Application note

Measuring the water contact angle on surface-treated polymer films according to ASTM D5946

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Understanding Interfaces

Surface treatments are an effective way to overcome adhesion problems in chemically inert and non-porous surfaces with low surface energy. They are of great importance in many fields, such as the coating, printing, and varnishing industries. Several surface-treating-techniques, such as plasma-treatment and corona-treatment, have been developed to improve the wetting behaviour. Generally stronger treatment leads to better wettability and adhesion characteristics of the treated surfaces. The contact angle is a crucial parameter that reflects the effectiveness of the surface treatment. The ASTM D5946[1]: "Standard Test Method for Corona-Treated Polymer Films Using Water Contact Angle Measurements" is a widely used method to determine the effectiveness of different surface treatments. In the following, the application of the ASTM D5946 will be presented using an optical contact angle measuring system of the OCA series (Fig. 2) from DataPhysics Instruments.

Fig. 1: Water droplets on a polymer film

Keywords: OCA ▪ Contact angle ▪ Surface Treatment ▪ Plasma & Corona Treatment ▪ Polymer Film ▪ ASTM D5946

Technique and Method

The contact angle of a liquid, dosed on a solid surface, gives an easily accessible indication of the surface adhesion between the solid and the liquid drop. According to the ASTM D5946 standard, the contact angle of water can be used to define the effectiveness of surface treatments on polymer films. As stipulated in the standard, the water contact angle can be measured by capturing an image of a liquid drop sitting on a solid and subsequently analysing it.

The optical contact angle measuring and contour analysis systems of the OCA series (Fig. 2) combine high resolution optics, exact liquid dosing and precise sample positioning into powerful and reliable measuring systems. Combined with its matching software, the contact angle can be evaluated easily and quickly.

Thus, systems of the OCA series are suitable for determining the wettability of treated films according to ASTM D5946.

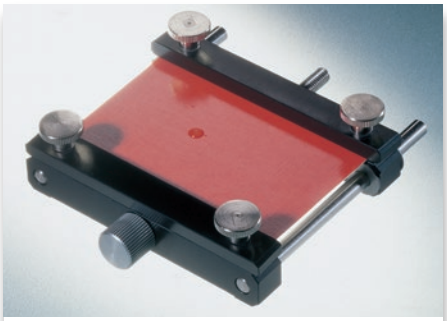

Fig. 3: The film and paper holder FSC 80 can be used to fix thicker films or papers with adjustable tension.

Fig. 2: Optical contact angle measuring and contour analysis system of the OCA series

Experiment

In this application note, the water contact angles on differently treated polymer films were determined with an OCA 25. According to the requirements of the ASTM D5946, the width and length of sample film are at least around 25 mm and 300 mm, respectively. As environmental conditions will influence the measured value to a certain extent, the tests are conducted at a temperature of 23 ± 2 °C and a relative humidity of 50 ± 10%. The handling of polymer films is prone to build-up of static charges, which potentially leads to inaccurate contact angle measurements. To avoid this, an air ioniser is used to keep the samples free of static build-up before and during the measurements.

As mentioned above, both plasma-treatments and corona-treatments can be applied to treat polymer surfaces. In this application, the sample film was plasma-treated immediately before the measurements using

the handheld piezobrush® PZ3 (with the "Module Standard"), developed by relyon plasma GmbH. The test areas were treated for 3 s, 5 s, 10 s, 20 s and 30 s, respectively.

To ensure the purity of the distilled water and cleanness of the syringe, a preliminary test was carried out. In it, the surface tension of the water used for the later measurements was verified tree times using the pendant drop method[2]. The measurements yielded a surface tension of 72.28 ± 0.32 mN/m, which is in good agreement with literature values[3].

To ensure the accuracy and reproducibility of the results, the samples were taken out of their packaging and placed on the device extremely carefully without directly touching the test area. The sample film was fixed inside the film and paper holder FSC 80 (Fig. 3) and vertically oriented on the sample stage of the OCA 25. The film holder made it possible to easily position the sample wrinkle free.

