A New Dynamic Contact Angle System

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Objective

To illustrate the use of contemporary personal computer technology and Windows™ software to acquire and process video images for surface science studies. The system described uses RS-170 video and 80486 VESA CPU hardware, both of which are very affordable. The system can capture images at surprisingly high rates.

A secondary objective is to show that significant information is available directly from the images themselves, in addition to the numeric data derived from image analysis.

Equipment

The instrument, the "FTÅ200," is built around a continuous frame grabber which is capable of digitizing full RS-170 (NTSC) frames at VGA resolution (640 x 480 pixels). These images are fed into a buffer set aside in Windows' main memory. When it is desired to capture one or more of the images, they are fetched from the buffer and set aside elsewhere in the main memory. A sequence of captured images is a "movie." The system functions as a transient recorder with a "trigger" selectively saving certain images into the movie.

A typical measurement is performed as follows: (1) setup the experiment and program the required fluid dispensing, lighting, movie triggering mode, and the desired length of the movie; (2) begin the experiment, waiting for the trigger event which causes the movie to be captured; (3) view each frame of the movie in turn and analyze what is desired from each frame. These analyses are based on the geometry and dimensions of the image. Finally, graph the resulting data or export to a database for further manipulation.

To summarize, the movie consists of an encapsulation of a timed sequence of images, the actual times at which they were taken, and any image analysis which the user has performed. The movie is stored and treated as a database.

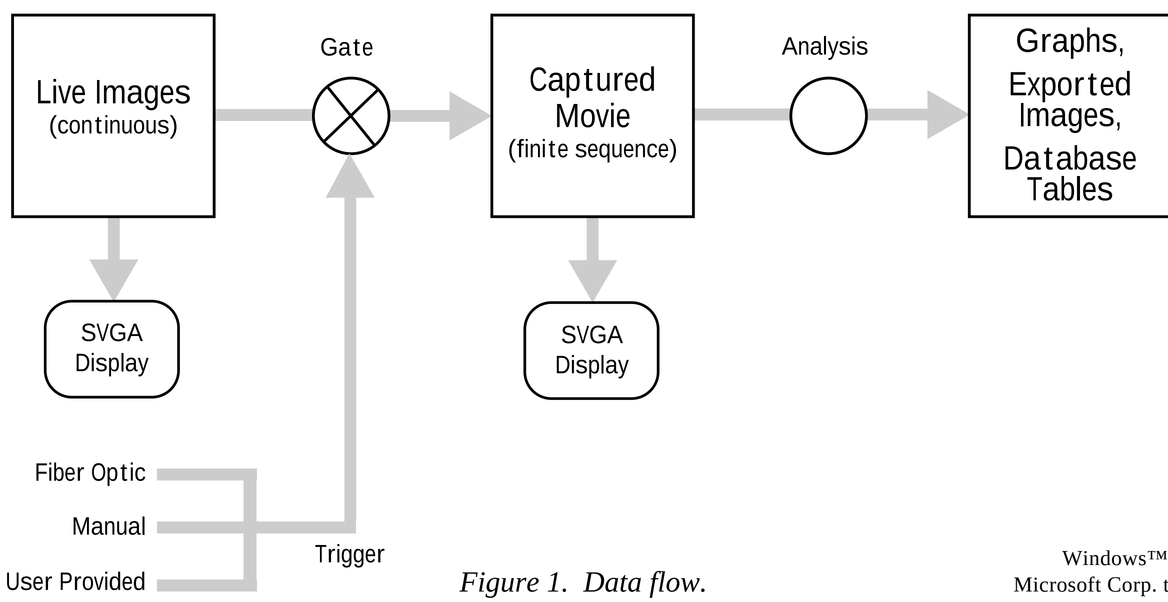


Figure 1. Data flow.

