

The application of Wettability-based Test methods to Fiber-based materials

Thermo Fisher Scientific, Process Instruments, Newington, USA

The Thermo Scientific CAHN 322 Dynamic Contact Angle Analyzer is a versatile, multi-use instrument which is ideally suited for the characterization of a wide range of fibers and fibrous materials used in a variety of industrial applications. In addition to its primary function as a contact angle wettability and surface tension system, the unique Fiber-Range feature of the Thermo Scientific CAHN DCA-322 can also be used in a secondary mode to evaluate:

- Fiber diameter profiles
- Surface cleanliness and homogeneity
- Wicking rate absorbencies
- Coating uniformities
- Microgram weight and density characteristics

A brief discussion of each of these functions follows in this note.

Fiber diameter profiles

Characterizing a fiber by making a diameter measurement along its length can be a difficult procedure, especially for the very small diameter high-performance carbon, polymer, and textile fibers. In addition, variations in fiber diameter along the length of the fiber can also be difficult to characterize with traditional techniques.

Fiber diameters are typically measured optically with a special microscope attachment, or estimated with the aid of a good quality micrometer. A simple extension of the modified Young Equation (used by the DCA for both surface tension and contact angle wettability measurements), however, can also be applied for accurate fiber diameter profiles. This is done by scanning the fiber with a liquid of known surface tension that will completely wet-out the fiber (ie; form a zero contact angle). By knowing the surface tension (γ) and the contact angle (θ),

and recording the wetting force (F) between the fiber and probe liquid, the fiber perimeter (p) can be calculated directly from the Young Equation.

$$p = F / \gamma \cdot \cos \theta$$

When $\theta = 0$ ($\cos \theta = 1$), it is possible to solve the equation directly for perimeter.

For a round cross-section (cylindrical) fiber, the perimeter is equivalent to the circumference ($\pi \cdot d$), thus by dividing the perimeter (p) by π , the diameter of the fiber is directly determined from a DCA scan. And by scanning along the full length of the fiber, diameter variations can be easily recorded and calculated at intervals as small as 2 microns when running the stage at the slowest speed (2 $\mu\text{m}/\text{sec}$).

To test this method, two wire samples, one platinum and one of nickel-chromium (nichrome) were used as test pieces. Wet-out liquids chosen were hexadecane (for nichrome wire), and water (for platinum wire). The surface tension of these liquids is below the surface energy of each wire, thus a zero contact angle is assured with each corresponding (clean wire surface). Average diameters calculated for each fiber in both advancing and receding modes (columns 4

and 5 respectively) are compared in the table below against the manufacturer's label (column 2), and a value obtained with a standard micrometer (column 3).

Note from the data above, that fiber diameters calculated from contact angle measurements are reproducible (see platinum wire runs 1 & 2), and report fiber diameters that reflect intermediate values averaged over the entire length of the fiber surface. With this technique, fiber diameters can be calculated for each data point making it possible to record a manufacturer in fiber diameter at the 2 micron scale.

Surface cleanliness/homogeneity

The sensitivity of a dynamic contact angle hysteresis profile to monolayer surface contamination, roughness, and homogeneity is another powerful feature of the DCA system. A wettability profile of the fiber surface with a standard liquid such as water can create very small changes in the fiber surface that might otherwise go undetected by conventional assays.

The graph in Figure 1 is a good illustration of a wettability cleanliness assay applied to metal wire.

An overlay of a wettability scan with water shows an advancing contact

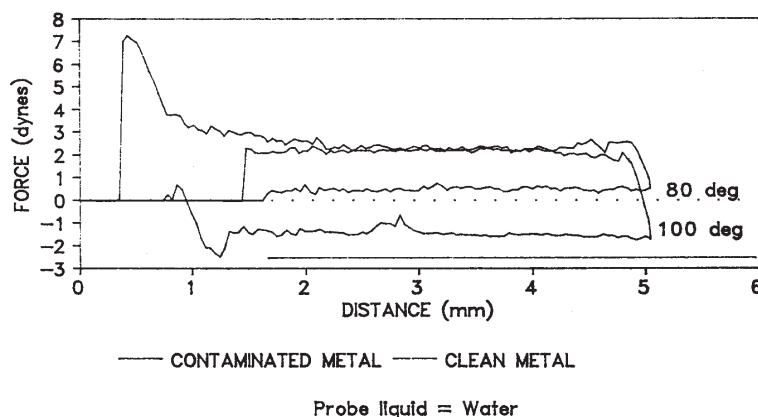


Figure 1: Cleanliness of metal fibers by contact angle hysteresis

Fiber diameter (mm)

Fiber type/ Probe liquid	Label reading	Micrometer measurement	From Advancing contact angle	From Receding contact angle
Nichrome/ Hex adecane	0.1778	0.1651	0.1696	0.1699
Platinum/Wat ER (Run #1)	0.0762	0.0635	0.0709	0.0710
Platinum/Wat ER (Run #2)	0.0762	0.0635	0.0709	0.0710

angle for the clean metal wire of 80 degrees, as compared to a 100 degree advancing contact angle for the con-taminated wire sample.

