Technical Note

Practical Contact Angle Measurement (1)

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Drop Shape Analyzer – DSA100

Force Tensiometer – K100

Method:

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With care to accuracy: preparations and general conditions for contact angle measurements

This is the first of a series of articles on practical contact angle measurement. In this and the following issues we will cover a wide range of topics concerning drop shape analysis: from preparations for the measurement through drop deposition conditions and up to the choice of the methods used for analyzing the drop shape and calculating the surface free energy.

This first installment answers questions about the correct handling of the samples and test liquids as well as suitable ambient conditions.

The wettability of a solid by a liquid can dramatically alter even if there is only a slight modification to the chemical or physical properties of the surface. This fact makes the contact angle a sensitive quantity in surface analysis. However, the contact angle also reacts very sensitively to unwanted alterations in the participating phases.

Some easy-to-follow tips about sample preparation and information about »pitfalls« in contact angle measurement should ensure that this method produces reliable results.

Sample contamination

Each solid surface has its own particular history. This is why a »spoilt« surface usually can no longer be saved – after cleaning it is no longer the same surface as before the contamination. This is why unwanted alterations to the surface are to be avoided. Normally the greatest effects are seen when the surface comes into contact with fats and grease or surface-active substances. We focus on these two cases below.

Contamination by fats

The first important rule for contact angle measurement: never touch the surface with your fingers. Even the slightest traces of grease will have an effect on the contact angle results.
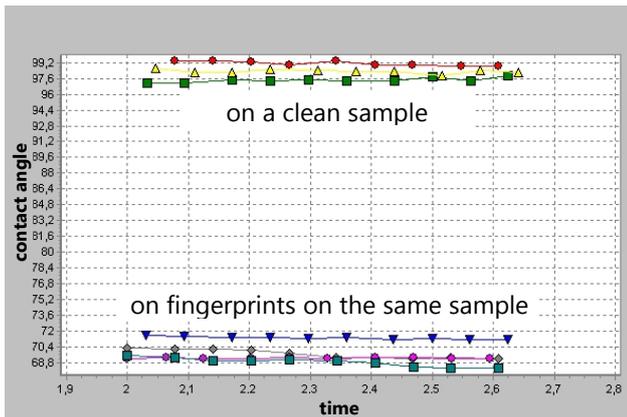
Fig. 1: Water contact angles on a polycarbonate sample

An often unexpected source of contaminants is compressed air produced by a compressor – if you want to dry a surface or remove dust particles by blowing them off with compressed air then it is essential that you use purified air.

Degreasing a sample

In many cases samples must not be cleaned before the measurement. In quality assurance the wettability of a material before the next process step is often important – cleaning it to prepare it for a laboratory measurement would alter the conditions.

Degreasing is advisable when the contact angle or surface free energy is to be determined as a property of the material. In this case cleaning should always be carried out under identical conditions – uniformly for all samples, and not just when the sample is obviously contaminated.

In many cases the acetone used in the lab for cleaning purposes frequently has proved to be unsuitable, as it does not evaporate without leaving residues. Acetone used for rinsing should therefore be very clean. Furthermore, the sample must be dried thoroughly since acetone adsorbs strongly to many surfaces. A solvent that can be used as an alternative is, for example, isopropanol. Cleaning should be carried out in an ultrasonic bath, so that the solvent can reach even microscopically small gaps and cracks.

During the measurement itself always measure the contact angle at a »fresh« position and never at one that has previously been wetted – even when the test liquid has already evaporated.

Surface-active substances

Never use surfactants for cleaning purposes. On many surfaces surfactants form tenacious adsorption layers, which in many cases cannot be removed by rinsing but only by mechanical means. And surface-active substances have a dual effect: not only do they alter the sample surface, they also reduce the surface tension of water, the most frequently used test liquid.

As many laboratories contact angle measurements and surfactant determinations go hand in hand, scrupulous cleanliness must be ensured. .

