Is the knowledge of the surface topology and contact angles enough to define the drop impact outcome?

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ABSTRACT: It is well known that a superhydrophobic surface may not be able to repel impacting droplets due to the so-called Cassie-to-Wenzel transition. It has been proven that a critical value of the receding contact angle (θₙ) exists for the complete rebound of water, recently experimentally measured to be 100° for a large range of impact velocities. On the contrary, in the present work, no rebound was observed when low surface tension liquids such as hexadecane (σ = 27.5 mN/m at 25°C) are concerned, even for very low impact velocities and very high values of θₙ and low contact angle hysteresis. Therefore, the critical threshold of θₙ = 100° does not sound acceptable for all liquids and for all the hydrophobic surfaces. For the same Weber numbers a Cassie-to-Wenzel state transition occurs after the impact due to the easier penetration of low surface tension fluids in the surface structure. Hence a criterion for drop rebound of low surface tension liquids must consider not only the contact angle values with surfaces, but also their surface tension and viscosity. This suggests that, even if it is possible to produce surfaces with an enhanced static repellence against oils and organics, generally the realization of synthetic materials with remaining self-cleaning and anti-sticking abilities in dynamic phenomena, such as spray impact for example, still remains an unsolved task. Moreover, it is demonstrated that also the chemistry of the surface and the physico-chemical interactions with the liquid drops and the possible wettability gradient of the surface asperity play an important role in determining the critical Weber number above which impalement occurs. Therefore the classical numerical simulations of drop impacts onto dry surfaces are definitively not able to capture the final outcomes of the impact for all the possible fluids, if the surface topology and chemistry and/or the wettability gradient in the surface structure are not properly reflected.

INTRODUCTION

The impact of drops onto dry solid surfaces is a phenomenon involved in many industrial applications, i.e. spray cooling, ink-jet printing, spray painting, fuel injection, raindrop erosion, etc. The investigation of the single drop impact is the first step toward the understanding and the control of the liquid–solid interactions in complex phenomena where the capillarity, the viscous forces and the impact momentum play a key role. In the last decade, notwithstanding the open issue of their durability, superhydrophobic surfaces (SHS) have attracted an increasing interest in both the academic and industrial sectors, due to their remarkable self-cleaning and anti-sticking properties, involving a wide range of potential applications: biomedical, microfluidic, corrosion resistance, drag reduction, anti-icing, contamination and oxidation protection, etc. SHS are highly hydrophobic surfaces on which liquid water adhesion is very low, therefore resulting extremely difficult to be wetted. Superhydrophobicity causes the water droplet to bead up on the surface instead of spreading on it. In addition, capillary adhesion forces are low and water drops typically preserve a high mobility on the surface. The wetting behavior is evaluated by the contact angle occurring when a liquid/vapor interface meets the solid surface. In this work, advancing (θₐ or ACA) and receding (θₙ or RCA) contact angles were measured, respectively, by expanding and contracting water and hexadecane sessile drops on a horizontal surface (sessile drop method). In static conditions, θₐ and θₙ are, respectively, the maximum and the minimum stable angles. Both of them provide the range of contact angles at equilibrium, while their difference Δθ = θₐ - θₙ - named as contact angle hysteresis (CAH) - provides an indication of the drop mobility (the lowest the Δθ value, the highest the drop mobility). As reported in the literature, the
standard conditions are θ > 90° and Δθ < 10° for superhy-
drophobicity, θ > 90° for hydrophobicity and θ < 90° for
hydrophilicity. A surface able to repel liquids with a dif-
ferent polarity and physical properties (i.e. surface ten-
sion) is called amphiphilic or lyophobic[16-20], with both
terms being usually related to the liquids molecules and
not to the surfaces. Such particular non-wetting surfaces
can provide additional performances with respect to the
hydrophobic ones in many sectors. Similarly, superam-
phiphobic surfaces exhibit even larger values of static
contact angles and CAH lower than 5°. Different ap-
proaches can be used to achieve these surface properties.
For recent trends in the development, fabrication, and
characterization of superamphiphobic surfaces, the pa-
ers of Cavalli and Mugele[26], Chua and Seeger[27] give use-
ful insights. Throughout the impact on such surfaces, the
drop mobility is related to a sort of “dynamic superam-
phiphobicity”, defined as the critical wetting value under
which an impacting drop with a given impact velocity
does not wet the surface, i.e. a complete drop rebound is
observed. In this case the impact velocity, the interfacial
tension, the viscosity of the liquid, the chemical and phys-
ical properties of the surface determine the drop outcome
as well as the spreading and the retraction dynamics.
Nonetheless, a few papers[22-23] have shown how superam-
phiphobic surfaces might not necessarily lead to a total
rebound of impinging water drops, especially above a giv-
en value of Weber number (We = ρv²D₀/σ), where ρ is the
density of the fluid [kg/m³], v is its impact velocity [m/s],
D₀ is the droplet diameter [m] and σ is the surface tension
[N/m]). Eventually impalement can occur, leading to the
droplet deposition on the surface. In this sense a surface
only hydrophobic, but smoother or with a proper topolo-
gy can be more effective in repelling liquids even for a
large value of We. Recently Antonini et al.[21] analyzed the
drop impact on horizontal surfaces and highlighted the
role of the θR on the drop rebound. Accordingly, the re-
bound can be observed only on surfaces presenting θR
greater than 100° with the rebound time decreasing as θR
increases. However, Antonini et al. only refer to water as
impacting liquid[21].
To better understand the existence of a general criteri-
on to predict the drop impact outcome, and the key para-
ters governing the drop-surface interaction, the norm-
the impact of both water and hexadecane drops on solid
surfaces with different wettability was observed using a
high-speed camera. The wetting behavior of sandblasted
aluminum foils before (TQ sample) and after the deposi-
tion of: i) organic-inorganic hybrid coatings (S samples),
ii) infused hybrid coatings (SI samples), iii) grafting fatty
acid treatments (LAU) and iv) grafting FAS (FAS) has
been analyzed. The present study aims at establishing the
relationship between drop impact outcomes and the dif-
ferent parameters for both the liquid drop (impact veloci-
ty, surface tension, viscosity) and the solid surface (mor-
phology and roughness, chemistry, wettability).