A water droplet of 6 µL, which lies within the ASTM standards range of 5 to 8 µL, was suspended at the end of a syringe needle. The sample platform was raised slowly until it touched the pendant drop. Then, the sample platform was moved down carefully to complete the transfer of the droplet to the sample film. Taking into account the possible variations in treatment and surface roughness of the sample films, measurements were carried out ten times per sample. Additionally, one contact angle measurement is taken every 25 mm across the sample, to determine any changes across the surface.

With the automatic evaluation function of the software, the mean static water contact angles on the treated and untreated samples were obtained using an elliptical fit to the drop shape.

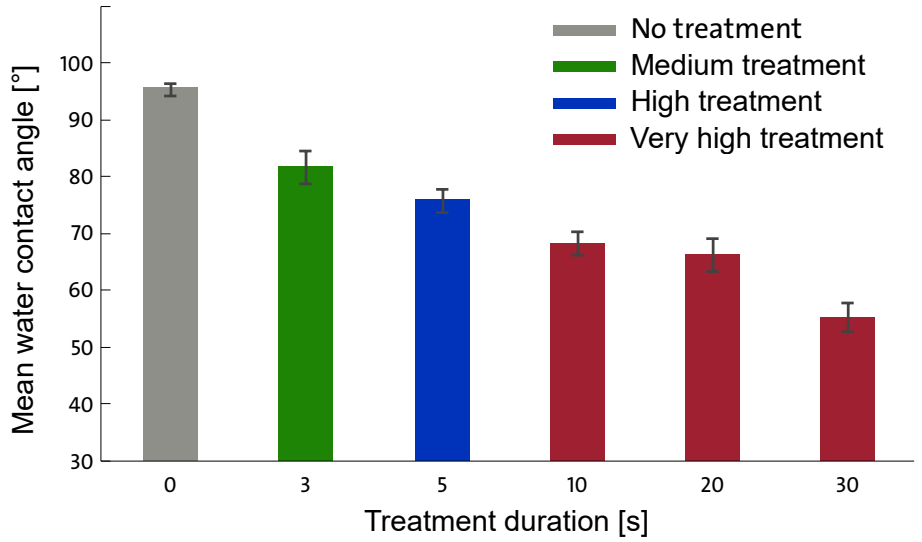

Fig. 4: The mean water contact angle on polymer films decreases with increasing treatment duration.

Results

Fig. 4 shows the static contact angles determined for the studied samples. There were only small deviations within the ten repetition measurements for the same sample, which results in low error bars (max. ± 2.9°).

The mean water contact angle on the untreated film is 97°, indicating the wettability of the original surface is low and the surface is therefore hydrophobic. In contrast to the untreated film, the water contact angles on the plasma-treated surfaces are all below 90°. Hence, the plasma-treatment altered the wettability of the polymer film. A surface with a lower contact angle is more hydrophilic.

According to the ASTM D5946 standard and as displayed in Table 1, the film samples treated with plasma for 3 s and 5 s can now be classified as having received medium or high treatment, respectively. In addition, when the film samples were treated longer than 10 s, they received a 'very high treatment' according to the standard.

Table 1: The level of surface treatment of polymer films with initially low surface energies is defined in the ASTM D5946 according to the range of water contact angles.

Level of surface treatment	Water contact angle
Marginal or no treatment	> 90°
Low treatment	85° to 90°
Medium treatment	78° to 84°
High treatment	71° to 77°
Very high treatment	< 71°

Summary

The optical contact angle measuring and contour analysis system of the OCA series from DataPhysics Instruments provides an easy and reliable way to determine the static contact angle on treated and untreated film surfaces. Hence, the level of surface treatment according to ASTM D5946 can be evaluated accurately.

This technique is one of the most powerful methods to study the wetting behaviour of treated surfaces, which is very important in the development of surface treatment methods.

References

[1] ASTM Standard D5946, 2017, "Standard Test Method for Corona-Treated Polymer Films Using Water Contact Angle Measurements", ASTM International, DOI: 10.1520/D5946-17, www.astm.org.

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[3] N. B. Vargaftik, B. N. Volkov, and L. D. Voljak , "International Tables of the Surface Tension of Water", Journal of Physical and Chemical Reference Data 12, 817-820 (1983), DOI: 10.1063/1.555688

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