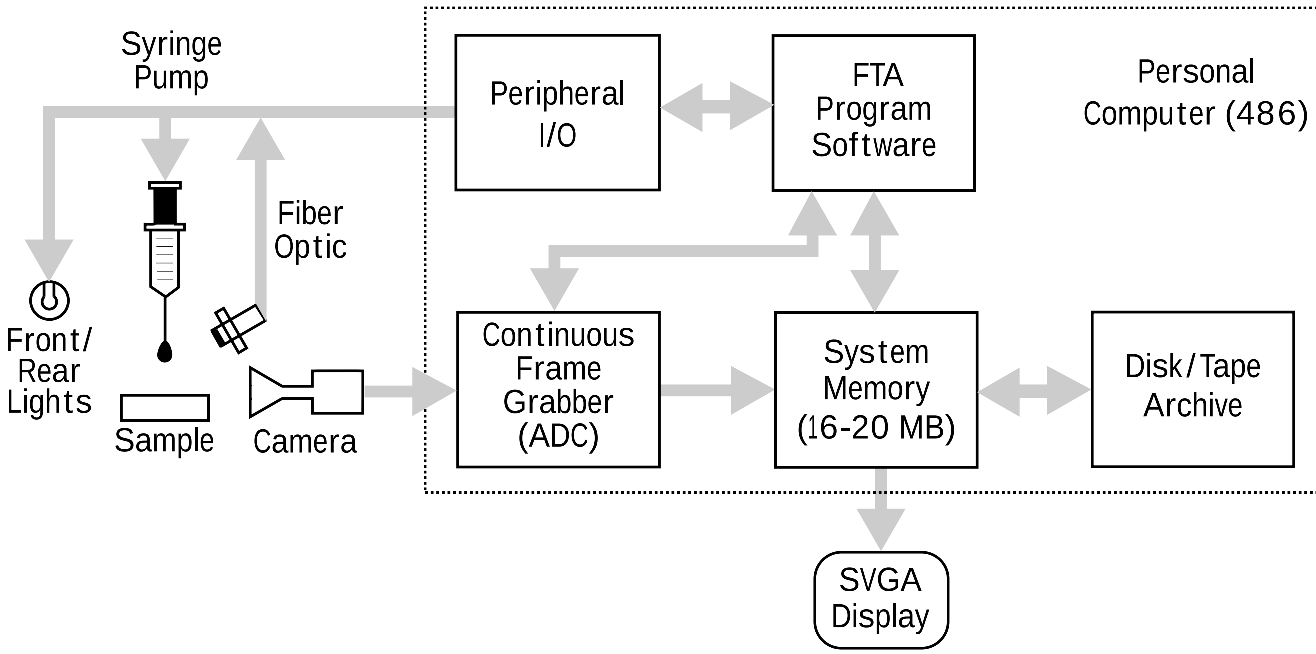


Figure 2. Hardware configuration.

The continuous frame grabber relies on the fast local bus to move all of the video information to the main memory. Data is digitized by the frame grabber as an 8-bit gray scale. The user's display shows all images through normal Windows display drivers, thus "what you see is what you get."

The integrated peripheral I/O controls the syringe pump, illumination lamps, and a fiber optic drop sensor, which are manipulated in a timed sequence to "run" the experiment. Other sensors and robots that handle specimens are also integrated into the system.

Live images are continuously captured by the frame grabber and transferred, as Windows has time, to the user screen. The update rate will be continuous ("real time") if Windows is not busy, but if Windows does load down, the screen update occurs less often. However, the continuous image stream is *always* being sent to main memory, even if it is not being forwarded to the user's screen. When a movie is captured, the required images will *always* be in memory, whether or not they were shown live on the screen. The movie capturing process opens the Gate in Figure 1, letting the images flow into a separate portion of memory.

Gray scale images are analyzed for linear dimensions, contact angle and surface tension, plus other data that can be derived from these results. A good reference explaining these techniques, and the numerical calculations for their practical determination, is "Surface Tension by Image Analysis: Fast and Automatic Measurements of Pendant and Sessile Drops and Bubbles" by F. K. Hansen, *Journal of Colloid and Interface Science,* **160**, 209–217 (1993).

Applications

The remainder of the paper will illustrate typical applications of the system. All graphs were plotted from data derived from movie images by the system. All photos were taken with a 35mm camera directly from the computer screen running the program. The photos are of representative images in the movie. Each movie has many more images than those shown here.

Contact Angle

Contact angle is the angle between the tangent to the drop's profile and the tangent to the surface, at the point of meeting between the atmosphere, the fluid, and the solid (see Figure 3). The difficulty is in accurately estimating the tangent to the curved drop profile because the curve ends at the point where one needs to measure it.

The graph shown in Figure 4 on the next page shows the contact angle of a 10ml drop of acetone placed on a fiberglass cloth. The acetone absorbed into the cloth quickly (in about 200 milliseconds). This movie was taken with 16-millisecond frames, or 60 frames per second. This sample was taken from a study of how

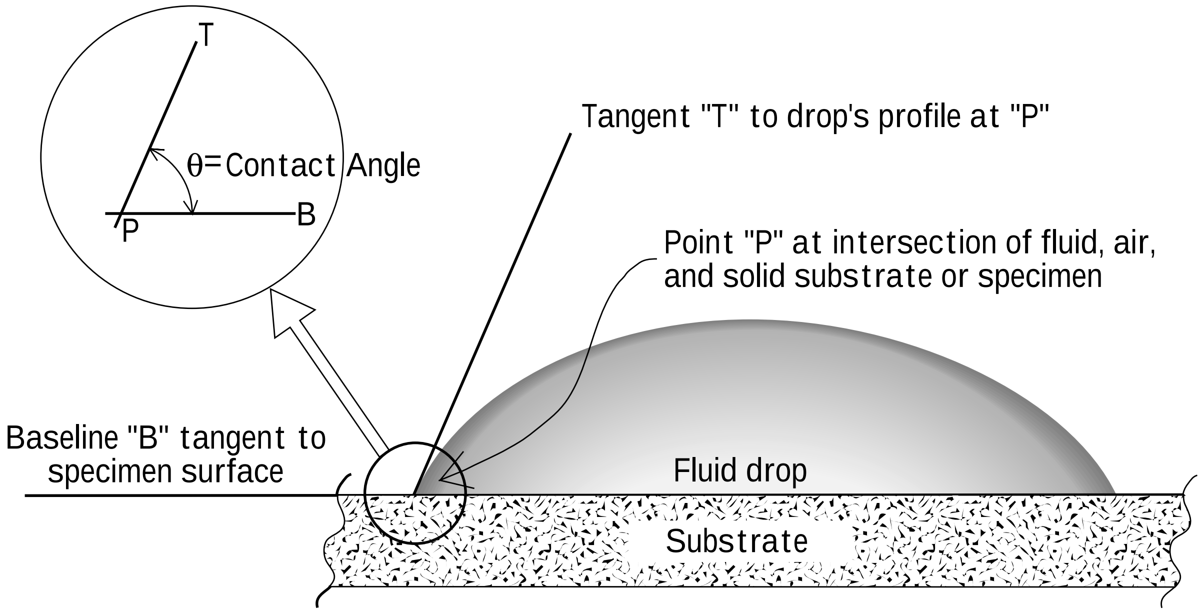


Figure 3. Contact Angle Measurement

organic residues affected wetting of this cloth used in electronic printed circuit board fabrication.

