SOL	43
Nonwoven Fabric	Zinc acetate	Facile hydrothermal and modification process	WCA: 156°		Oil/water mixtures	95%	-	SHB	44
Nonwoven Fabric	polydopamine (PDA), iron oxide (Fe3O4) particles, poly (vinylidene fluoride-co-hexafluoropropylen	In situ polymerization	WCA: 151.7°	-	Oil/water mixtures	99.5%	-	SHB	45

Nonwoven Fabric	poly(N-isopropylacrylamide) (PNIPAM)	Dip Coating	UOW: 150°	-	Oil/water mixtures	99%	≈21850	SHL/ UWSOB	46
Nonwoven Fabric based sieve membranes (NWF@Cs/Gx)	Chitosan-graphene oxide	Ultrasonication casting and phase inversion techniques	WCA: 30.31°	-	Oil/water emulsions	98%	≈ 165	SHL	47
Nonwoven Fabric	ZnO	Atomic layer deposition	WCA: 110°	-	Oil/water mixtures	97%	3300-6900	UWSOB/ UOSHB	48
Nonwoven Fabric	diesel soot (Ds)	Coating	WCA: 140°	-	Oil/water mixtures	95%	-	SHB	50
Nonwoven Fabric	silica and fluorine resin	Lamination	WCA: 150°	-	Oil/water mixtures		1465	SHB/SHL	51
Nonwoven Fabric	zeolite imid- azole framework	In situ grown method	WCA: 137.45°	-	Oil/water mixtures	-	-	SHB	52
Nonwoven Fabric	polydimethylsiloxane (PDMS) and zeolite imid- azole framework-8	Coating	WCA: 153.5°	--	Oil/water mixtures	97%	-	SHB	53
Nonwoven Fabric	Zeolitic imidazolate frameworks, polylactic acid	In situ growth, spraying	WCA: 135°	20°	Organic solvents/water mixtures	99%	-	SHB	55
Nonwoven Fabric	Dopamine, polyethylenimine	Chemical bath deposition	UOW: 165°	UOW: 2.5°	Oil/water mixtures	99.2%	115000	SHL/ UWSOB, anti-corosion	18
Nonwoven Fabric	N-Methyl pyrrolidone, formic acid, polyethylene glycol	Water vapour induced phase inversion	UOW: 160.7°	-	Oil/water emulsions	99.9%	478 000	SHL/ UWSOB	56

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