**EXPERIMENTAL SECTION**

**Surface fabrication** – Four kinds of SHS have been
pre pared: S, SI, LAU and FAS. Moreover, the uncoated sand-
blasted aluminum surface taken as reference is named
TQ. S samples were prepared by dip-coating aluminum
foils in an aqueous alumina sol with average particle size
of about 30 nm. After thermal treatments, also including
boiling in deionized water, a nanostructured inorganic
coating was obtained, which was then further functional-
ized by dipping in a commercial solution of fluoroalkyl-
silane in isopropanol (Dynasylan® SIVO CLEAR EC,
Evonik), resulting into a hybrid organic-inorganic coating.
More details on the fabrication of S samples were report-
ed by Raimondo et al. in a previous work[24]. SI samples
were obtained by the same route, with an additional step
of immersion into a fluorinated lubricant (Fluorinert™
FC-43. 3M™) allowing for the formation of a continuous
liquid film on the material surface with the aim of chang-
ing the physical nature of the interface (from the solid-
liquid-air of S samples to the liquid-liquid-air of SI ones,
according to the so-called SLIPS approach[25-26]). Fatty acid
surfaces (LAU) were fabricated on an aluminum sub-
strate, previously etched in acid solution, to achieve the
desired surface roughness, and subsequently grafted in an
ethanol lauric acid solution. FAS surfaces were fabricated
by etching the surface in acid solution as for the LAU sur-
face, and then functionalized in a water solution of per-
fluorocetyltriethoxysilane (FAS)[27].