The graph shown in Figure 5 shows the contact angle for a drop of water placed on a polypropylene fabric. In this case, the drop stood on the fabric for about 22 seconds, then absorbed quickly. The sample was of a material used to make disposable diapers. The time resolution in this movie was 500 milliseconds.

Many variations in the basic measurements are possible, each having significance to particular industries. The specimen may be tilted, held in an environmental chamber or mounted on a customized stage. The basic contact angle data can also be manipulated within the program to derive other quantities of interest.

When absorption is the focus, standing volume (volume of drop not yet absorbed) may be derived from contact angle and the drop's base width. When wettability is the focus, advancing and receding contact angles are significant.

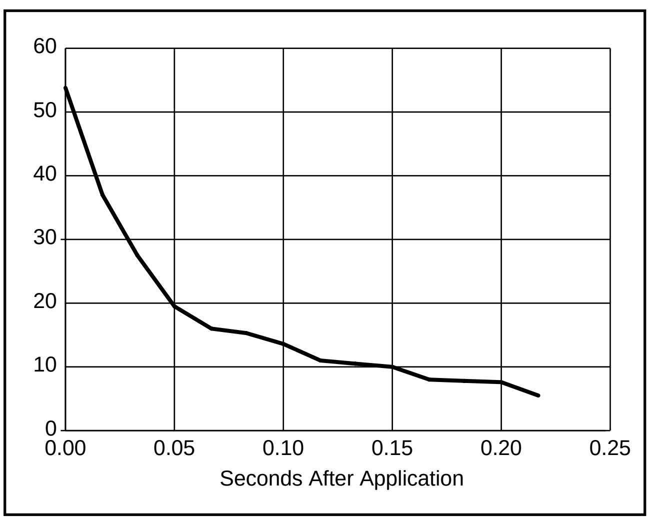


Figure 4. Fiberglass / acetone contact angle.

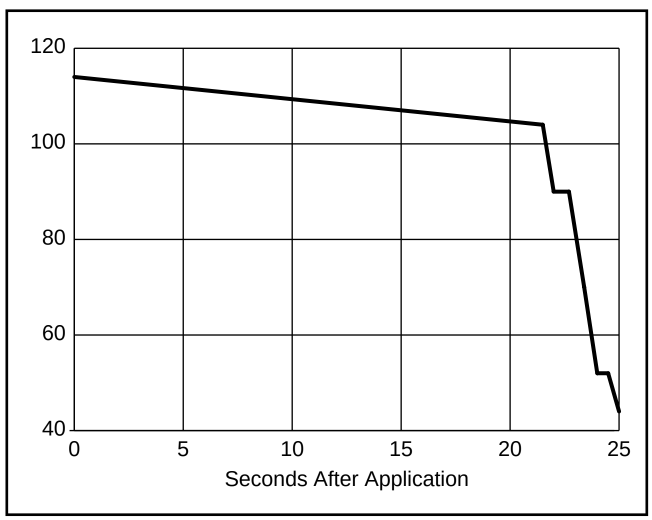


Figure 5. Non-woven fabric / H_2O contact angle.

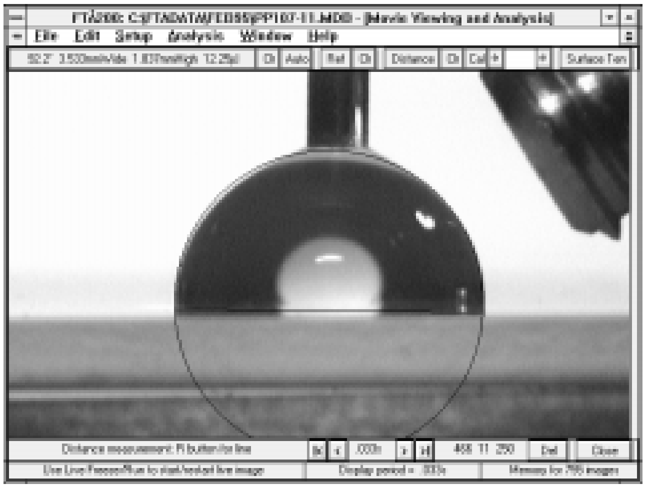


Figure 6. An advancing contact angle.

An *advancing* contact angle occurs when the drop is expanding its periphery; a *receding* contact angle occurs if the drop is retreating and its periphery is becoming smaller. The specimen in the photos above and below is a vinyl printing plate and the fluid is water. The advancing angle in Figure 6 shows a contact angle of 92.2˚ and the receding angle of Figure 7 is 67.1˚. The difference is called *hysteresis*. This plate was barely printable because of the high (advancing) contact angle.

