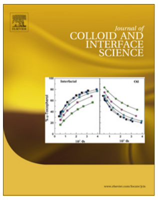
Journal of Colloid and Interface Science 409 (2013) 204–210

Contents lists available at SciVerse ScienceDirect

Journal of Colloid and Interface Science

www.elsevier.com/locate/jcis



X-ray computed microtomography for drop shape analysis and contact angle measurement

Maurizio Santini [a,]*, Manfredo Guilizzoni [b], Stephanie Fest-Santini [a]

[a] Department of Engineering, University of Bergamo, Viale Marconi 5, 24044 Dalmine (BG), Italy
[b] Department of Energy, Politecnico di Milano, Via Lambruschini 4, 20156 Milan, Italy

ARTICLE INFO

Article history:
Received 22 April 2013
Accepted 11 June 2013
Available online 26 June 2013

Keywords:
X-ray computed microtomography
Contact angle measurement
Drop shape
Surface reconstruction

ABSTRACT

The interaction between an atomized fluid and a solid surface has a great importance in many fields, both in adiabatic conditions and when heat transfer is involved. To investigate the behavior of many drops in contact with a surface, the first step is to study a single one of them and in that, surface wettability is key parameter. Wettability analyses are usually performed by contact angle measurement, in most cases using the sessile drop or captive bubble techniques. Such techniques require optical acquisition of a side view of the drop or bubble, with a series of drawbacks when conventional optics are used, in particular for not uniform, not planar or rough base surfaces. X-ray micro-computed tomography is therefore used to acquire a 3D scan of a drop gently deposited on a surface, with the aim to reconstruct the drop surface and to perform contact angle measurements on true cross-sections of the drop-surface couple. Comparison with contact angle measurements performed on conventional images is performed. The results evidence that the proposed technique is very promising for surface characterization and to get more accurate and detailed information about wettability characteristics.

1. Wettability, drop shape and contact angle

1.1. Introduction

The interaction between an atomized fluid and a solid surface has a great importance in many fields, ranging from engineering (e.g. fire fighting systems, heat transfer systems, cooling towers) to agriculture (e.g. irrigation and pesticide spraying) and medicine (e.g. aerosol therapies or needleless injection). To investigate the behavior of many drops in contact with a surface, the first step is to understand what happens to a single one of them. Both in adiabatic conditions and when heat transfer is involved, a key parameter is the surface wettability, i.e. how much the surface tends to keep the fluid adherent or on the contrary to repel it. Wettability is involved also when a bulk fluid comes in contact with a surface. The most widely used technique to evaluate wettability is the study of the shape that a liquid drop assumes on the same and to measure the macroscopic contact angle, i.e. the angle which at a macroscopic observation such drop seems to form with the surface, at the triple line. The contact angle can be evaluated in static or dynamic (drop advancement or recession) conditions. The static contact angle can be ideally related to the interfacial energies of the phases in contact, the advancing and receding contact angles to the dynamics of the triple line (e.g. pinning to the edges of the surfaces). In addition to its use as a “lumped parameter” for surface characterization, the contact angle is nowadays more and more required for CFD simulations, as a boundary condition when a three-phase flow is involved.

On an ideal surface (chemically homogeneous and inert, perfectly flat and smooth) and in absence of gravity or other external fields, the shape of a liquid drop resting on a solid surface would be a perfect spherical cap. Its equilibrium contact angle θ_Y would be directly related to the interfacial energies σ_{IJ} of the phases alone as described by the well-known Young equation [1] $\cos\theta_Y = (\sigma_{SV} - \sigma_{SL})/\sigma_{LV}$, where the subscripts indicate the phase: V vapor or in general gaseous phase, L liquid, S solid. Thus the contact angle would give a direct measure of how much the liquid tends to wet, to spread on the surface, or of the ratio between cohesion within the liquid and adhesion to the surface.

On real surfaces and including gravitational effects, many other phenomena take place so that the drop surface is not spherical and the contact angle differs from θ_Y. First of all, due to hydrostatic pressure the drop is no longer a spherical cap, but it is flattened. It can be described by the Laplace–Young [2] equation $\Delta P = \sigma_{LV}(1/R_1 + 1/R_2)$, where R_1 and R_2 are the two principal curvature radii. No general closed solution is known for the Laplace–Young equation, but it can be integrated using numerical techniques. Concerning the contact angle, many of the real-world effects on the contact angle are still partially open problems and

---

* Corresponding author.
E-mail address: maurizio.santini@unibg.it (M. Santini).

0021-9797/$ - see front matter © 2013 Elsevier Inc. All rights reserved.
http://dx.doi.org/10.1016/j.jcis.2013.06.036

topics debated in literature. Among them the influence of gravity (and consequently of the drop size) [3], of the dynamics of the triple line with its pinning to the microscopic or macroscopic edges of the surface texture [4,5], of physicochemical interactions between the liquid, the surface and the surrounding atmosphere [6,7]. A discussion of such issues is out of the scopes of this work and only their global effects will be taken into account. Such effects imply that on surfaces which are chemically not homogeneous or curved or rough, the contact angle may be, and in general is, different from point to point along the triple line. On rough surfaces, the apparent contact angle, i.e. the angle that the drop contour forms with an ideal horizontal line cutting the base surface at the contact points, is usually considered instead of the true contact angle, as it is able to provide valuable information about the macroscopic behavior of the surface when in contact with the investigated liquid. The apparent contact angle may be related to the real contact angle by means of different models, among which the most credited are still the Wenzel model [8] – for homogeneous surfaces in which the drop liquid completely fills the grooves in the surface texture – and the Cassie-Baxter model [9], to be used for chemically heterogeneous surfaces or when the drop does not fill the grooves.

