

Use of UV–visible diffuse reflectance spectroscopy for chromophore research on wood fibers: a review

John A. Schmidt and Cyril Heitner

ABSTRACT: *ISO brightness, the reflectance of paper determined at a wavelength of 457 nm, is an important industrial measure of the optical quality of paper. However, it is a poor probe of (a) the chemical changes that occur as wood is processed into paper and (b) how these changes influence paper color. The results presented here demonstrate that spectra of absorption coefficients in the ultraviolet–visible range of wavelengths provide much more detailed information on the nature and chemistry of the chromophores that contribute to the color of paper.*

KEYWORDS: *Brightness, chromophores, color, diffuse reflection, ISO, optical properties, paper, review, ultraviolet spectroscopy, wood fibers.*

Substitution of bleached mechanical and ultra-high-yield pulps for chemical pulps has become a topic of increasing interest during the past decade. While the economic incentives for substitution are compelling, there are several technical barriers standing in the way of this goal.

- The maximum commercially attainable brightness for softwoods, achieved by alkaline-peroxide bleaching, is about 83 points ISO, 7–8 points short of a fully bleached kraft pulp.
- Bleached mechanical pulps have a yellowish cast not found in chemical pulps of identical brightness, a problem important to color printing.

- Mechanical pulps undergo photochemical reactions that produce yellow chromophores, which drastically shorten the useful life of paper grades containing mechanical pulp.

All of these phenomena arise from the presence of chromophores—chemical groups that absorb light—in the pulp. If we are to breach the identified technical barriers, we must thoroughly understand (a) the bleaching reactions that remove chromophores and (b) the thermal and photochemical reactions that produce chromophores.

A standard measurement of the optical quality of paper is ISO brightness, the reflectance of a sample measured at a narrow wavelength

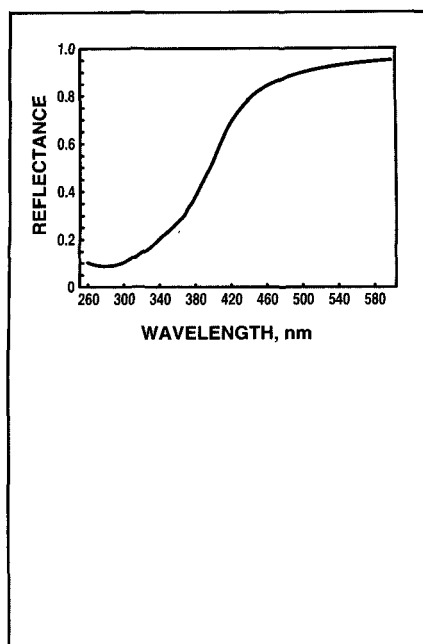
envelope centered at 457 nm, in the blue region of the visible spectrum. However, ISO brightness is an inadequate probe for studying the chemistry of the chromophores that contribute to the yellow color of pulp. It is insensitive to many important chromophores and, in a kinetic study, can lead to incorrect conclusions if surface effects (e.g., photochemical yellowing) are involved.

In this review, we will show that much chemical information can be obtained from absorption spectra, which are calculated from diffuse-reflectance spectra of low-basis-weight hand-sheets. After describing briefly the arguments for this approach, we present, equally briefly, the Kubelka–Munk theory and describe the experiment. In the final section, we give examples showing how ultraviolet–visible spectra can be used to monitor chemical changes that are not observable using standard brightness measurements.

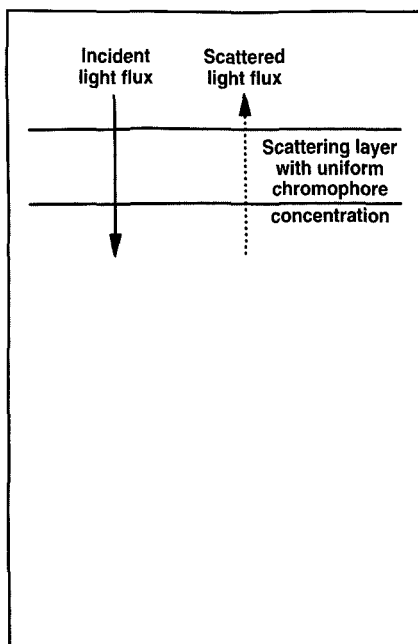
Rationale for measuring spectra and absorption coefficients

