

A new method for determining the relative bonded area

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ABSTRACT: Correlations of scattering coefficient with sheet tensile strength and with sheet density were examined to see if they could be used to determine the scattering coefficient of an unbonded sheet, from which the relative bonded area (RBA) could then be calculated. The data showed that neither correlation was useful because the fiber cross-sectional shape in the sheet changes with the extent of wet pressing. A new method has been developed in which the scattering coefficient and sheet density are corrected for changes in the fiber cross-sectional shape. With the corrected data, the scattering coefficient was linearly related to sheet density, from which a simple new formula was derived for calculating the RBA. The total area available for bonding was not constant, so the RBA alone is not sufficient to describe fully the bonding in the sheet.

Application: A new method has been developed for calculating the relative bonded area from sheet density and fiber cross-sectional shape.

The relative bonded area (RBA) is the fraction of the total available fiber surface that is bonded. It is a quantity that is applied in theories of paper mechanical properties. The relative bonded area is defined as equal to $(A_T - A)/A_T$, where A_T is the total area available for bonding and A is the unbonded area in the sheet after it has been formed.

To make up for shortcomings in the two methods commonly used to calculate the relative bonded area, we have developed a new method based on a correlation between scattering coefficient and sheet density. In our method, however, the measured values for scattering coefficient and sheet density are corrected for changes in the cross-sectional shape of the fibers.

BACKGROUND

Two methods are generally used to measure the RBA, the nitrogen-adsorption method of Brunauer, Emmett, and Teller (BET) [1] and the light-scattering method of Ingmanson and Thode [2]. The BET method involves directly measuring the free fiber surface available for adsorption, which is denoted as A_f . The total area available can be determined by spray-drying fibers to produce dried fibers with minimal bonding and then applying the nitrogen-adsorption method.

In the light-scattering method, it is assumed that $S = c A_s$, where S is the light-scattering coefficient of a sheet, c is a coefficient of proportionality that depends both on the wavelength and on the fiber properties, and A_s is the total surface area available for scattering. The sur-

face area available for scattering is always less than that available for nitrogen adsorption [3]. If S_0 is the total scattering coefficient for a completely unbonded sheet, then $RBA = (S_0 - S)/S_0$.

A major difficulty with this technique lies in the measurement of S_0 , since an unbonded sheet cannot be prepared. In their original work, Ingmanson and Thode solved the problem by measuring the scattering coefficient as a function of tensile strength for a series of sheets made from the same pulp. The strength of the sheets was varied by refining with a ball mill and by wet pressing. All of the sheets measured in this way collapsed onto a single curve. The y -axis intercept, corresponding to zero-tensile strength, was then taken as S_0 .

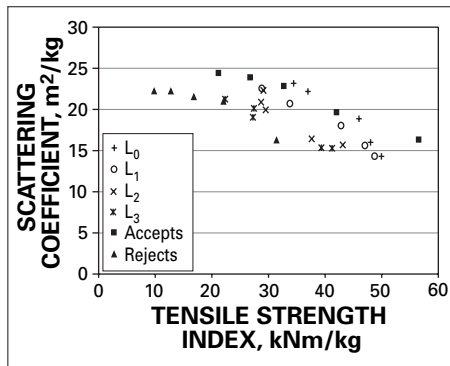
Generally, the light-scattering method has been preferred over the nitrogen adsorption method because it is much quicker. However, the method presents many difficulties. The curve for scattering coefficient vs. tensile strength found by Ingmanson and Thode was nonlinear, and it flattens out at very low strengths [2]. Ingmanson and Thode obtained an accurate extrapolation because they produced sheets with strengths ranging from 5 lb/in. to 26 lb/in. The critical value determining the accuracy of the extrapolation is the lowest strength that can be obtained, which corresponds to an unrefined pulp and unpressed sheet. For any pulp being tested, the accuracy of the extrapolation will be questionable if the unrefined, unpressed sheets already have significant strength.

Niskanen has also criticized this method on the grounds that the tensile strength will fall to zero long before bonding completely disappears in the network [4]. Furthermore, the method is completely unsuitable for measuring bonding in machine-made papers since different sheets cannot be prepared over a range of tensile strengths.