1.2. Contact angle measurement and reconstruction of the drop surface

Despite its apparent simplicity, the measurement of the contact angle is far from being straightforward. In addition to the issues described in the previous section, problems may arise due to the measurement techniques. The most used method is probably the sessile drop technique [10], which is based on the acquisition of a picture of the drop-surface couple, usually a side view. In such a technique, the drop profile and the surface profile (which will be referred to as the baseline in the following) are extracted from the pictures via image processing algorithms and the angle they form is measured as the contact angle (see Fig. 1). This implies a series of drawbacks when conventional optics are used: image distortion by the lenses, small depth of field with focus problems (particularly for the identification of the baseline when the contact angle is very low or very high and the surface is rough), low or excess lighting or non-parallel light direction, low resolution of the image, diffraction near the contact points (particularly for very high contact angles) and evaporation of fluids due to the high intensity thermal radiation of illuminations.

Such issues are common also to the other measurement techniques, e.g. captive bubble, pendant drop, immersed plate (the Wilhelmy plate but with focus on the contact angle). Moreover, side views of the drop may not be equivalent to real cross sections of the same. This is particularly true when the base surface is not planar, so that optical access to the drop-solid contact surface system may be difficult or impossible. Finally, for rough surfaces only the apparent contact angle is experimentally accessible for conventional techniques.

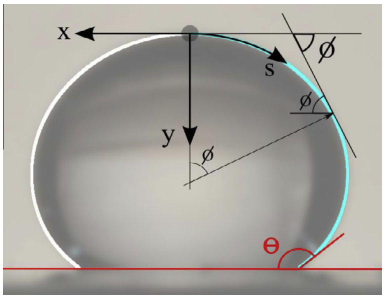


Fig. 1. Contour extraction and contact angle measurement for a water drop on a hydrophobic surface by photographic acquisition. Cartesian (left part of the figure) and arc-length (right part of the figure) coordinate systems for drop shape analysis are also shown.

1.3. Limitations of optical approaches

Estimations of contact angle are mainly based on contouring of 2D images acquired by traditional shadowgraph approaches. In several circumstances such optical transparency is not achievable due to intrinsic geometrical complication (opaque medium, asymmetries that shadow the contact line, concave surfaces or with curvature not allowing a free optical pathway, etc.). Recently, the interest in CFD simulation of liquid flow into porous media is growing, particularly for microscale structures, where information on governing parameters regarding the validity of standard contact angle measurements are still suitable of deep investigation.

On the contrary, using the X-ray micro computed tomography (microCT) the conventional optics problems do not play a role and true cross-section of the 3D volume can be obtained.

For this work, contact angles were measured and drop surface were reconstructed using the sessile drop technique, both with optical acquisition and with microCT. The three-dimensional and volumetric information offered by the latter approach allows to verify the effective axisymmetry of the drop and of the triple line, thus giving much deeper insight about the surface planarity and uniformity and validating the microCT technique applied to contact angle measurements for future applications in more complex (and opaque) geometries.

2. X-ray experimental set-up and procedures

2.1. Introduction to microCT

The investigation of matter using monochromatic and collimated X-rays is traditionally performed in synchrotrons.

In these facilities, high quality results can be achieved thanks to the very suitable, controlled parallel beam emission, with high brilliance, they are able to grant. However, the access to these facilities is limited to short beam-times, usually to be booked in large advance, and preferably allocated to non-conventional experiments.

Nowadays, an alternative can be offered by compact tomographic scanning systems, whose forefather was the first X-ray computed tomographic (CT) head-scanner – based on polychromatic and incoherent X-ray beams, differently from synchrotrons – by Hounsfield and Cormack (Nobel prize in Medicine 1979). Since that pioneering device, several research efforts have been developed to overcome the limitations of compact tomographic scanning systems, which are currently used in medical imaging.

Such systems generally use a cone beam X-ray emission, whose potential and required minimal spatial resolution (in the order of magnitude of some hundreds of microns) are adapted to the human clinical application.

During the last years, compact CT became an highly interesting instrumentation also in non-destructive technologies (NDT) for several industrial fields, thanks to the fact that X-rays allow non-invasive (at least in a certain range of energy) investigations even in opaque medium, in almost any condition and without requiring any special treatment of the sample (differently from, e.g., SEM analysis).

One of the emerging application is the use of NDT for 2D imaging of micro electro-mechanical system (MEMS) components, in order to evaluate localized defects in a scale range of a few microns. Such application requires an improvement obtaining a high spatial resolution, therefore this industrial request results in the