**Surface characterization** – The average values of θA, θR
and CAH with both water and hexadecane are reported in
Figure 1. The contact angle θ was calculated automatically
from drop images by measuring the tangent of a circle fit-
ting the drop profile at the contact point (OCA 15, Data
Physics Instr.). Typical drop volume was about 2 µl.
Surfaces have also been characterized in terms of topog-
raphy and roughness. SEM images were obtained with a
tescan MIRA3 equipment, while roughness measure-
ments were performed with a ContourGT-K (Bruker Nano
GmbH) optical profilometer (vertical resolution < 0.01
nm, lateral resolution 0.38 µm). SEM images of S and SI
surfaces (Figure 2) show a flower-like nanostructure made
up of crossed, 200 nm long flakes and nanometric cavi-
ties. Previous results[28] proved that such structure is due to
the γ-alumina coating obtained by deposition of Al₂O₃
nanoparticles and to the subsequent boiling in water.
LAU and FAS samples display a terrace-like structure with
sub-micrometric edges, as expected from the similar etch-
ing conditions held for both samples. On the sandblasted
TQ surface taken as a reference, microabrasion by sand
grains produced an irregular microstructure with asperi-
ties and cavities. Roughness data (S₀, S₁, S and S₂)[28] are
shown in Figure 3. $S_a$ defines the average of the absolute values of the profile heights $Z(x,y)$ in the measured area, while $S_q$ is the root mean squared of $Z(x,y)$ in the measured area. $S_v$ expresses the maximum value of valley depth on the surface within the measured area. $S_z$ expresses the maximum value of peak height and the maximum value of valley depth on the surface within the measured area. A 15x15 mm$^2$ area was analyzed for every measure, and a minimum of three measurements were done on the same sample for repeatability reason. The difference between coated (S, SI) and etched (LAU, FAS) samples can be remarked: the former have lower average roughness ($S_a, S_q$) but higher peaks ($S_v, S_z$). A comparison with the data obtained for a TQ surface led us to conclude that the hybrid nanostructured coating has a small influence on the micrometric roughness of S and SI, while the main contribution belongs to the microstructure provided by sandblasting. On LAU and FAS surfaces, etching provided a rougher structure, but with less pronounced asperities and cavities.

![Water Contact Angles](image1.png)

**Figure 1.** Average advancing (ACA) and receding contact angles (RCA) and contact angle hysteresis (CAH) with water (left) and hexadecane (right).

![Hexadecane Contact Angles](image2.png)

**Figure 2.** SEM images of the sample surfaces: as received sandblasted (TQ), hybrid organic-inorganic coating (S), hybrid plus infusion coating (SI), grafting fatty acid treatment (LAU) and grafting FAS treatment (FAS).
Drop impact experiments - A typical experimental apparatus for drop impact studies was used: a drop was generated at the tip of a hydrophobic needle, and then was accelerated by gravity and impacted onto a dry, solid surface. Experimental conditions were the following: impact speed in the 0.05 < V < 4.2 m/s range, drop diameter in the 1.5 < D < 2.6 mm range, Weber numbers in the 0.0023 < Oh < 0.0186 range, where μ is the liquid viscosity [Pa s] in the 635 range, and Ohnesorge number (Oh = μ/(ρσD)^1/2), where μ is the liquid viscosity [Pa s]) in the 0.0023 < Oh < 0.0186 range. Images of drop impacts were recorded using a high-speed camera (PCO 1200-HS) with typical frame rates of 1568 and 2477 fps and a pixel resolution of 31 μm/pixel. Tests were repeated at least 10 times for each condition to assess experimental reproducibility. Images were manually analyzed to identify the drop impact outcome and eventually to measure the time of the drop rebound when occurring.

RESULTS AND DISCUSSION

In Table 1 and in Figure 4 the summary of all the outcomes of the drop impact tests is reported. Five main regimes stand out: complete rebound, partial rebound, prompt splash, receding breakup and deposition. All the functionalized surfaces are able to generate a complete or partial rebound of water drops, while TQ surface promotes drop deposition for We up to about 200 and receding break-up for higher Weber numbers. Remarkably, surfaces S and SI are able to repel the water drops up to the maximum We value of 620. No splash is observed for the entire range of We, confirming the previous literature results. In the case of hexadecane, no rebound was observed for any of the surfaces, even if the contact angles and the hysteresis reached, respectively, very high and very low values (i.e. FAS surface, see Figure 1). The prompt splash appears to be an important outcome for We > 200. FAS sample shows a peculiar behavior, since it produces a partial rebound with water even for moderate values of We, while with hexadecane it is able to allow the generation of a singular jet for very low We, and allows the receding breakup behavior for the higher analyzed We.

A schematic representation of the drop impact test outcome is reported in Figure 4.

Table 1. Summary of the outcomes of the drop impact tests. R = rebound, PR = partial rebound, PS = prompt splash, RB = receding breakup, D = deposition. *Singular jet. ** Transition region.

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<td>D/PR**</td>
<td>RB</td>
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Figure 4. Schematic representation of the drop impact test outcome. In the graphs, the mean values of each We interval are reported.

Figure 5. Image sequences of water drops (average diameter $D_0 = 2.45$ mm) impacting on three functionalized surfaces: (a) partial rebound with fragmentation on FAS sample at $We = 312$ at $t = 0, 14.6$ ms and $31.2$ ms after impact; (b) rebound on SI sample at $We = 54$ at $t = 0, 11.5$ ms and $31.2$ ms after impact; (c) rebound on S sample at $We = 21$ at $t = 0, 14.0$ ms and $21.6$ ms after impact. The scale bar in (a) is valid for all images.

