# An ultrasonic technique to study wetting and liquid penetration of paper

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**ABSTRACT:** *A new ultrasonic apparatus was designed to study wetting and liquid penetration of paper. The apparatus can measure both fast and slow transient changes in ultrasonic attenuation by a sheet of paper touched by a rising surface of test liquid pumped into the measuring cell. Tests with water showed the time dependence of attenuation, varying with different degrees of sizing of the paper sample. The fast change shows a good correlation with the Bristow absorption test and can be related to the wetting delay of the paper surface. The slow change was interpreted as caused by liquid penetration into the bulk pores of paper sheets.*

**KEYWORDS**
Absorption
Analysis
Instrumentation
Sizing
Ultrasonic attenuation
Wetting

In spite of the importance of wetting and liquid penetration into paper in coating and printing operations, our understanding of these phenomena is limited. A basic equation to describe the capillary absorption of liquid by porous material was derived by Lucas and Washburn (*1*). Unfortunately, the complexity of the sheet structure and its change after wetting with liquids make quantitative analysis difficult. Therefore, it is important to establish experimental methods to evaluate the wetting and liquid penetration of paper.

Several techniques have been proposed for measuring the wetting and absorption properties of paper (*2*). Bristow's absorption tester seems to be the most widely used. In this test, a paper strip mounted on a rotating wheel is moved at a constant speed under a contacting headbox containing the test liquid. The length of the band of applied liquid gives the information about the wetting time and the penetration rate. With this apparatus, Bristow (*3*) and, more recently, Lyne and Aspler (*4*) showed that there is a time delay in the wetting of paper with a polar liquid such as water. This delay is called "wetting time" or "wetting delay," and it ranges from about 20 ms for typical newsprints to a few seconds for sized papers (*3, 5–7*). This phenomenon and the successive capillary absorption of liquid are important in coating and printing operations, where coating colors or inks are applied to paper at high speeds.

Optical techniques (*8–10*) measure the rate of change in the reflectance of paper as liquid penetrates it in the *z*-direction. Recently, Anderson and Higgins reported that this method can detect rapid phenomena properly measured in milliseconds (*11*). Another advantage of this technique is that the measured reflectance can be converted into an indication of the depth of penetration by using the Kubelka-Munk equation. However, the technique suffers from the effect of the color of paper and the considerable variation in the measurement at the beginning of penetration. To minimize the uncertainty, it is desirable to have the value of the specific light scattering coefficient of the penetrant as small as possible.

There are several other methods to evaluate liquid penetration by continuously measuring some physical property of paper, such as electrical conductivity (*5, 12*), ultrasonic velocity (*13, 14*), infrared absorption (*15*), or surface polarity by the IGT test (*16*).

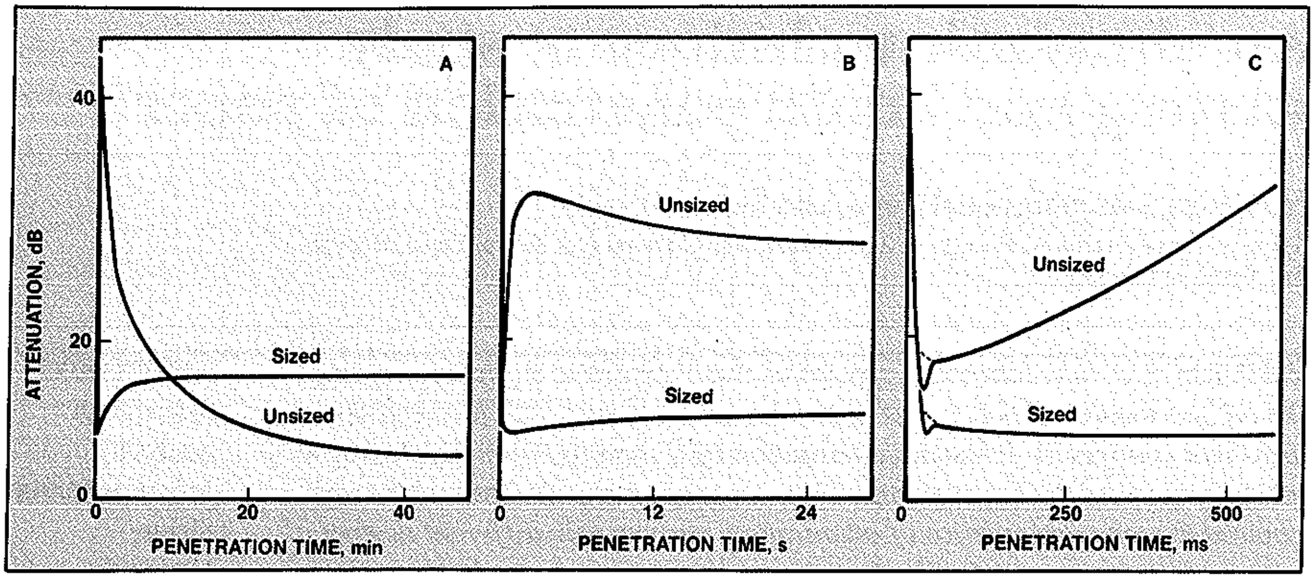
In a previous paper, we presented an apparatus to measure the time dependence of ultrasonic attenuation by paper vertically immersed in the test liquid (*17*). We introduce here a new ultrasonic apparatus with a horizontal sample arrangement. This new design makes it easier to reproduce the initial contact of paper and liquid, thus allowing more detailed studies of the fast transient process in the wetting of paper.