It is less well known that cigarette smoke is also unhealthy for contact angle measurements. The nicotinic acid contained in the smoke is very surface-active. Although smoking is not allowed in laboratories anyhow, even cigarette smoke from a neighboring office can have a demonstrable effect.

Irrespective of the ambient conditions, the sample should always be stored and transported in a clean and airtight container (e.g. a desiccator) and measured as soon as possible after sample preparation.

Static electricity

The next »trap« looms when the cleaned sample is dried. For plastics in particular, rubbing them dry produces electrostatic charges. As a result the deposited drops are deformed; in extreme cases they actually burst on the surface and form satellite drops. This is why it is better to dry the samples in a drying oven.
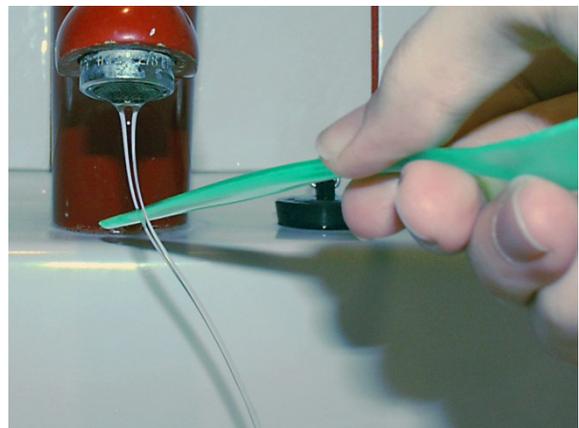
Fig. 2: The effects of static electricity on water
(photo: mariospla.net)

For the same reason samples should not be transported or mailed in plastic containers or bags. Aluminum foil is better suited for this.

However, with some materials electrostatic charges can hardly be avoided. For such samples ionization blowers are available on the market; these can be used to neutralize charges. Such an ionizer is already incorporated in the KRÜSS Tensiometer K100.

Test liquids

The same purity considerations as for the samples also apply to the test liquids. The surface tension of water reacts most sensitively to contaminants. This is why KRÜSS recommends the use of water in HPLC quality or bi-distilled water. In order to avoid the influence of plasticizers water should not be stored in plastic containers.

Special rules apply for the standard test liquids diiodomethane and ethylene glycol. The first decomposes in time due to the effects of light – this is why it is stored in amber glass bottles; it should no longer be used when a yellow-brown coloration can be clearly seen. In contrast, ethylene glycol – just like other diols or glycerol – is strongly hygroscopic; its surface tension alters as the amount of water it contains increases. Such substances should be stored under dry conditions in airtight containers.

The containers used for all test liquids should never be left open for a long time. In instruments with built-in storage reservoirs, for example the DSA100, the test liquids should be replaced after longer periods of use – after one week at the latest.

Ambient conditions

The chemical structure of solids is different at the surface from that in the interior. For example, under ambient conditions most metals immediately form an oxide layer. Above all, high-energy surfaces also shield themselves with a gas adsorption layer consisting primarily of air and water vapor. As this layer forms a thermodynamic equilibrium with the surrounding air this means that the atmospheric humidity also influences the measurement. Strong variations in the room climate should therefore be avoided. The same applies for the ambient temperature, which also has an influence on the surface free energy and, in particular, on the surface tension of the test liquids. If the measurements are to have an extremely good reproducibility then the instrument should be set up in a temperature and humidity-controlled laboratory (but not with the instrument exposed to an air stream). In addition, various environmental chambers are available from KRÜSS for measurements under defined conditions of temperature, pressure and humidity.

Summary

A pre-requirement for the reliable use of the contact angle method is the careful preparation and execution of the measurements. Some of the basic rules for the most important stations and general conditions have been collected and described. In sample preparation the primary aim is to avoid contaminant by substances such as fats or greases and surface-active substances. Cleaning procedures are also described. Further main topics are the avoidance and elimination of electrostatic charges and the proper use of the test liquids. Finally the importance of the ambient conditions in the laboratory is mentioned.

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