



# Progress toward reversible electrowetting on geometrically patterned superhydrophobic surfaces

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## ABSTRACT

By combining the fascinating wetting properties of geometrically patterned surfaces with the versatility of electrowetting in modifying the apparent material wettability, novel systems with significant technological applications can arise. Applications, however, are limited by irreversible transitions between highly mobile and sticky wetting states. We review recent research efforts aiming either to design surfaces or to design actuation mechanisms to achieve an extended range of electrowetting-induced apparent wettability modification.

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## 1. Introduction

Functional surfaces with addressable wettability, dynamically responding to external stimuli, have attracted strong scientific and technological interest due to their potential use in applications involving liquid handling without moving mechanical parts. Phenomena observed first in nature, from the self-cleaning of the lotus leaf in rainy days [1] or the ability of insects to walk/jump on liquid surfaces [2,3], to the collection of drinking water by the beetle in the desert [4,5], continuously inspire scientists toward developing new materials and devices. An ideal stimuli responsive surface should perform a wide range of wettability modification in a reversible and fast switchable manner. Geometric patterning or surface roughening amplifies the hydrophobicity/hydrophilicity of an inherently hydrophobic/hydrophilic material [6] giving rise to surfaces with extreme water- or oleo-repellency. One of the most versatile, and simple to implement, method to actively control the apparent wettability of dielectric solids by conducting liquids is electrowetting on dielectric (EWOD) [7]. We term ‘apparent wettability modification’ since electrowetting does not alter the microscopic contact angle (usually called Young’s contact angle) but a macroscopic one [8]. However, from now on, aiming to avoid to repeatedly mention the terms ‘apparent contact angle’ or ‘apparent wettability’, we simply mention “contact angle” or ‘wettability’, respectively. When a reference to the material or microscopic angle is required, we term it ‘Young’s contact angle’.

Electrowetting is used to enhance the wettability of smooth solid surfaces in a fast, switchable and reversible manner, by simply applying a voltage. The upper limit of the contact angle (CA) is set by the material chemistry ( $\sim 120^\circ$  for Teflon like surfaces), whereas the lower limit is set by the well-known contact angle saturation [9–13]. The latter limit is

around  $60^\circ$  [14]. Thus, the combination of roughness and electrowetting is rendered as an ideal recipe for switchable wettability. However, wettability modification on geometrically structured surfaces is usually accompanied by irreversible wetting transitions and this is due to the multiplicity of possible wetting states. Nonsticking and highly slippery Cassie states (commonly also called Fakir, or Pearl, suspended, or Cassie-Baxter, or CB states), where air pockets are trapped underneath the liquid, can coexist (in the sense that the solid geometry can accommodate both states) with sticking Wenzel states where the liquid has collapsed by penetrating the surface patterning. Electrowetting-induced Cassie to Wenzel transitions are easily realizable even at surfaces with very robust superhydrophobicity [15]. The spontaneous reverse transitions, however, have not been observed, when applied voltage is removed i.e. once the Cassie – Wenzel transition sets in, a liquid droplet remains pinned and stucked to the solid surface, even if the Cassie state is of lower surface energy than the Wenzel one [16,17]. It is clear that understanding the mechanisms of the aforementioned wetting transitions, and in particular their dependence on several geometric or electrical parameters, is of crucial importance when aiming to make the transitions reversible or prevent them from happening. The full picture of the wetting states’ space, even for a 2-d striped solid surface, is fairly complicated, as shown by the thorough computational analysis of Kavousanakis et al. [18]; it is manifested by the plethora of possible apparent wetting behaviors, in terms of contact angle and adhesion between the liquid and solid surface [18–22], for a given and fixed material wettability. Material wettability is the key property, quantified by the Young contact angle (CA), which governs the energetics of the possible wetting states. Briefly, Kavousanakis et al. [18] found wettability regimes where only Cassie, or only Wenzel can be accommodated by structured surfaces. In addition, they found regimes where all types of states, i.e. Cassie, Wenzel or even mixed, called also partially impregnated, can satisfy the governing equations and therefore they can be

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observed in experiments. Computing their relative stability, they reported that a transition from Wenzel to Cassie states could be spontaneous if the Young contact angle is very high i.e., Young's CA > 130° (depending, though, on the stripe cross sectional shape) [18"]. Recovering a Cassie type state, however, does not mean that fully reversible wettability modification is achieved. Complete de-wetting, i.e. recovering a high apparent CA (>150°), as computations showed, required even higher Young's CA. Interestingly enough, the required high CAs were found to be independent on the stripe cross sectional shape [18"]. Since the most hydrophobic materials (silanized or fluorinated materials) do not exceed 120 degrees [23], the requirement for very high Young CA, and concomitantly for reversible Wenzel to Cassie transitions, could only be achieved effectively, via roughening at the nano scale [19]. If the material wettability (effective or intrinsic) is not low enough, transitions between Wenzel and Cassie states require surpassing considerable energy barriers that separate the states [18",24–26]. This practically means that extra energy has to be supplied in order to recover a Cassie type state.

It is, therefore, important to clarify when we refer to 'reversible electrowetting' whether spontaneous wetting and de-wetting is performed or if extra energy is required to reverse an electrowetting-induced Cassie to Wenzel transition. The latter case could be more accurately described as wettability switching rather than reversible wetting modification. In what follows, we review and comment on the most promising alternatives for performing electrowetting in a reversible or switchable manner on geometrically patterned surfaces, when the surrounding medium is air.

