



Historical perspective

Selective separation of oil and water with mesh membranes by capillarity

Yuanlie Yu ^a, Hua Chen ^b, Yun Liu ^c, Vincent S.J. Craig ^{d,*}, Zhiping Lai ^{a,*}^a Advanced Membrane and Porous Materials Center, Division of Physical Science and Engineering, King Abdullah University of Science and Technology, Thuwal 23955-6900, Saudi Arabia^b Centre for Advanced Microscopy, Australian National University, Canberra, ACT 0200, Australia^c Research School of Chemistry, Australian National University, Canberra, ACT 0200, Australia^d Department of Applied Mathematics, Research School of Physics and Engineering, Australian National University, Canberra, ACT 0200, Australia

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ABSTRACT

The separation of oil and water from wastewater generated in the oil-production industries, as well as in frequent oil spillage events, is important in mitigating severe environmental and ecological damage. Additionally, a wide range of industrial processes require oils or fats to be removed from aqueous systems. The immiscibility of oil and water allows for the wettability of solid surfaces to be engineered to achieve the separation of oil and water through capillarity. Mesh membranes with extreme, selective wettability can efficiently remove oil or water from oil/water mixtures through a simple filtration process using gravity. A wide range of different types of mesh membranes have been successfully rendered with extreme wettability and applied to oil/water separation in the laboratory. These mesh materials have typically shown good durability, stability as well as reusability, which makes them promising candidates for an ever widening range of practical applications.

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1. Introduction

1.1. Need for oil/water separation

The separation of oil and water from oil/water mixtures is an ancient challenge that is becoming more important [1–3]. Discharged oil can

cause severe environmental and ecological damage directly and because the oil is flammable and can decompose to form other harmful chemicals, further polluting the natural environment and threatening aqueous habitats and affecting human health [2]. Furthermore, chemicals used in clean-up operations can cause environmental harm. Apart from these environmental and ecological issues, a wide range of industrial processes require the separation of oils or fats from aqueous solutions and the scarcity of water in many arid regions adds to the demand for efficient oil/water separation technologies [4]. Another important consideration in choosing the technology used to effect

* Corresponding authors.

E-mail addresses: vince.craig@anu.edu.au (V.S.J. Craig), zhiping.lai@kaust.edu.sa (Z. Lai).

separation is the possible reuse of the recovered oil. Although traditional methods such as skimmers, centrifuges, coalesces, settling tanks, depth filters, magnetic separations, flotation technologies, and ignition of oil can remove oil from oily wastewater, these techniques are mainly useful for oil water mixtures and unstable emulsified oils, as their working mechanisms are sensitive to the oil droplet size and/or the density difference between water and oil [5–7]. Moreover, because they require long processing times or gas or chemical input, these conventional separation systems usually are expensive and require a large amount of space and energy [5,6]. More stringent environmental controls and the pressure of economic development have led to a strong desire to produce more efficient, cost-effective processes for oil and water separation [8].

Utilizing engineered wetting of solid surfaces to design an oil/water separation process based on capillary processes has been recognized as an effective approach [9–11]. For practical applications it is necessary to preferentially maintain one phase at the surface in addition to preventing fouling of the surfaces, therefore materials with extreme surface wettability of two types are employed. These are solids with surfaces that are superhydrophobic and superoleophilic or surfaces with the inverse wettability that are superoleophobic and superhydrophilic. Such surfaces when exposed to mixtures of oil and water preferentially attract one of the phases in order to reduce the overall interfacial energy of the system.

1.2. Weak assembly

From a molecular viewpoint the interfacial energy arises due to the loss of cohesion energy when a molecule is moved from the bulk to the interface. For liquid oil, we can approximate the cohesive energy of a molecule to the translational kinetic energy. Assuming half the cohesive interactions are lost for a molecule at the interface, the molecular energy shortfall at the interface is $\sim 3 K_B T/4$ for an air interface. As such, wetting is an example of weak assembly, where the lowering in interaction energy for an oil molecule moving from the liquid–liquid interface to the liquid–solid interface is $\sim K_B T$, whereas the lowering in interaction energy for a water molecule moving to a hydrophilic surface is $> K_B T$, due to both dispersion and polar interactions and is therefore an example of strong assembly. It is the difference in these energies that is used to effect oil/water separation using mesh membranes. The self-structuring of fluids at the interface can be devised to affect macroscopic separation of the fluids, provided the external forces are insufficient to overcome the capillary force on one phase, but not the other. For separating oil–water mixtures under gravity, a membrane that is superhydrophilic and superoleophobic (when submerged) will allow water to pass but provide a capillary barrier to the passage of oil, provided the hydrostatic head is insufficient to overcome the effect of the Laplace pressure that arises from the weak assembly of the oil molecules. A membrane of the other type (superhydrophobic and superoleophilic) will allow oil to pass and prevent the passage of water.

