

Granules typically consist of various compounds including organic excipients and dispersion liquids (Fig. 1). They play a very important role in many different areas as an easy to handle raw material from which pharmaceuticals, filling and building materials can be made through extrusion processes or injection molding amongst others. While raw material in granule shape is easy to handle regarding transportation and storage, they are normally processed in a **molten condition**. To plan the processing the **surface tension of granules** needs to be measured. However, the surface tension has to be determined at **high temperatures in the molten state**, which is a challenge for general surface tension measurements. The dynamic contact angle measuring device and tensiometer **DCAT** with temperature chamber **TEC 250/DCAT** from DataPhysics Instruments can perform surface tension measurements **easily and precisely** at temperatures of **up to 250 °C (300 °C with heat shield)**. In the following the application of the method will be presented with examples of three different granules.



Fig. 1. Example of commercial granules.

Keywords: DCAT • Wilhelmy Plate Method • Surface Tension • High Temperature (up to 300 °C) • Molten Granules

Technique and Method

The surface tension acts to contract and minimise any liquid surface. If a surface is being enlarged by the formation of a liquid lamella, the surface tension exerts a tensile force that can be measured.

The **Wilhelmy plate method** is a well-established method for determining surface and interfacial tensions with a tensiometer. The method utilises a Wilhelmy plate as the test body. It is typically made of platinum–iridium and is a few centimetres in length and height. The plate is attached to the weighing system of the tensiometer and positioned at the liquid surface in such a way that a liquid lamella forms (Fig. 2).

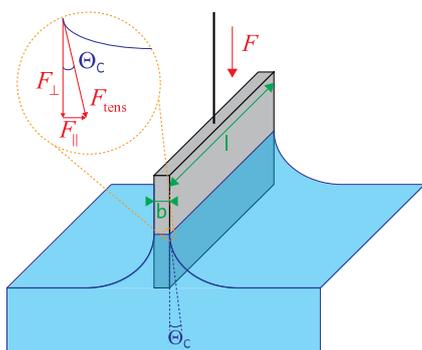


Fig. 2. Wilhelmy plate with liquid lamella; Wilhelmy equation

The gravitational force of the lamella, which equals the perpendicular part of the tensile force F_{tens} caused by the surface tension σ , is measured. Together with the definition of the surface tension as tensile force per length of the contact line this yields the Wilhelmy equation:

$$\sigma = \frac{F_{tens}}{L} = \frac{F_{\perp}}{L \cdot \cos \theta_c} = \frac{m_{lamella} \cdot g}{L \cdot \cos \theta_c}$$



Fig. 3. Tensiometer of the DCAT series with electrical temperature control unit TEC 250/DCAT

$m_{lamella}$ is the measured weight of the lamella, L is the length of the contact line and θ_c is the contact angle. The Wilhelmy plate is made from platinum-iridium with a very high surface energy and gets thus completely wetted by any liquid (θ_c is 0°). This enables a direct determination of the surface tension knowing the plate dimensions and the measured force.

The tensiometers of the DCAT series from DataPhysics Instruments (Fig. 3) are universal measuring systems for the **force-based** study of interfacial parameters and phenomena by a **high precision weighing system**. With the electrical temperature control unit **TEC 250/DCAT** (Fig. 4), measurements at temperatures of **up to 250 °C** can be performed and with an optional heat shield up to **300 °C** are possible. Hence, **resins, hot melt adhesives, solder and other molten solids** can be analysed easily.

Experiment

The surface tension of three different molten granule types was studied:

- (i) Fractional palm fatty acid (F100) which is mainly used as additive for pet food
- (ii) PE wax that is used as a coating to influence mechanical or optical properties
- (iii) Zinc stearate which is used as stabiliser of emulsions

At room temperature, all granule samples were solids but became fluid at a temperature of 150 °C with the temperature chamber TEC 250/DCAT. Depending on the sample type, the relevant analysis temperature of F100, PE wax and zinc stearate were 80°C, 110°C and 150°C, respectively.



Fig. 4. Electrical temperature control unit TEC 250/DCAT with accessories.

For the surface tension measurement, a vessel with the granule sample was placed in the DCAT and heated up until the material melted. The Wilhelmy plate was immersed 3 mm into the molten sample. The weight of the lamella formed on the plate was detected (at zero immersion depth) by the high-precision balance inside the DCAT and the surface tension was evaluated automatically by the DCAT Software.

To ensure the accuracy of the results, each sample was measured three times. To avoid influencing the results by a pre-wetting effect, the Wilhelmy plate was cleaned after each measurement.

Results

Table 1 shows the average surface tension of the samples at different temperature.

Table 1. Mean value of surface tension of three molten granule samples at different temperatures.

Sample	SFT [mN/m]	T [°C]
F100	25.83	80
PE Wax	26.61	110
Zinc stearate	22.40	150

All samples showed relatively low surface tensions below 30 mN/m. The mean values of surface tension of F100 at 80 °C, PE wax at 110 °C and zinc stearate at 150 °C were 25.83 mN/m, 26.61 mN/m and 22.40 mN/m, respectively.

Summary

The tensiometer of the DCAT series with an electrical temperature control unit from DataPhysics Instruments was able to **fast and easily** determine the **surface tension** of **molten granules** in a **temperature range of 80-150°C**.

This technique provides an easily accessible and **reliable** way to study the **wetting behaviour** of **molten solids** at **high temperatures**, which is of great significance for characterizing new raw materials in various areas, like building materials, pharmaceuticals, automotive etc.