Note the computer-generated guidelines that describe the circumference of the drop and the surface of the plate in these photos. In many applications the computer can accurately determine these lines and make all calculations totally automatically. In less than ideal lighting conditions, the FTÅ200 program provides a

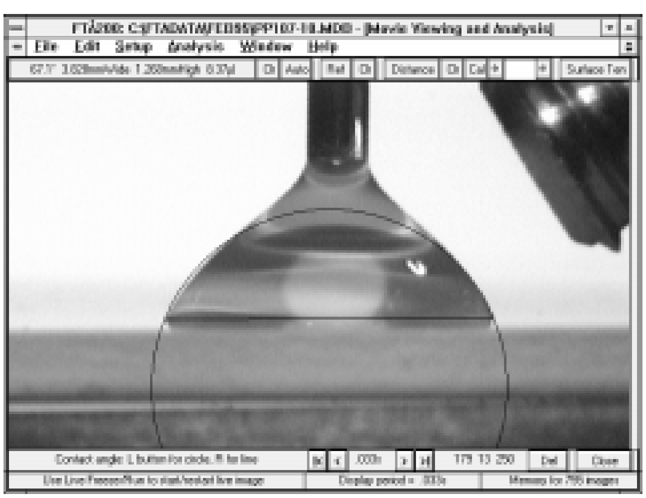


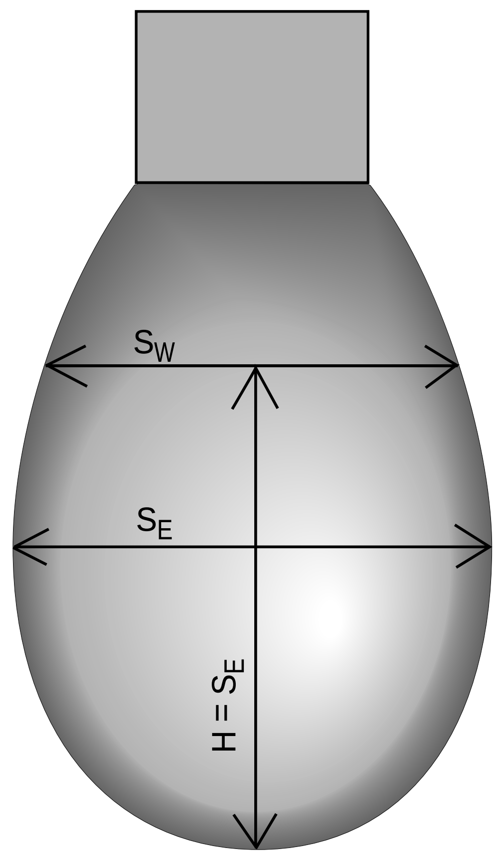
Figure 7. A receding contact angle.

mechanism that allows the user to manually adjust the guidelines to accurately describe the geometry of the experiment.

Surface Tension

Surface tension can be measured from a drop's shape (see Figure 8). Given the fluid density and two measurements of the drop's shape, a lookup table provides the surface or interfacial tension.

The Bashforth-Adams technique of measuring surface tension requires careful calibration of magnification because the result is sensitive to dimensional errors.



Surface tension is derived from drop shape. Given fluid density, the value of S_W / S_E is an entry into a lookup table which yields surface tension.

Figure 8. Pendant drop surface tension measured by the Bashforth-Adams Technique.

Dynamics

The four photos and the graph on this page show a 10µl drop of water falling a short distance (roughly 2mm) onto a Teflon fabric cloth. The first photo (Figure 9) shows the drop just before it detaches from the dispensing needle. Figure 10 shows the drop 1/60 of a second later, as it falls. Fast camera speed (short aperture time) freezes the motion of the drop. The next image, Figure 11, is the most interesting. It shows the extensive deformation of the drop as it impacts the sample. The last photo shows the drop restored to a more normal shape. The steady-state contact angle at the end of the movie is 116˚. Figure 13 plots the height of the drop. The ripple is the drop vibrating with a period of 57 milliseconds.

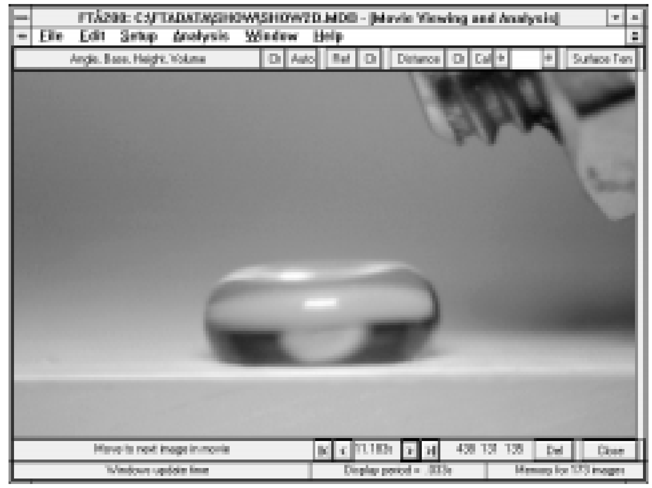


Figure 11. The drop impacting the sample.