## 2. Switchable wetting modification - recovering Cassie-Baxter states

When a voltage is applied on a conducting droplet, sessile on a geometrically patterned superhydrophobic dielectric surface, the liquid spreads on the solid substrate. Upon exceeding a threshold voltage, the droplet penetrates the pattern features and the Cassie to Wenzel transition sets in. Then, the droplet remains at the collapsed state even if the voltage is removed [27]. The droplet does not return spontaneously to its original suspended state even if this state is energetically preferable, i.e. of lower surface energy.

It is clear that in order to overcome the energy barriers that separate distinct wetting states corresponding to local energy minima, a suitable actuation methodology for energy supply is required. Ideally, this methodology should feature fast actuation, high energy efficiency, addressability in space and in time and biocompatibility in case of application in biological systems. The advantageous ability of electrowetting to modify wettability in a fast and switchable manner renders the speed of actuation for reversing the transitions as of primary importance. Another important aspect is the energy efficiency in terms of appropriately delivering energy for de-wetting. Changing the chemistry of the solid/liquid interface either directly using chemicals (e.g. changing the pH) [28–30"] or indirectly using UV radiation [31–34] is an alternative; however those techniques lack speed and versatility. Usually the droplet has to be removed before the change of surface wettability is performed.

For a given surface chemistry and roughness, the forced triple line movement toward droplet retraction is the key actuation. This can be accomplished through several mechanisms including: a) the gas-pressure induced elimination of the liquid/solid interface and the concomitant droplet retraction by surface tension or b) by droplet vibration. What is important here is to efficiently deliver the required mechanical energy, i.e. to deliver the minimum energy to surpass the energy barriers for the reverse transition.

### 2.1. Gas-pressure induced de-wetting

The first successful Wenzel to Cassie transition has been achieved by Krupenkin et al. [16"]. Rapid heating of the solid substrate surface by a short electric current pulse (duration of 10–50 ms) is so intense that

instantly (~50 ms) increases the surface temperature up to 200 °C. Then a thin layer of vapor is created at the solid liquid interface that pushes the entire droplet to move upwards and to disconnect from the solid surface (see Fig. 1); the droplet, then, lands in the rolling ball Cassie state. The approach is purely electric in the sense that only electric signals are involved for wetting (i.e. electrowetting) and de-wetting (i.e. resistive heating). It requires, however, significant power consumption for accomplishing the intense heating of the substrate. For an electric pulse of 30 ms and a surface energy density of 15 J/cm<sup>2</sup>, required to achieve the reverse transition, the estimated power density is 450 W/cm<sup>2</sup>. This significantly high power is spent: a) on increasing the temperature of the substrate, b), on increasing the temperature of the liquid droplet, c), on the latent heat of liquid evaporation, d), the gas expansion that pushes the droplet up, e), the work of adhesion to detach from substrate and f), the kinetic energy of the jumping droplet. From the above one can see that only a small part of the supplied energy is practically used to de-wet the droplet.

The high latent heat of evaporation of water is probably the main factor (assisted by the high water heat capacity) that prevents the increase of the liquid temperature to rise above 60 °C [16"] since evaporation cools down the remaining liquid. As the authors claim, the estimated liquid loss due to evaporation in each de-wetting transition is about 0.5 μL (this loss excludes the loss of liquid after landing on the hot solid surface). The temperature of the drop, however, rises up to 100 °C, when it touches down the solid surface which is still hot. The authors do not mention whether this temperature rise, except from enhanced evaporation, can additionally induce a collapse transition due to the thermocapillary effect. Substrate cooling by optimizing its thermal conductivity, or the use of a suitable heat sink could eventually help, as the authors suggest, however one has to consider that if the heat energy is rapidly dissipated from the substrate, then it could difficultly heated up during the current pulse, thus requiring higher energy.

Evaporation induced jumping of a droplet on a superhydrophobic surface has been recently presented by Schutzius et al. [35], where the evaporation is not induced by temperature rise rather by low-pressure environment. A combination of low environmental pressure with a moderate temperature increase could be an alternative in order to make the reverse transition at low temperatures, friendly for sensitive bio-substances.

Other options of creating back pressure and induce Wenzel to Cassie transition include the direct application of gas pressure through the pores of a porous surface [36"] or gas generation by electrolysis [37]. Those techniques are far more energy efficient as compared to the one of thermal gas generation. However pneumatic actuation requires the use of closed surface structures which unavoidably increase the solid liquid contact area, even in the Cassie state, thus the contact angle hysteresis increases as well, unless gas is continuously supplied through the pores of the surface [36"] in order to make the droplets float on an air cushion. The solid substrate thickness, also, is a parameter that strongly affects the efficiency of such systems since airflow through a thick solid could be hard to achieve in a switchable manner. Also electrolysis gas generation is not limited to closed structures; however the restoration of the superhydrophobic state requires times of tens of seconds [37]. Such backpressure systems are not yet demonstrated/paired with electrowetting actuation and the combined implementation might be quite complicated.

In order to achieve addressability in space i.e. in order to utilize the above techniques for open droplet microfluidics one might consider to use micro-heaters for thermal vapor generation or microchannels for pneumatic actuation. Electrolysis gas generation seems to be more convenient alternative here, despite the relatively slow response times.

### 2.2. Interfacial oscillations induced de-wetting

The droplet oscillations have been realized either by a direct vertical vibration [38"] of the substrate or by electrowetting actuation [39"].

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