Other researchers have examined the correlation between scattering coefficient, S , and sheet density, ρ_a . Two different trends emerge. If the density was changed by refining while the pressing pressure was kept constant, then a linear relationship of the form $S = -A \rho_a + B$ was generally found, where A and B are both positive for chemical pulps [5-9]. However, when the density is changed by wet pressing alone, then the relationship between S and ρ_a is nonlinear, with the slope of the data falling as the density decreases and with S approaching S_0 asymptotically as ρ_a is reduced [4, 6, 10]. In contradiction to these results, El-Hosseiny and Abson have claimed that varying the wet pressing pressure will also produce a linear relationship between S and ρ_a [5]. However our analysis of the data on which this claim is based [11] indicates that this linear relationship may hold true only over a limited range of density.

Whatever the relationship between scattering coefficient and sheet density, no method for calculating the RBA from sheet density has found widespread acceptance. However, a correlation with density is still more reasonable than a correlation with tensile strength, since the

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1. Correlation of scattering coefficient with tensile strength for the six sets of sheets tested.

density is a direct measure of the compaction of the sheet and the interaction between tensile strength and bonded area is much more complex [12].

In this work, we tested correlations of scattering coefficient with tensile strength and with density for a set of data obtained from sheets made from a never-dried, unbleached kraft pulp from radiata pine. We found neither correlation to be correct. Therefore, we developed and tested a new method for calculating the RBA by correcting the measured scattering coefficient and sheet density for fiber cross-sectional shape.

EXPERIMENTAL METHODS

Data were collected for a series of pulps generated from a single starting stock of an unbleached kraft pulp, kappa no. 30, made in the laboratory from radiata pine [13]. Handsheets were also made from the accepts and rejects produced by double hydrocyclone fractionation of this starting material furnish. We also varied the fiber length of the starting stock by cutting wet handsheets. Three sets of handsheets were produced from wet cutting. The procedure was to form the starting stock into handsheets and then to cut the wet handsheets with a die.

We varied the number of cuts to give each set a different length-weighted fiber length. The length-weighted fiber lengths of the three sets were 2.53 mm, 2.10 mm, and 1.80 mm, whereas the fiber length for the starting stock was 3.14 mm. The starting stock was labelled L_0 , and the other three sets were labelled L_1 , L_2 , and L_3 , respectively. The cutting process reduced the fiber length without affecting the other sample dimensions.

For each of the pulps, five sets of handsheets were made at different press-

ing levels. Changing the pressing level varied the degree of bonding, the tensile strength, and the scattering coefficient. Further details of the experiments are given elsewhere [13, 14].

Fiber shape and fiber dimensions were measured on the fibers in the sheet cross sections with a combination of resin embedding and confocal microscopy [13–15]. We calculated the dimensions of the fibers in the sheet by fitting a rectangular bounding box around each measured fiber, by calculating the width and height of the bounding box, D_w and D_h , and by calculating the fraction of the bounding box filled with fiber, f .

Tensile strength and sheet density were tested by the relevant ISO standards. The scattering coefficients were measured at 700 nm.

EXTRAPOLATION METHODS

Scattering coefficient vs. tensile strength

Figure 1 plots the scattering coefficient against tensile strength. Six data sets are shown, one for sheets made from the starting pulp, three made after cutting the starting pulp to reduce fiber length, and two made by fractionating the starting pulp into accepts and rejects.

The scattering coefficient measurements have also been presented elsewhere [13]. In that research, the same six sets of sheets were examined in light of nitrogen-adsorption measurements to determine the total surface area in the sheets. We also measured the surface area of unbonded sheets, which we prepared by spray drying very dilute suspensions of fibers onto Teflon and allowing them to dry. The measured surface areas of unbonded sheets for samples L_0 , L_1 , L_2 , and L_3 were found to be 905, 920, 921, and 927 m^2/kg , respectively. For the accepts and the rejects, the surface areas measured were 993 m^2/kg and 1063 m^2/kg , respectively [13]. Thus there is only a 15% difference between largest and smallest total surface area, as determined by nitrogen adsorption, which indicates that the values determined for the different pulps should be quite close to each other.