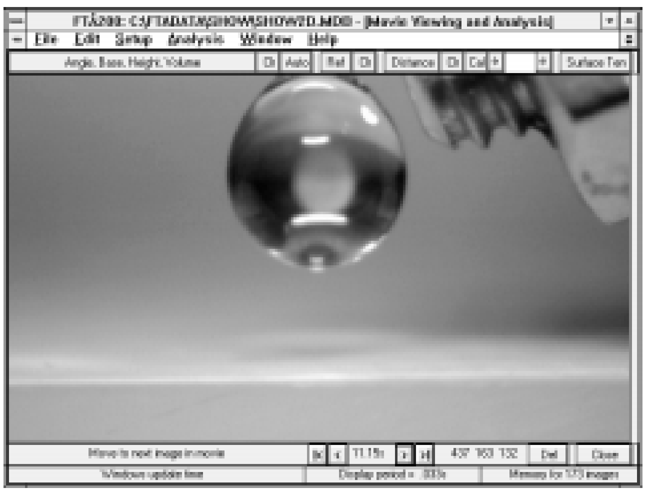


Figure 9. A drop of water about to detach..

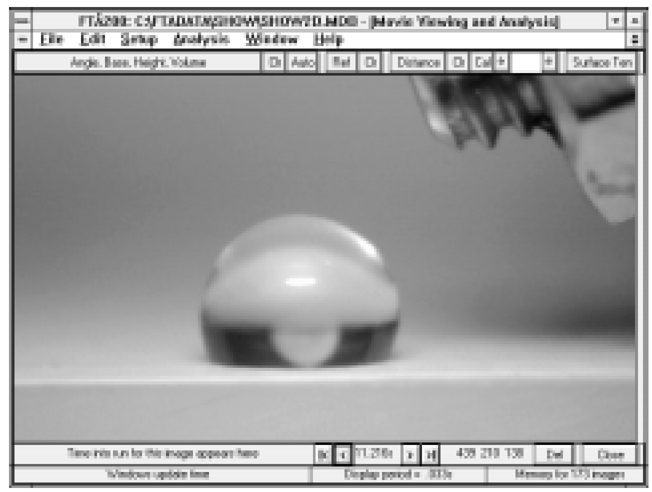


Figure 12. The drop at rest.

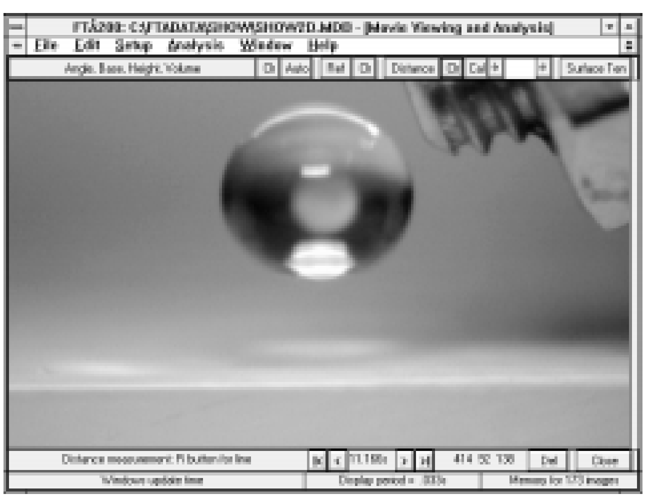


Figure 10. The drop after detaching from the needle.

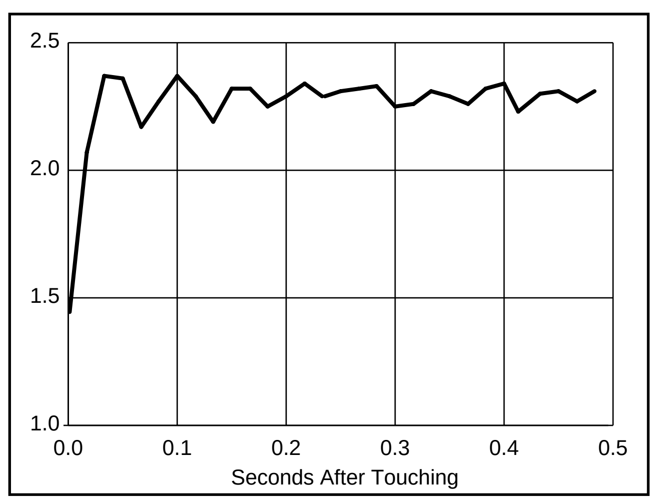


Figure 13. Drop height (mm) showing oscillation.

Absorption

The photos and graphs on this page and the next show water being absorbed by heavy kraft paper. This study was concerned with the effects of using different “sizings” which are used as fillers or glazes. Notice how the internally wetted portion of the paper just beneath the center of the drop can be seen to expand from Figure 17 through Figure 21.

The three graphs, Figures 22, 23 and 24, show the measured contact angle, the volume of liquid standing above the paper, and the width of the drop for the duration of the movie. The latter two measurements are sometimes used to describe absorption processes.

Figure 16.

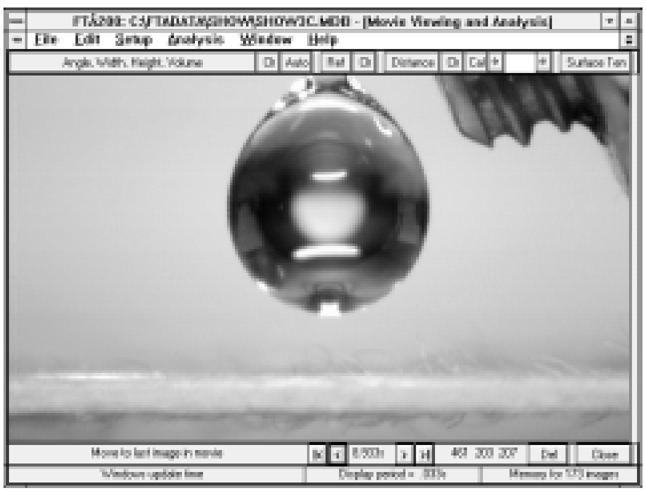


Figure 14.