1.3. Theory of superhydrophobicity and superhydrophilicity

The wettability of a surface is usually quantified by the contact angle of a droplet of liquid on the surface. The droplet can either be in air or in another immiscible fluid. For smooth, homogenous, ideal surfaces the contact angle of a droplet on a surface immersed in another liquid is described by the balance of the interfacial energies in Young's equation;

$$\gamma_{s,l} = \gamma_{s,d} + \gamma_{d,l} \cos \theta_{smooth} \quad (1)$$

where s , is the solid and d is a liquid droplet immersed in a liquid l .

Surfaces are often described as hydrophilic or hydrophobic based on their contact angle with water in air on a smooth surface. There is no widely accepted definition, but a common one is that a hydrophobic surface has a contact angle of $>90^\circ$ and consequently a hydrophilic

surface has a contact angle $<90^\circ$. When the surfaces are rough more extreme wetting can be observed. Superhydrophilic surfaces arise when an innately hydrophilic surface is produced in a roughened form (such as from sintered particles) such that the contact angle is reduced to zero. The effect of roughness on the contact angle for a homogenous surface is described by the Wenzel equation, [12]

$$r = \frac{\cos \theta_{rough}}{\cos \theta_{smooth}} \quad (2)$$

where r , is the roughness parameter ($r > 1$), which is the ratio of the projected surface area of the rough surface to a perfectly flat surface. The contact angle of surfaces with oil is less often reported, as oils have low surface tension and therefore wet most flat surfaces. That is, surfaces are typically oleophilic. Therefore a superhydrophilic surface will typically also be a superoleophilic surface. However, when a superhydrophilic surface is first immersed in water it can be both superhydrophilic and (under water) superoleophobic as the water may fill the texture of the surface. This is discussed further below.

Superhydrophobic surfaces can be produced from rough surfaces that are inherently hydrophobic. For a homogenous surface the Wenzel equation (Eq. (2)) also applies, but as $\theta_{smooth} > 90^\circ$, $\theta_{rough} > \theta_{smooth}$. Additionally, gas can be trapped between the solid and the liquid phases forming a heterogeneous composite interface, in which case the contact angle is given by a modified version of the Cassie equation, [13]

$$\cos \theta_{Cassie} = f_1 [\cos \theta_{smooth} + 1] - 1 \quad (3)$$

where f_1 is the fractional area of the surface (or three phase line) in contact with the liquid. For surfaces in which $\theta_{smooth} > 90^\circ$, high effective contact angles result and as f_1 becomes smaller the contact angle approaches 180° . Superhydrophobic surfaces are usually also superoleophilic. Note that as oils do not give surfaces in which $\theta_{smooth} > 90^\circ$ this approach will not work for producing superoleophobic surfaces. However, this can be achieved using surfaces engineered to have a re-entrant structure [14,15].

In membrane separation processes it is important to recognize that air is not present at the interface, that is, both of the fluid phases are liquids, and the desired properties are obtained when the wetting liquid penetrates the surface roughness, as air does in the Cassie state described above. This causes the non-wetting liquid to present with a very high (super) contact angle, as described by Eq. (4) where f_{NW} is the fractional area of the surface (or three phase line) in contact with the non-wetting liquid and $\theta_{NW,smooth}$ is the contact angle of the non-wetting fluid in the presence of the wetting fluid on a solid substrate.

$$\cos \theta_{NW,Cassie} = f_{NW} [\cos \theta_{NW,smooth} + 1] - 1 \quad (4)$$

This heterogeneous wetting state is favored by very rough surfaces. The condition under which this occurs is given by [16];

$$\cos \theta_{W,smooth} > \frac{1-f_W}{r-f_W} \quad (5)$$

where f_W is the fractional area of the solid surface in contact with the wetting fluid. Thus a critical angle is defined such that when $s\theta_{W,smooth} < \theta_C$, the wetting film will impregnate the textured or rough surface such that the non-wetting fluid has a very large contact angle. This is the preferred circumstance for mesh membranes, whether the wetting phase is the aqueous or oil phase as it results in a very high contact angle for the non-wetting phase. For an in-depth discussion of wetting the interested reader is referred to the excellent monograph [16].

Membranes for oil/water separation are readily produced by treating a weave or a mesh to produce rough surfaces with the desired wetting properties. These mesh membranes can effectively separate immiscible oil/water mixtures and oil-in-water or water-in-oil emulsions,

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