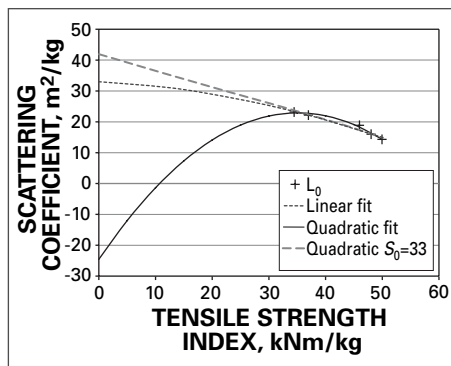
In Fig. 1, the data sets are quite scattered, although each data set shows a clear trend of the scattering coefficient decreasing as tensile strength increases.

Initially we attempted a straight line fit to each data set. While the R^2 statistics for the fits were reasonably good, ranging from 0.87 for the rejects data set to 0.98 for the accepts, the calculated values of S_0 were well spread, ranging from 25.7 m^2/kg for the rejects to 42.2 m^2/kg for the L_0 pulp.

Furthermore, linear fits of the data from the fractions L_0 – L_3 have produced scattering coefficients of 42.2, 34.1, 33.1, and 28.8 m^2/kg , respectively. The fits to the L_0 – L_3 data sets have been presented previously [13]. The cutting process used to generate these fractions will only have created new fiber ends, which would not contribute significantly to scattering. The fact that the total unbonded surface area measured by nitrogen adsorption increased only slightly as the fiber length was reduced further indicates that S_0 should be constant for these data sets. However, a constant value for scattering coefficient is definitely not observed in Fig. 1.

In addition, the relationship between scattering coefficient and tensile strength is not linear, as Fig. 1 shows if values are examined over a large enough range. This lack of linearity is clearly evident with the rejects data, which shows the lowest strength of any of the data sets. The unpressed sheet made of the rejects pulp has a strength of only 9.8 $kN\cdot m/kg$, and the scattering coefficient decreases only a little between tensile strengths of 9.8 $kN\cdot m/kg$ and 22 $kN\cdot m/kg$. It is not until the last two points in the set, when the tensile strength increases from 22 $kN\cdot m/kg$ to 31.4 $kN\cdot m/kg$, that the scattering coefficient begins to decrease in a manner seen in the curves of some of the other data sets.

Similar nonlinear behavior can be seen in the original data of Ingmanson and Thode [2]. A nonlinear relationship can make it impossible to determine S_0 with any accuracy. An example of this difficulty is shown in Fig. 2, which shows three attempts at fitting the L_0 data set. The three fitting functions attempted on the data set were a linear function, a quadratic function, and a quadratic function with S_0 set at 33 m^2/kg . This value had been estimated previously from the linear relationship between scattering coefficient and the nitrogen adsorption area [13].



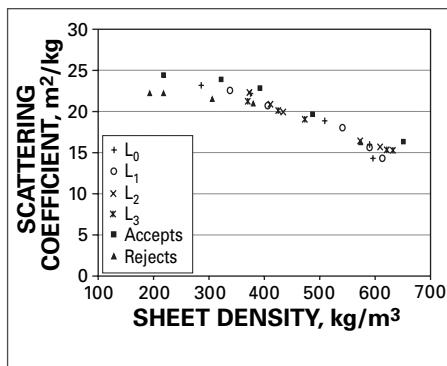
2. Three attempts to fit the L_0 data set to determine the y-axis intercept.

The R^2 values for the three fits in Fig. 2 were 0.9431, 0.9848, and 0.9538, respectively. However, the values determined from the linear and quadratic fits were $42.0 \text{ m}^2/\text{kg}$ and $-24.5 \text{ m}^2/\text{kg}$, respectively, even though there is actually little difference in terms of the fit of any of the three functions across the range of the data. The problem is that the minimum strength seen in the data set, which was obtained for unpressed sheets, was still $34.45 \text{ kN}\cdot\text{m}/\text{kg}$, and it is impossible to extrapolate accurately from this minimum strength to a tensile strength of zero.