Although both scans show a similar receding trace, the hysteresis between advancing and receding angles is significant.

Absorbency

The absorbency of a fabric or paper product can be characterized with both wettability and wicking rate data for an individual fiber or fiber bundle. The DCA-322 is ideal for wicking rate measurements when utilized in a force vs time mode. In doing these experiments, the liquid level is raised up at a slow speed until contact is made between sample and liquid at the interface. Data is collected in real time until saturation is reached, and the rate of liquid uptake into the sample is calculated. The example in Figure 2 shows a wicking rate comparison between two lots of a wood pulp material. The reproducibility of these measurements is highlighted by the data in which two runs of each fiber bundle were made with water as the absorbing liquid.

Coating Uniformity

In the textile and composite industries, the nature and uniformity of coatings applied to protect the fiber surface before, during, and after processing must be carefully controlled. As in the cleanliness application described above, contact angle hysteresis is a simple, yet sensitive technique that can be applied to scan the entire fiber surface for uniformity characteristics. Typically, a fiber sample is mounted to a wire support, and immersed several times into a probe

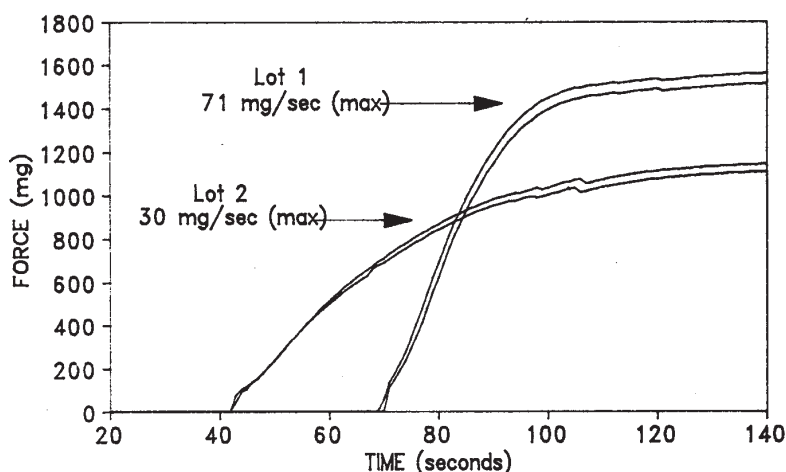


Figure 2: Wicking rate (Paper pult in 18% sodium hydroxide)

liquid of known surface tension. If the contact angle formed with the fiber is non-zero, the hysteresis between advancing and receding angles is a good measure of coating uniformity. For this application, the choice of probe liquid is important not only to insure a non-zero contact angle, but also to insure chemical compatibility.

Microgram weight and density measurements

At the heart of every DCA-322 system is a Thermo Scientific CAHN electronic microbalance coupled to a software controlled data acquisition system. The high precision of the Thermo Scientific CAHN microbalance and positioning capability of the programmable stage mechanism support traditional weighing applications such as density and differential weight measurements. The manual experiment mode of the DCA-322 can be used for these applications, and additional accessories such as sample pans and other weighing accessories are also available from Thermo Scientific CAHN.

Popular fiber Applications for the DCA System

- Protective chemical treatments applied to carpet fibers and other textiles.
- Plasma, corona discharge, and chemical sizings applied to carbon, glass, polymer, or wood fibers used to improve adhesion at the fiber/matrix interface in composite material systems.
- Surface treatments applied to cellulosic and synthetic superabsorbent fibers to improve wettability and absorbency for sanitary applications (ie; diapers, feminine care products, surgical dressings), and to prevent condensation or absorb moisture in food packaging containers, pharmaceutical products, and medical devices.

Thermo Fisher Scientific
Process Instruments

USA
25 Nimble Hill Rd.
Newington, NH 03801
Tel. 603 436 9444
info.mc.us@thermofisher.com

www.thermo.com/cahn

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