

Preparing single-sided samples for DCA analysis

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Samples that have undergone a surface modification treatment have often been exposed on only one side to the treatment, with the other side unaffected by the process. We refer to samples that have been treated on only one face as “single-sided” in recognition of the one-sidedness of the treatment process. Because the DCA is an immersion technique, by definition all sides of the sample will be exposed to the wetting liquid during the experiment. If only one side of the sample has been surface-modified, the meniscus that forms around the perimeter of the sample will in this case be non-uniform, reflecting the difference in wettability between the treated and untreated sides of the sample. The Thermo Scientific CAHN DCA, which records the wetting forces associated with meniscus formation, cannot separate the contribution of the treated side as distinct from the untreated side. Thus the wetting force recorded will be essentially an “average” or “composite” over the entire perimeter of the sample. Failure to consider the effect of one-sidedness when carrying out a DCA experiment, can lead to very serious errors in data interpretation.

There are at least two approaches that could be used when dealing with one-sided samples. One approach assumes that the wettability of the untreated side of the sample is well known and can thus be treated as a constant. Using this approach, the DCA experiment could be carried out in the normal fashion using the one-sided sample, and the computer could then be used when analyzing the data to factor out the wettability of the untreated surface from the composite sample to yield wetting data specific to only the treated surface.

In theory, this is a very straight-forward approach that is easily done using a computer program, but from

an experimental perspective this approach has limitations. The primary limitation concerns an important assumption that must be made for the method to work: the wettability of the untreated surface must be known in advance or treated as a constant. If this assumption is made in error, the computer correction cannot be accurately applied and the approach fails to yield the desired results. If this assumption is valid, computer corrections can be readily applied.

The alternative approach is taken from the sample prep side and does not require computer corrections. Whenever possible, this is the preferred approach to use when dealing with single-sided samples. Very simply, this approach involves the use of a double-sided tape or adhesive that is inserted between two like pieces of the sample such that only the sample side of interest is exposed to the wetting liquid. In other words, the double-sided tape or adhesive is applied to the untreated sample surfaces only. Figure 1 illustrates the use of this method in preparing a single-sided sample for analysis. This method is particularly convenient when preparing film samples. With thin films in particular, the tape also adds rigidity to the sample making it easier to penetrate the liquid interface during an analysis. If the edges of the sample are also untreated, it is best to prepare a wider sample so as to minimize the contribution of the edges with respect to the perimeter of the sample. If the edges contribute less than 5% to the entire perimeter, the error caused by the different wettability of the edges can be considered minimal.

When using double-sided tape, it is best to apply a strip of tape initially that is wider than the sample width and then trim the edges to ensure that the tape extends all the way across the width of the final sample.

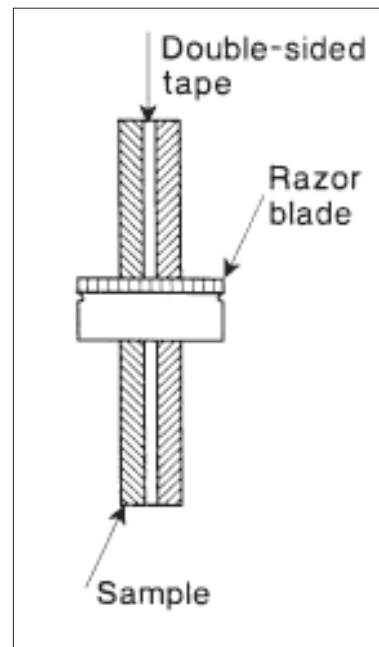


Figure 1: Razor blade cutting “sandwich” of 2 samples with double-sided tape

This will minimize the possibility that liquid will penetrate through the middle of the sample near the edges during the experiment. If an adhesive is used instead of double-sided tape, it is important to insure that the adhesive does not migrate to the surface of the sample and affect the wettability.

This mounting technique has been used extensively and has proven to be a good approach in dealing with single-sided samples, thus it is recommended over the computer correction approach which has limitations that can be difficult to control by the operator.

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C163_05.11.07

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