Scattering coefficient. vs. sheet density

Figure 3 shows the correlation of the scattering coefficient with sheet density. The data nearly collapse onto a single curve, with the accepts being slightly above the data for L_0 to L_3 , while the rejects are slightly below. This correlation is far more consistent with the measurements of nitrogen adsorption, which showed only small differences in the total surface area of the unbonded samples from the different data sets [13]. As Fig. 3 also shows, the correlation between scattering coefficient and density is not linear. The general shape of the curve is consistent with previous results, where the density was varied by wet pressing [4, 6, 10].

To estimate RBA values from these data, we have to define a minimum density, ρ_{min} , which corresponds to S_0 . The data also suggest that scattering coefficient is approximately constant at around $25 \text{ m}^2/\text{kg}$ for densities up to $300 \text{ kg}/\text{m}^3$. If this value were selected as S_0 , then it would yield an RBA value of approximately 0 for all sheet densities up to this limit, which is obviously incorrect, given that all of these sheets have some level of tensile strength and bonding.



3. Correlation of scattering coefficient with sheet density, ρ_s , for the six sets of sheets tested.

Light-scattering from a fiber mass

Neither the plot of scattering coefficient vs. tensile strength nor that of scattering coefficient vs. density can be applied to calculate the relative bonded area for the data presented here. To examine why this is the case, let us consider the light scattering from a unit mass of fibers.

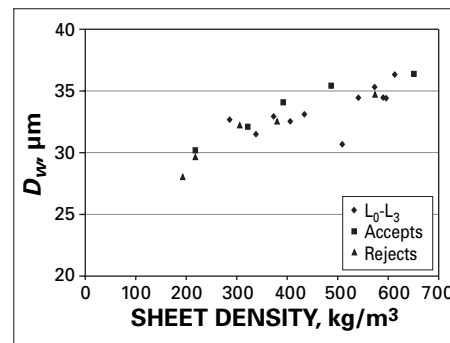
The fibers are largely lying flat in the sheet with the fiber width sitting approximately parallel to the sheet surfaces [14]. The reflectivity at the interface of the air and the fiber wall strongly depends on the angle between the interface and the incoming radiation. The reflectivity is highest if the interface is perpendicular to the light, and it falls away rapidly as the angle decreases under 90° [16]. Thus what is important for light scattering is the width of the fibers, rather than the perimeter, since the sides of the fibers will have little light-scattering capacity.

Thus if the fibers have the dimensions of width D_w , height D_b , fiber wall area A_w , and fiber length L , and if we assume that the lumen area does not contribute to scattering, then the total surface area available for scattering is $(2 D_w L)/(A_w L \rho_w)$, where ρ_w is the fiber wall density. The scattering from the lumen is ignored. Even for the most lightly pressed sample we tested, two-thirds of the fibers were either fully or partially collapsed [14], and the lumen surface areas of the uncollapsed fibers are smaller than the outside surface areas.

From this expression, the total light-scattering available from these fibers in the absence of bonding is given by

$$S_0 = c2D_w/(A_w\rho_w) \quad (1)$$

where c is a constant of proportionality.



4. Plot of fiber width, D_w , against sheet density.

Thus S_0 is constant only if the fiber width and fiber wall area are constant.

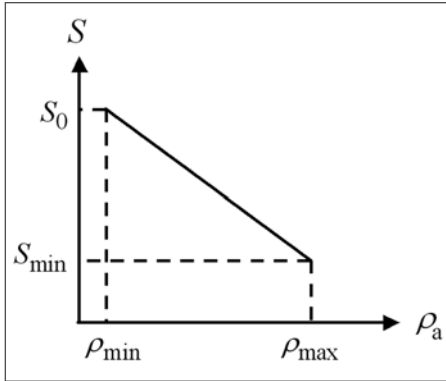
For our experiments, the measured average fiber wall areas for the cut fibers, accepts, and rejects were $201 \mu\text{m}^2$, $194 \mu\text{m}^2$, and $221 \mu\text{m}^2$, respectively, and were not affected by either the degree of wet pressing or the level of fiber cutting. The fiber width data are plotted against sheet density in Fig. 4. Three data series are shown, one for the accepts, one for the rejects, and one for sheets made from the pulps with different fiber lengths (L_0 - L_3). This last data set has only 11 points because the shape of the fibers in the sheets was not measured for all of the 20 sheets in this data set. As Fig. 4 shows for each data set, D_w increases by around 15% across the range of sheet density.