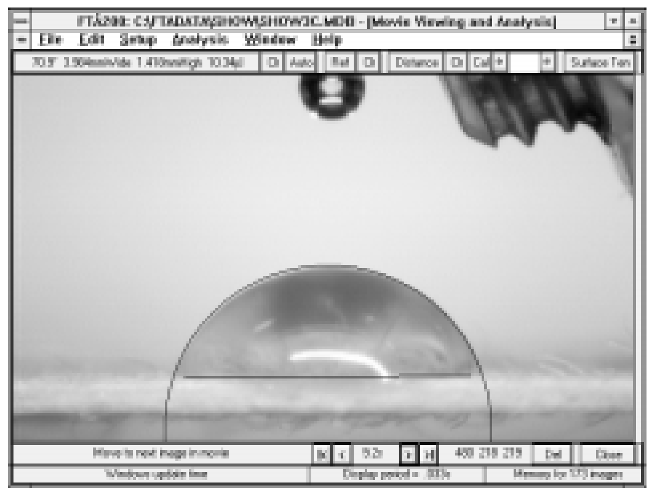


Figure 17.

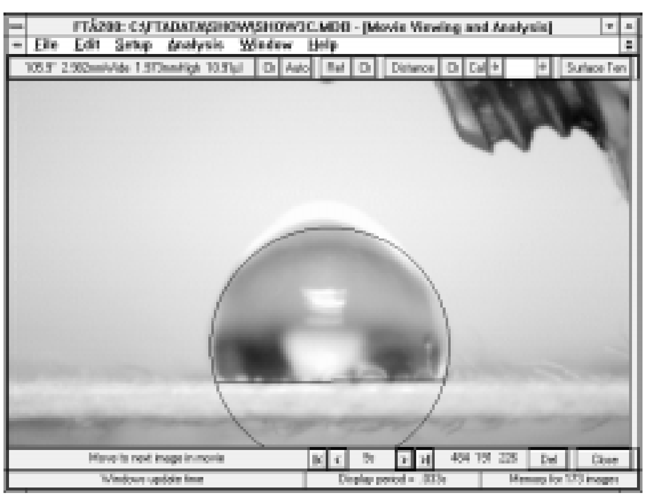


Figure 15.

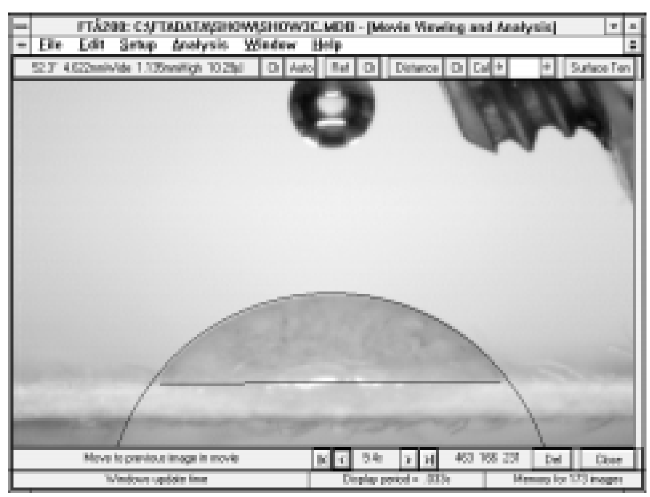


Figure 18.

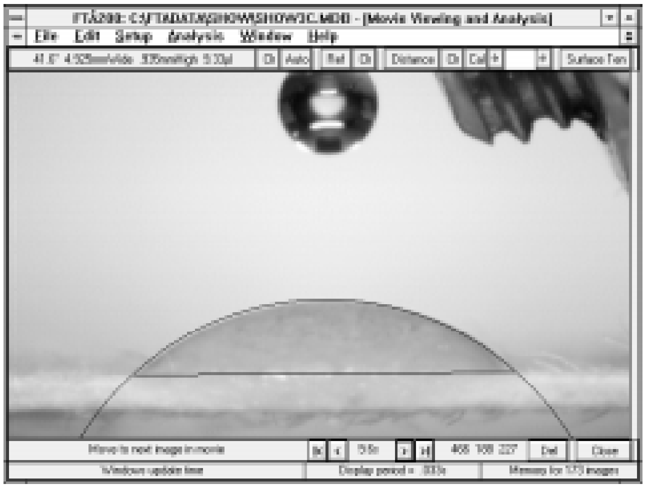


Figure 19.

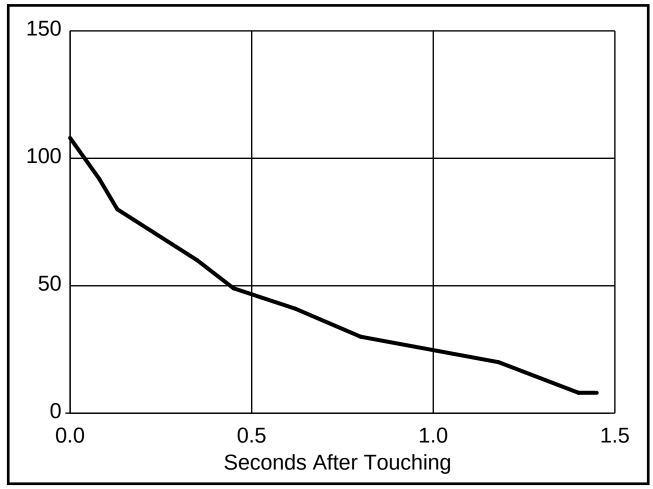


Figure 22. Contact angle (degrees).