## Results and discussion

**Figure 1** shows the time dependence of acoustic attenuation represented on three different time scales for typical sized and unsized papers. The time scale is magnified from left to right by a ratio of 1:100:4800. The long-time profiles (in minutes) shown in Fig. 1A are the same as those obtained in the previous study. The sized paper shows a moderate increase followed by a plateau, and the unsized paper shows an exponential-type decrease.

The curves in Fig. 1B show short-term profiles of the initial part of the curves in Fig. 1A. The curve for unsized paper in B reveals the existence of a rising stage in attenuation shortly after wetting. On the other

1. Typical time dependence of acoustic attenuation represented on three different time scales for sized and unsized papers.

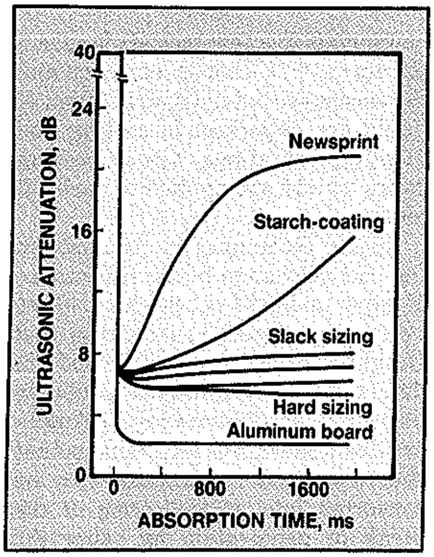



hand, the initial increase in attenuation with the sized paper is almost eliminated. By expanding the time scale further, a sharp drop in attenuation after wetting was found with both samples (Fig. 1C). Because this part of the attenuation curve was independent of the type of the test specimen (Fig. 2), it corresponds with results from the instrumental response to the initial contact of liquid with the paper surface.

As discussed earlier (*17*), the long-term decrease in unsized paper (Fig. 1A) corresponds with the process of liquid penetration into the paper, whereas the long-lasting high attenuation value in the sized paper is apparently caused by the retention of air in the test sheet of sized paper. Therefore, the close resemblances between the profiles of sized paper in A and the unsized paper in B and the profiles of sized paper in B and unsized paper in C are interesting. If these resemblances result from the similarity in the microscopic physical processes involved, the effect of sizing is to slow down these processes by a factor of, say, one hundred for the present case.

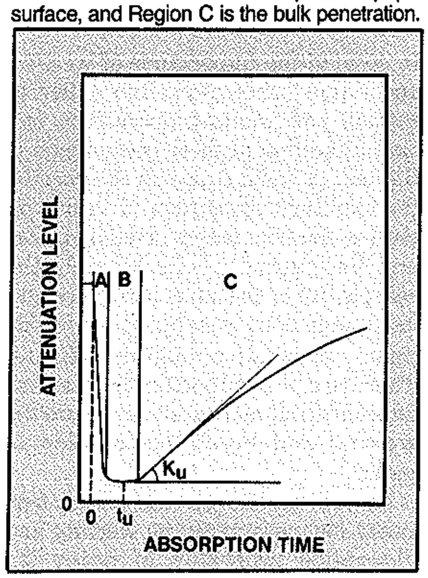
**Figure 2** shows the rapid change in attenuation after wetting for various papers. The pattern was characteristic of the nature of the paper tested. The profile is lowered and more leveled with an increase in sizing. To find some indices to describe these profiles and try to relate them to conventional quantities used to describe the wetting properties of paper, a master curve for the attenuation–time profile can be drawn schematically.