Thus for our experiments, no simple extrapolation of scattering coefficient based on a correlation with tensile strength can be meaningful, because sheets of different density will have different values of S_0 . In other words, if it were somehow possible to take a sheet and separate the fibers so that there was no bonding, while the fibers still retained their positions, the measured scattering coefficient would be equal to S_0 and would differ from sheet to sheet. No simple extrapolation based on a correlation between scattering coefficient and tensile strength could be applied because a single value of S_0 would not exist. Therefore a method needs to be found to correct the measured scattering coefficients before the RBA value can be calculated.

A NEW METHOD FOR DETERMINING RBA

We start by assuming that the light-scattering coefficient, S , is linearly related to sheet density, ρ_s , as shown in Fig. 5, provided that the fiber cross-sectional shape

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5. Assumed linear relationship between scattering coefficient and density.

is constant. This figure shows two sets of points. One set is a minimum density, ρ_{min} , which corresponds to a completely unbonded sheet with scattering coefficient S_0 .

The second set of points is the maximum attainable density, ρ_{max} , which gives the density of the sheet when the fibers are closely and perfectly packed. The corresponding scattering coefficient is S_{min} , which is not zero. It cannot be zero because the irregular shape of the fibers will ensure that fibers are not necessarily in contact with each other across their entire width even if the fibers are closely and perfectly packed. In addition, light will always scatter at the surfaces of the sheet, and such surface scattering is also included in S_{min} .

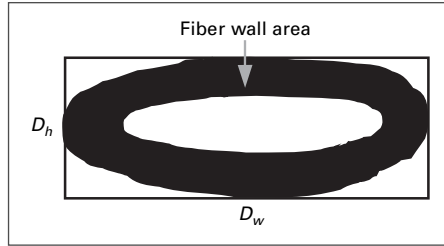
Derivation

From Fig. 5, we see that S is related to ρ_a by Eq. 2:

$$S = S_0 - \left(\frac{S_0 - S_{min}}{\rho_{max} - \rho_{min}} \right) (\rho_a - \rho_{min}) \quad (2)$$

It is thus necessary to determine S_0 , S_{min} , ρ_{max} , and ρ_{min} in order to calculate S and RBA as a function of sheet density. The derivations of these quantities are given below.

Following our previous work [13–15], we define a fill factor, f , for a fiber as the cross-sectional area of the fiber divided by the area, $D_h \times D_w$, of the smallest rectangular bounding box that can surround the irregular shape of the fiber. The concept is illustrated in Fig. 6, which shows that f is related to the degree of collapse of the fibers in the sheet, with a higher value of f corresponding to a greater degree of collapse. The overall density of the bounding box is $f\rho_w$, where ρ_w is the density of cell-wall material.



6. The bounding box surrounding a model fiber.

The maximum density, ρ_{max} , will occur when all fibers are lying flat, parallel, and stacked perfectly on top of one another, such that there are no spaces between the bounding boxes surrounding the fibers. By definition, then, we have Eq. 3:

$$\rho_{max} = f\rho_w \quad (3)$$

Figure 7 shows the variation of f with sheet apparent density for the accepts, for the rejects, and for a combined set of the sheets in the L_0 to L_3 series. For all data sets, f increases with sheet density. The measured values of f ranged from 0.428 to 0.577, which correspond to ρ_{max} values of 642 kg/m³ and 866 kg/m³, respectively. The value of f increases with increasing wet pressing and sheet density because pressing causes the fibers to collapse and flatten out.