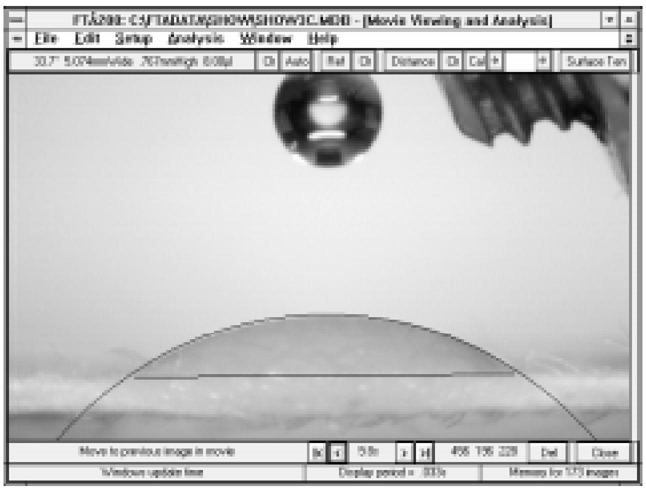


Figure 20.

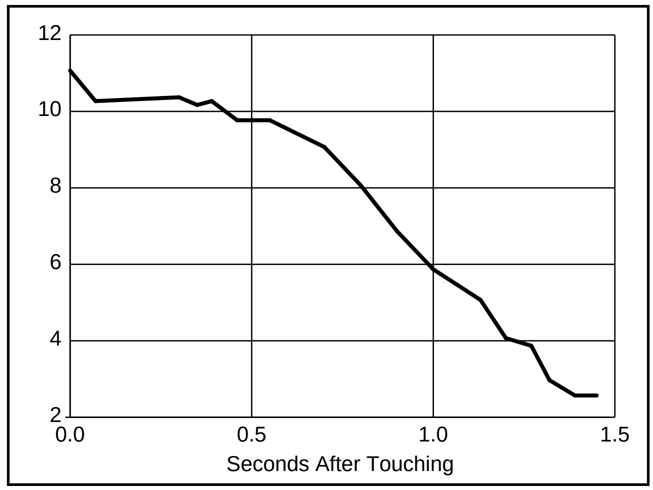


Figure 23. Standing volume (µl).

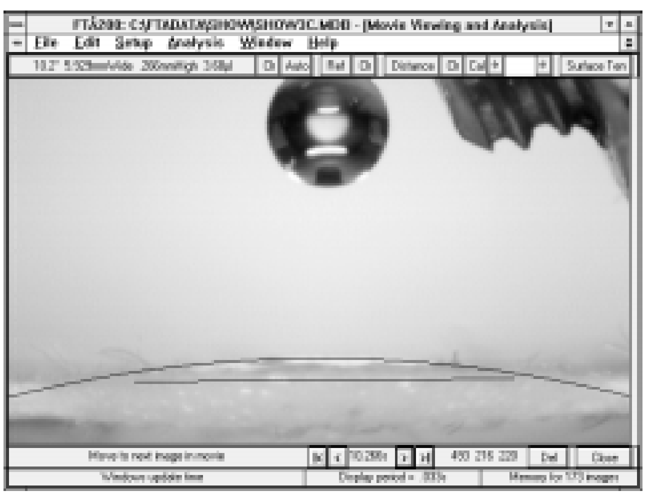


Figure 21.

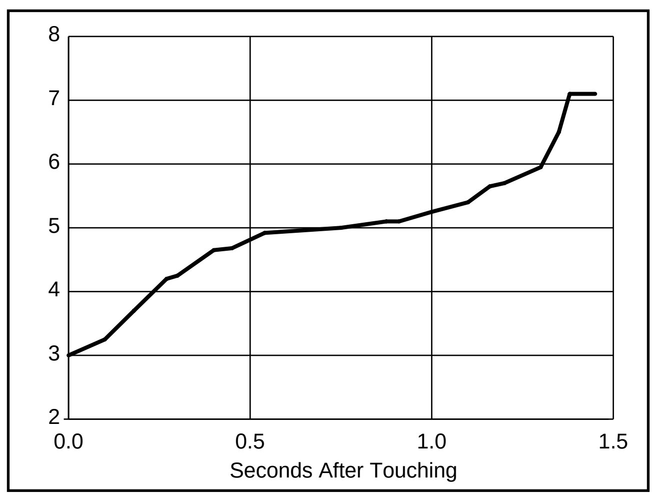


Figure 24. Drop base width (mm).

Surfactant Concentration

For a pharmaceutical application, the instrument was fitted with a robotic arm and precision syringe pump to automatically carry out dilutions and analyze surface tension as a function of time. After the drop is dispensed, surfactants in the diultion will preferentially migrate to the surface. As a consequence, surface tension will decrease in time. The kinetics of the migration can be studied by these surface tension/ time/dilution profiles.