Figure 6. Image sequences of hexadecane drops (average diameter $D_0 = 1.66$ mm) impacting on three functionalized surfaces displaying different behavior: (a) receding breakup on FAS sample at $We = 560$; (b) splash on SI sample at $We = 580$; (c) deposition on LAU sample at $We = 17$. For every surface, images at $t = 0, 2.0$ ms and $17.6$ ms after impact are reported. The scale bar in (a) is valid for all images.

Figure 7. Image sequence of water drop (average diameter $D_0 = 2.35$ mm) impacting on TQ surface at $We = 21$. A deposition occurs with the generation of a secondary drop. The scale bar in the first frame is valid for all images.
The evolution of the spreading film diameter of water and hexadecane after the instant of impact is shown in Figures 11a and 11b, respectively. The dimensionless diameter of the spreading film, called the spread factor, $\xi(t) = D(t)/D_0$ (i.e., the contact diameter at time $t$, made dimensionless by dividing it for the initial drop diameter) is shown as a function of time. Different outcomes were observed from deposition to rebound (occurring for $t=0$ when $\xi = 0$). In SHS cases, drops do not stick on the surface and the rebound of the droplets can be observed. When the drop is detached from the surface, the contact diameter is zero, therefore the spread factor is zero. The time evolution of the spread factor suggests that the drop impact dynamics depends not only on the $\theta_K$ value, since, contrary to what reported by Antonini et al.\textsuperscript{28}, even when $\theta_K$ reaches $120^\circ$ indicating a very low wettability, complete drop rebound does not occur (see tests with hexadecane on FAS surfaces, Figure 8b).

Figure 8. Spread factor time evolution, $\xi(t) = D(t)/D_0$, for the tested samples (single runs). (a) Water test conditions: $V = 1.6$ m/s, $D_0 = 2.5$ mm, and $We = 90$. (b) Hexadecane test conditions: $V = 1.5$ m/s, $D_0 = 1.7$ mm, and $We = 110$. The fact that a surface is superhydrophobic, i.e. with an extremely high advancing contact angle and low hysteresis, is not enough to assure the complete rebound, since impalement can occur.\textsuperscript{28} Therefore it is interesting to understand when the impalement impedes the bouncing and which are the possible outcomes of drop impact for the different fluid and surface characteristics. The impalement is usually described using alternatively four different pressure mechanisms, the water hammer pressure $P_{WH}$, the dynamic spreading pressure $P_{SD}$, the capillary or anti-wetting pressure $P_c$, and the gas layer pressure $P_{GL}$\textsuperscript{22}. 12 This last pressure term comes out considering the compression effects of the gas under the drop rather than the liquid compressibility. The initial impact of the droplet onto the surfaces generates a water hammer pressure $P_{WH}$ due to the compression of the liquid\textsuperscript{32-34}. The water hammer pressure $P_{WH}$ is equal to $\rho \cdot U^2/2$, where $\rho$ is the droplet density, $C$ is the velocity of sound in water, and $U$ is the droplet impact velocity normal to the wall. Due to the complexity of the drop wall interaction, for example looking at the air entrainment, such water hammer pressure is usually reduced of a factor $k$, which has been experimentally evaluated lying in a very broad range, for example 0.2 by Deng et al.\textsuperscript{33} or 0.002 by Maitra et al.\textsuperscript{22}. The real value of the impact pressure due to the liquid compressibility is here called effective water hammer pressure $P_{EWH}$. At the spreading stage, a liquid jump overtakes the outward moving contact line.\textsuperscript{36} At this stage the spreading process implies a dynamic pressure $P_{SD} = 0.5 \rho U^2$, where $V$ is the spreading velocity. For a normal impact $P_{EWH}$ is supposed to be greater than $P_{SD}$. The capillary pressure can be defined as the liquid pressure level that it is necessary to overcome in order to squeeze a droplet through a pore cavity, such as a surface topology throat. For that reason it is also called “anti-wetting” pressure. The capillary pressure can be written as $P_c = \sigma (\cos \theta_A)/r_p$, where $\sigma$ is the surface tension, $\theta_A$ is the advancing contact angle of the corresponding smooth surface and $r_p$ is a length scale of the surface cavities. The analysis of droplet impact on textured surfaces showed that the compressibility of the air layer between droplet and the substrate is a key feature for the impalement. Maitra et al.\textsuperscript{22} showed that the meniscus penetration is probably linked to the compressibility of the draining air, rather than the water hammer pressure effect of the liquid. Therefore, instead of the water hammer pressure, using a dimensional analysis they defined a pressure term $P_{GL} = 0.88(\rho_g \mu_g U^2/\rho_l \mu_l Ca)^{1/3}/St^{1/9}$, where $R$ is the droplet radius, $\mu_g$ the air viscosity, $Ca$ the Capillary number, and $St$ the Stokes number, as the critical pressure above which Cassie-to-Wenzel Transition occurs.