We have no rigorous method for determining ρ_{min} , which is defined as the density at which fibers oriented in the plane of a sheet will start to come into contact with each other. However, we will attempt to estimate an approximate upper bound for ρ_{min} using a model we recently developed for the number of fiber-fiber contacts [13]. The model gives the following relationship between ρ_a and the number of fiber-fiber contacts:

$$\rho_a = \frac{2}{3} \frac{f\rho_w}{(1 + \beta)} \frac{D_w}{\sin 2\theta_{avg}} \frac{n_c}{L} \quad (4)$$

where n_c is the number of contacts for each fiber of length L , β is a constant dictating the spacing between the layers (approx. -0.33 for the samples we tested), and θ_{avg} is the average angle a fiber makes through any given cross section of the sheet.

We consider an isotropic network of fibers 3 mm long with $f = 0.5$, with $D_w = 30 \mu\text{m}$, and with four contacts per fiber. Four is the minimum number of contacts for a network in which each fiber rests on two other fibers and supports two other fibers. For an isotropic network,

θ_{avg} is 32.7° [15]. Substituting these values into Eq. 4 yields 33 kg/m³ as the value of ρ_a . Thus, even for this network of very low density, there is still some bonding. This circumstance suggests that ρ_{min} can be set to 0 kg/m³ without seriously compromising the accuracy of the relationship between scattering coefficient and density.

The final quantity required for the Eq. 2 is S_{min} . No theoretical expression for this quantity is available. Instead, we assume that $S_{min} = rS_0$, where $r < 1$ and r will possibly depend on the type of pulp and certainly on grammage. The value of r will be determined from the experimental data.

Substituting the expressions for S_0 , S_{min} , and ρ_{max} into Eq. 2 and setting ρ_{min} to zero yields Eq. 5:

$$S = \frac{c2D_w}{A_w\rho_w} \left(1 - \frac{\rho_a}{f\rho_w}(1-r) \right) \quad (5)$$

Substituting Eq. 5 into $RBA = (S_0 - S)/S_0$ yields Eq. 6:

$$RBA = \frac{\rho_a}{f\rho_w}(1-r) \quad (6)$$

Equation 6 is a remarkably simple expression, from which RBA can be calculated without the need for any extrapolation to determine S_0 , provided that the unknown constants r and c can be estimated. No simple extrapolation of S against ρ_a can be used to determine the missing quantities r and c in Eqs. 5 and 6, since both D_w and f may vary with the refining level or pressing conditions. However, rearranging Eq. 5, we obtain Eq. 7:

$$S \frac{A_w\rho_w}{2D_w} = c - c \frac{\rho_a}{f\rho_w}(1-r) \quad (7)$$

Normalizing the measured values

Thus if we plot $S A_w \rho_w / (2 D_w)$ against $\rho_a / (f \rho_w)$, then all the points should lie on one straight line, provided only that r and c remain constant as the level of bonding changes. Plotting these two groups against each other has the effect of normalizing the measured values of S and ρ_a for any change in fiber cross-sectional shape produced by refining or wet pressing.

These two groups are plotted in Fig. 8. The data from the accepts, the rejects, and the series L_0 to L_3 have all been fitted

quite well with a single straight line, suggesting that the starting assumption behind the theory is valid. In other words, the scattering coefficient is linearly related to sheet density if the fiber cross-sectional shape is constant. From the y -axis intercept, it can be seen that $c = 0.158$, from which it can be calculated that $r = 0.246$. Substituting this value into Eq. 6 produces Eq. 8:

$$RBA = 0.754\rho_a/(f\rho_w) \quad (8)$$

With Eq. 8, RBA can be calculated from only the sheet density and the fiber fill factor.

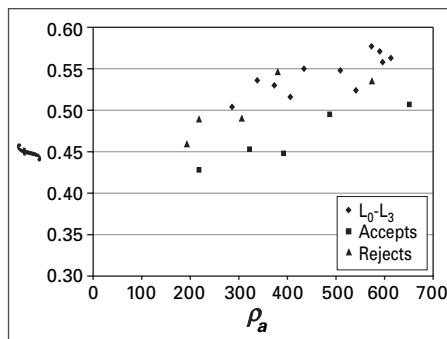
This equation for RBA is exactly the same form as one we developed previously [13] based on a model for the number of fiber-fiber contacts, and it is almost numerically identical to it. The factor c , which is the proportionality constant between the estimated surface available for scattering and the scattering coefficient, is much higher than the literature values of around 0.045 [17]. The difference can be ascribed to several causes.