The two graphs show surface tension for a solution of Tween-80, a simple detergent, and water. Figure 25 shows a period of almost three minutes. The movie rate was one frame per five seconds and the first frame was taken after 5 seconds. The surface tension at five seconds is 42.5 dynes/cm and it decreases to 38.1 dynes/cm at 175 seconds. The first image of this movie is shown in Figure 27, the last in Figure 28.

The same fluid was run a second time on a much shorter time scale (Figure 26). In order to capture the initial kinetics, it was necessary to dispense the drop very rapidly, so no significant migration took place before the pendant drop was formed. This resulted in some vibration and scatter in the data. The *initial* tension is higher in the second run because we are measuring much sooner than we did in the first run. These topics are examined in detail in “Practical Use of Concentration-Dependent Contact Angles as a Measure of Solid-Liquid Adsorption.” 1. Theoretical Aspects, by Erwin A. Vogler, *Langmuir*, **8**, 2005–2020 (1992).

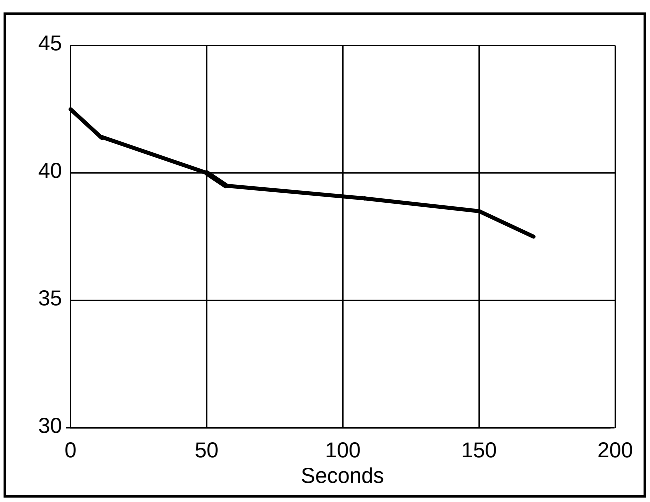


Figure 25. 0.4% Tween-80 surface tension.

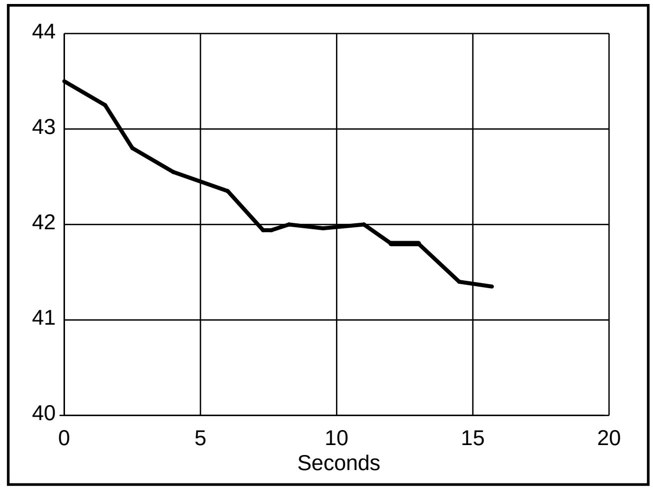


Figure 26. 0.4% Tween-80 surface tension.

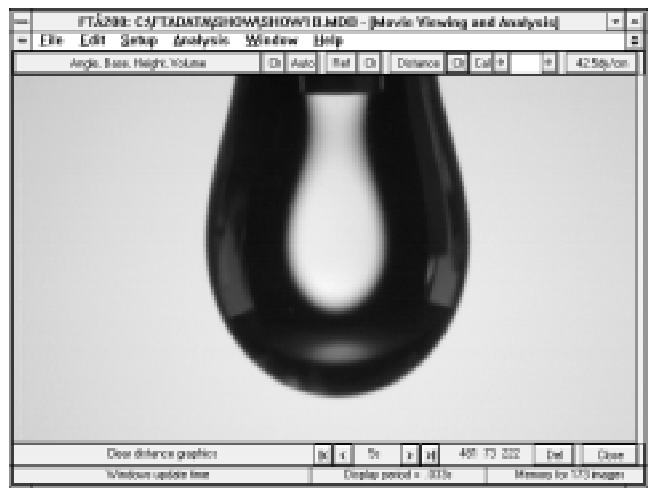


Figure 27. First image of first run.

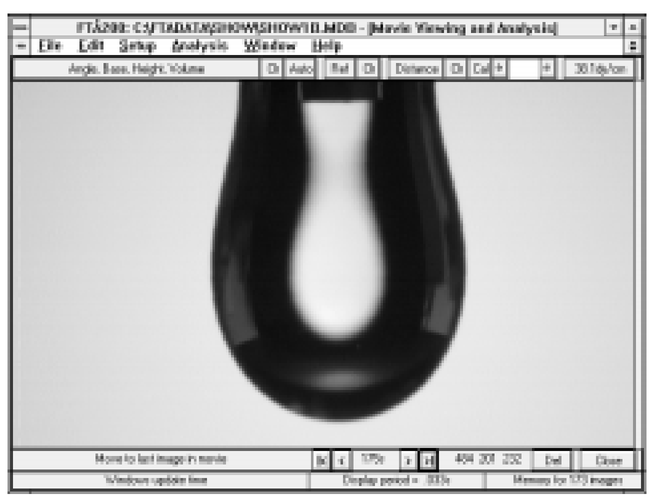


Figure 28. Last image of first run.