The impalement can be total, i.e. the liquid is reaching the bottom of the surface when the same is textured with pillars or is completely wetting the surface structure for a random topology, or partial, when the liquid is not able to wet completely and some air is still present in the surface throats and cavities. One can distinguished three regimes, the Wenzel regime where impalement occurs and is pre-
sent until the final deposition, being the liquid imbibed into the surface structure, a fakir (or Cassie-Baxter) regime, when no impalement and no imbibition occur and all the duration of the drop impact, from the early stages until the possible rebound or deposition, and a third stage, called partially impaled state, when a partial imbibition of water occurs. In the case of \( P_e > P_{\text{crit}} > P_{\text{slh}} \), the surface is textured and resists wetting in the contact and in the spreading phase; in the case of \( P_{\text{EWH}} > P_e > P_{\text{slh}} \), the fluid penetrates during the contact stage. In the fakir state, the liquid is partially imbibed as outcome of the drop impact is necerary to have sufficient energy at the end of the recoiling phase, thus promoting the drop detachment from the surface, i.e. it is necessary to have enough impact kinetic energy (the ki) for the netic energy of the impacting droplet to be larger than the surface energy dissipated during the retraction stage\(^{3,36} \), (bouncing fakir droplet – B); otherwise the drop does not rebound (non-bouncing fakir state - NB). When the liquid meniscus penetrates the surface topology, the partially impaled state occurs: at low impact velocity, partially penetrated bouncing droplet state (PPB) occurs instead of higher drop impact velocity, i.e. for a given critical velocity \( U_{\text{c,p}} \), the so-called second non-bouncing droplet (2NB) state\(^{32} \) occurs. The main problem is that there is still an open debate on the criteria for impalement, since, if it is clear that it is probable to either splash at high \( \text{We} \) or deposit at low \( \text{We} \) and the kinetic energy is increased, drop spreading and contact area become larger, spreading is limited, thus a high area between the drop and the surface. At \( \text{We} < 150 \), drop rising back from the partial penetration to a Cassie-Baxter regime. For water drops with \( \text{We} = 300+360 \), different outputs are observed for LAU and FAS surfaces notwithstanding their identical morphology (see Table 1 and Figures 2 and 8) and wettability (see Figure 1). The difference between these surfaces must lie in their chemical composition, as LAU is functionalized with non-polar alkyl chains while FAS exposes polar fluorinated groups. Murase and Fujibayashi demonstrated that water forms hydrogen bonds with such fluorinated moieties on hydrophobic surfaces. Therefore, we may consider that these interactions cause kinetic energy dissipation in the drop. Nakajima et al. calculated that this dissipation depends on the contact area between the drop and the surface. At \( \text{We} < 150 \), drop spreading is limited, thus water-surface contact area is small and the interaction negligible. Increasing impact speed, drop spreading and contact area become larger, therefore hydrogen bonds start to play a role causing dissipation and hindering rebound. When \( \text{We} > 500 \), water drops penetrate the terrace-like morphology of both LAU and FAS samples (e.g. partial rebound), therefore the role of chemistry is no longer relevant. Instead, for hexadecane drops, no influence of the surface chemistry can be recognized: the very low surface tension makes more probable to either splash at high \( \text{We} \) or deposit at low \( \text{We} \), when the kinetic energy is insufficient to break the drop. Only a small influence of the surface structure is observed, causing the splash-deposition transition to shift to lower Weber values for the S and SI samples with a
flower-like surface morphology (Table 1). This is the first time that this influence of chemistry on the drop impact behavior on textured surfaces is reported. However, other explanations can be evoked as well, e.g. a more inhomogeneous surface topology with respect to LAU molecules. Static or quasi-static wetting measurements might not highlight such differences in surface chemical composition. Furthermore, the present results emphasize how an accurate design of the surface properties must be pursued in the future research towards dynamically amphiphobic surfaces.

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