2. Time dependence of acoustic attenuation of commercial papers at different amounts of sizing




This master curve, as shown in **Fig. 3**, is divided into three parts, A, B, and C. The first part, A, is likely to be the instrumental response. The last part, C, results from the liquid penetration process into the bulk pores of paper. Therefore, the middle part, B, reflects the process of transient interaction between the liquid and the paper surface. This part of the profile is characterized by a shallow minimum followed by a remarkably steep rise. We chose a characteristic time, $t_u$, which is the time for the minimum attenuation, and the maximum rate of increase in attenuation, $K_u$, determined at the point of inflection.

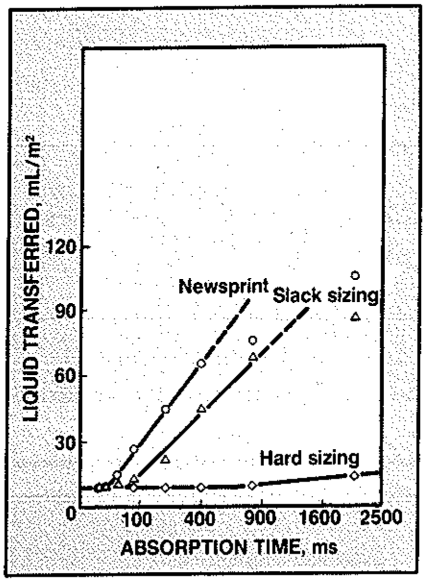
3. In a master curve of the time dependence of acoustic attenuation, Region A represents the instrumental response, Region B is the transient interaction between liquid and paper surface, and Region C is the bulk penetration.



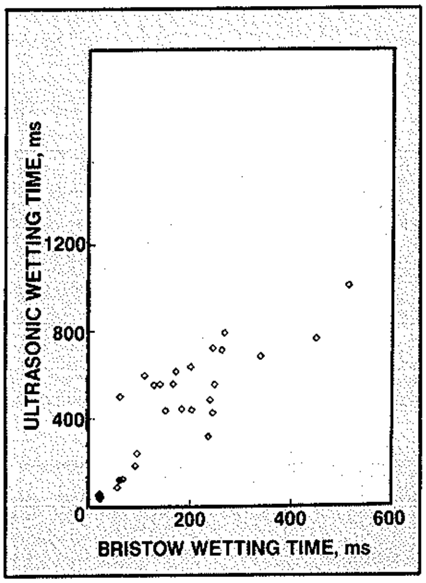

A well-established test for the wetting of paper is the Bristow test, which determines a set of quantities to characterize the paper (*3, 4*):

$K_r$ = the roughness index

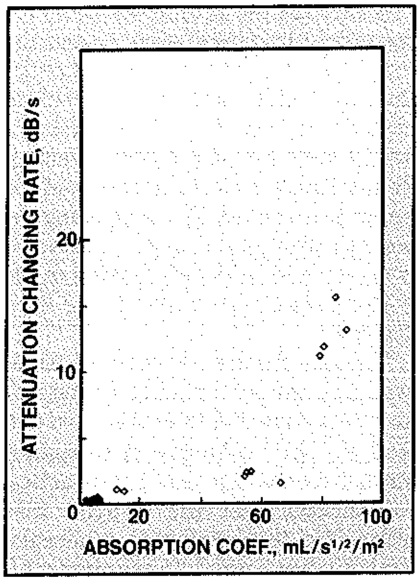
**4.** Results of the Bristow test, where the initial flat part is the wetting time, $t_w$, and the absorption coefficient, $K_a$, is determined from the slope of the graph



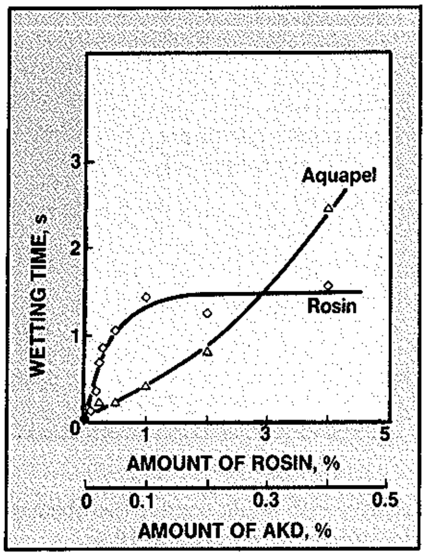

**5.** Correlation between the wetting time determined by the Bristow test and by the ultrasonic technique, where the correlation coefficient is 0.87, the proportionality constant is 1.83