The first reason is that the values by Swanson and Steber [17] were obtained from the slope of a plot of scattering coefficient versus surface area measured by nitrogen adsorption. Thus to obtain c from such a plot, one must assume that the total area measured by nitrogen adsorption is available for light-scattering. In fact, the area that scatters light appears to be about half that available for nitrogen adsorption [3] because of the difference in the scale of surface features that the two methods can "see," with a nitrogen molecule being much smaller than the 700-nm wavelength of light used in our work. The strong reduction in reflectivity as the angle between surface and incident light decreases from 90° will also play a role.

The second factor is that the fiber widths shown in Fig. 4 are those calculated by fitting a rectangular bounding box around each irregular fiber cross section. Consequently, the equivalent scattering surface will likely be larger than the assumed scattering surface of $2D_w$.

DISCUSSION

The good linear fit to the data in Fig. 8 indicates that our initial assumption was



7. The fill factor, f , as a function of the sheet density, ρ_a .

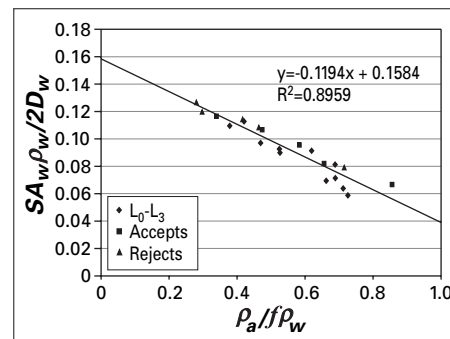
justified. Scattering coefficient is linearly related to sheet density, provided that the shape of the fiber has not changed. Thus, previous work showing such a linear relationship when density is altered by refining [5-9] can be considered a special case of the more complex general relationship between scattering coefficient and density. We expect such a linear relationship only when the fiber cross-sectional shape stays unaltered as the level of bonding changes. Kibblewhite has found that fiber width and the fill factor are largely unchanged when previously dried kraft pulps are refined in an Escher-Wyss laboratory refiner [7].

Figure 8 demonstrates the validity of Eq. 6 for determining the value of the RBA variable. For the first time we have a way of estimating the relative bonded area without requiring extrapolation. This method has the potential to be used to measure the RBA in machine-made papers, where it is not possible to vary the level of bonding. Still, it would be necessary to check the value of r for a given pulp from measurements of handsheets made from the same pulp.

Finally, Eq. 1 and Fig. 4 demonstrate that the RBA variable alone does not provide a complete description of the level of bonding in a sheet. While the value of RBA increases with increasing sheet density, the total area available for bonding increases too, as Fig. 4 shows. It is the total bonded area that is critical in determining the mechanical properties of paper.

CONCLUSIONS

To calculate the relative bonded area, it is necessary to find the scattering coefficient, S_0 , of a completely unbonded sheet. Previously, extrapolated correlations of scattering coefficient against either sheet density or tensile strength have been used to estimate S_0 . However, to obtain data



8. Plot of the two groups to determine r and c and to verify the theory.

over the required range, whoever makes the paper has to vary the sheet density and tensile strength by changing the level of wet pressing and/or the degree of refining.

Nevertheless, either of these two simple correlations can be successful only if the fiber cross-sectional shape remains constant. The collapse and flattening out of fibers, typically observed with increasing wet pressing pressure, will increase S_0 . Thus, for the RBA to be determined, S_0 has to be determined separately for each data point, and no simple correlation can be applied to determine one single value of S_0 .

To overcome these difficulties, we developed a method to correct measured values of scattering coefficient and sheet density for changes in fiber cross-sectional shape. Corrected data from six sets of pulps were then shown to fall onto one straight line. We used this linear relationship to derive a remarkably simple expression for determining the relative bonded area. **TJ**

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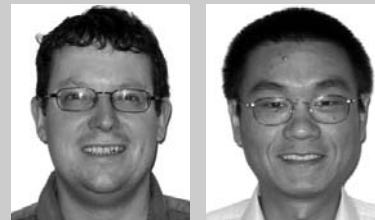
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