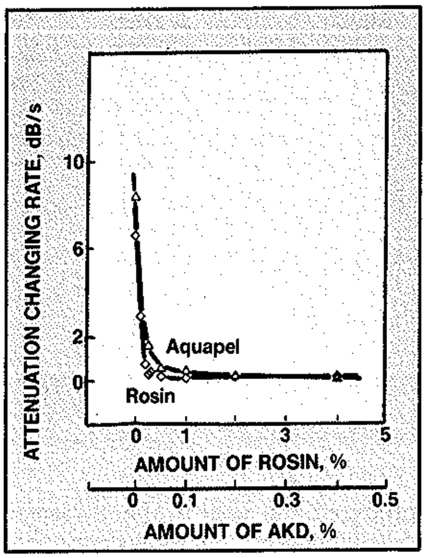

**6.** Correlation between the absorption coefficient by the Bristow test and by the attenuation changing rate




**7.** Ultrasonic wetting time of handsheets as a function of addition of AKD or rosin




**8.** Ultrasonic attenuation changing rates of handsheets as a function of addition of AKD or rosin




$K_a$ = the absorption coefficient

$t_w$ = the wetting time.

**Figure 4** shows the results of this test for some of our samples. Here, $K_r$ is determined as the intercept of the graph at $t = 0$, $t_w$ as the length of time of the initial flat part, and $K_a$ as the slope of the graph beyond $t_w$. Comparing Figs. 2 and 3, we find a striking similarity in the curve shape and its dependence on the type of paper. This leads us to correlate $t_u$ with $t_w$ and $K_u$ with $K_a$, respectively.

**Figure 5** shows the plot of $t_u$ vs. $t_w$ for the same paper samples. Though there are some scattered points, a simple linear correlation between $t_u$ and $t_w$ is evident. Linear regression analysis gave a correlation coefficient of 0.87 and a slope of the line of 1.83. Thus, $t_u$ is closely related to $t_w$ and can be used to estimate $t_w$ by dividing it by a factor of 1.83. In other words, $t_u$/1.83 can be interpreted as the "ultrasonic wetting time."

On the other hand, the plot of $K_u$ vs. $K_a$ (**Fig. 6**) seems to be split into two parts with different slopes, and $K_u$ cannot be interpreted as straightforwardly as $t_u$. Nonetheless, an overall positive correlation between $K_u$ and $K_r$ is evident, indicating that $K_u$ can be used as an index for absorptivity.

**Figures 7 and 8** show the dependence of $t_u$ and $K_u$, respectively, on the amount of sizing agent, measured with two series of handsheets prepared by acidic (rosin) and alkaline sizing (alkyl ketene dimer, or AKD). Both $t_u$ and $K_u$ were characteristically influenced by the amount of sizing agent for either type. The curve shape for $t_u$ is somewhat different with the acidic and alkaline-sized papers, implying that there is a clear difference in the hydrophobicity of the two kinds of sizing agents. In consideration of the addition amount, AKD is much more effective than rosin, as is commonly known.

On the other hand, the curve profiles of $K_u$ for the two types are similar to each other. Apparently, in both cases, $K_u$ is strongly influenced by small amounts of sizing agents. Thus, the measurement is useful in assessing the sizing effect, especially at low concentrations of sizing.

## Conclusions

A new design of ultrasonic apparatus indicates the rapid transient phenomena after wetting of paper surfaces, as well as the slow processes following these. The attenuation-time profiles obtained could be analyzed in terms of a single master curve. On this curve, two indices were defined to characterize the profile: the time for the minimum attenuation, $t_u$, and the maximum rate of increase in attenuation, $K_u$. These indices for a series of commercial papers and handsheets with widely varying degrees of sizing do sensitively reflect the nature of the paper and are useful in evaluating the wetting property of paper. In particular, $t_u$ could be closely related to the wetting time determined by the Bristow test.

Though further experimental as well as theoretical studies are needed to substantiate the interpretation of $t_u$ and $K_u$, this technique of ultrasonic measurement has several practical advantages over the Bristow test. First, the test procedure is simpler and quicker; second, the equipment is smaller and less expensive; and third, a smaller test piece is required. This third point is important when one wants to apply the technique to handsheets, which usually have limited sizes that are not large enough for the Bristow test.

## Experimental procedures

### Apparatus

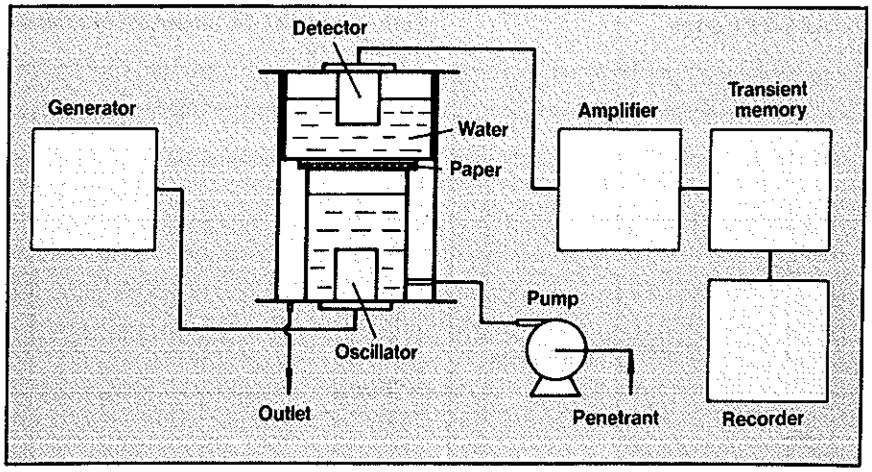
**Figure 9** schematically shows the apparatus for ultrasonic measurement, consisting of the upper (receiver) unit and the lower (generator) unit. The most important feature of this design is that the ultrasonic wave irradiates the horizontally held paper specimen through a liquid layer which is brought into contact with a dry specimen from below. The test liquid is pumped into the lower unit until it just touches the paper surface.

A pair of 2-MHz piezoelectric oscillators of 2.5 cm diameter were used as generator and receiver. The receiver unit was placed on top of the generator unit and was always filled with water to transmit ultrasonic waves. The test piece (4 cm × 4 cm) was attached to the outer bottom of the receiver unit with a piece of double sticking tape as large as the test piece.

### Test sheets

Paper samples of various degrees of sizing were prepared with both alkaline-sizing and rosin sizing, by changing the amount of sizing agent used (*17*). Ten or more test pieces (4 cm × 4 cm) of each sample were prepared and used in the ultrasonic measurement. All measurements were carried out at 20°C and a relative humidity of 65%.

**9.** Schematic diagram of the apparatus for the ultrasonic measurement of wetting and liquid penetration of paper




**I.** Commercial paper samples

| Sample | Basis weight, g/m² | Cobb degree, mL/m² | Description of samples |
|---|---|---|---|
| Wood-free paper | 59.1 | 16.72 | Rosin-sized |
| | 58.0 | 14.73 | Rosin-sized |
| | 69.9 | 17.94 | AKD-sized (hard sizing) |
| | 67.5 | . . . | AKD-sized (slack sizing) |
| | 68.3 | . . . | Starch-coating unsized |
| Wood-containing paper | 76.5 | 23.15 | Rosin-sized |
| | 53.1 | 20.35 | Rosin-sized |
| | 60.7 | 19.52 | Rosin-sized |
| | 62.5 | 16.81 | Rosin-sized |
| | 63.2 | 33.93[a] | Rosin-sized (slack sizing) |
| | 63.5 | 17.93[a] | Rosin-sized (slack sizing) |
| Newsprint | 51.1 | . . .[b] | Newsprint |
| | 43.5 | . . .[b] | Aged newsprint |
| Handsheet | 59.1 | 17.20 | AKD-sized |
| | 59.1 | 15.78 | Rosin-sized |
| PPC Paper | 60.3 | 19.62 | |

[a]Cobb-sizing degree measured at absorption time of 10 s.
[b]Cobb-sizing degree could not be measured because of excessively rapid water penetration.

### Bristow test

The test was carried out according to Bristow (*3*). Because the test requires a large test piece (2.5 cm × 100 cm), only commercial papers and some pilot machine papers could be used. They are listed in **Table I** together with their characteristics.

### Cobb test

The measurement of Cobb sizing degree was carried out by following TAPPI method T441 om-84. For the papers with slack sizing, the absorption time was reduced to 10 s.

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