Figure 1 shows the reflectance spectrum of an optically thick sample of bleached thermomechanical pulp (TMP) of black spruce. The strongest absorption (the smallest reflectance) occurs at wavelengths below 400 nm, because this is where the absorption maxima of the various structural units of lignin occur. Thus, when measuring ISO brightness ($\lambda = 457$ nm), we are looking at the tails of absorption peaks whose maxima are located at shorter wavelengths. To get the most chemical

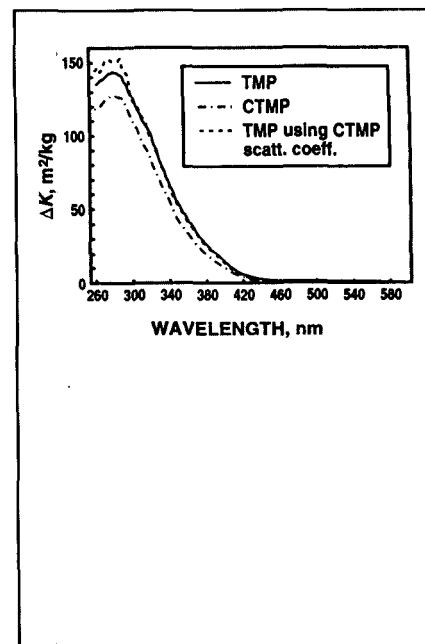
1. Reflectance spectrum of an optically thick sample of bleached TMP of black spruce



2. Essential elements of the Kubelka-Munk theory for diffusely reflecting materials. Reflectance is the ratio of the reflected and incident light fluxes at the sample surface.



3. Absorption spectra calculated for peroxide-bleached spruce TMP and CTMP, and for TMP using the CTMP scattering coefficient



information from a reflectance measurement, we need to look at a range of wavelengths, i.e., spectra. For lignin-containing pulps, the region from 300 nm to 500 nm is particularly important.

The relationship between reflectance and chromophore concentration is complex. For optically thick samples, these two quantities are connected by the Kubelka-Munk remission function $f(R_\infty)$, as seen in Eq. 1A.

$$f(R_\infty) = K/S = (1 - R_\infty)^2 / 2R_\infty \quad (1A)$$

where

R_∞ = measured reflectance

K = absorption coefficient

S = scattering coefficient.

Note that R_∞ is related to the ratio of absorption to scattering and not to the separate values of K and S .

The absorption coefficient K is given by Eq. 2A.

$$K(\lambda) = 2\epsilon(\lambda)C \quad (2A)$$

where

λ = wavelength of the measurement

ϵ = Beer-Lambert extinction coefficient

C = concentration of the absorber (chromophore).

If more than one chromophore absorbs at a given wavelength, the absorption coefficient will be the sum of the absorption by each chromophore, as shown in Eq. 2B.

$$K(\lambda) = 2 \sum_i \epsilon_i(\lambda)C_i \quad (2B)$$

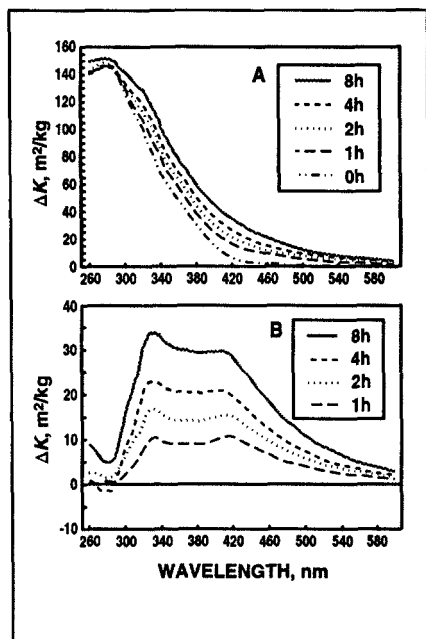
Since K is proportional to the concentration of chromophores, it can be used for kinetic analysis of changes in chromophore content. In principle, one can determine changes in K from changes in R_∞ through Eq. 1A. However, we shall see that in practice, it is often difficult to eliminate (a) effects of simultaneous changes in scattering coefficient during pulping and bleaching or (b) effects of sample inhomogeneity if chromophore content changes only at the surface of the paper sheet.

Determining changes in absorption coefficient

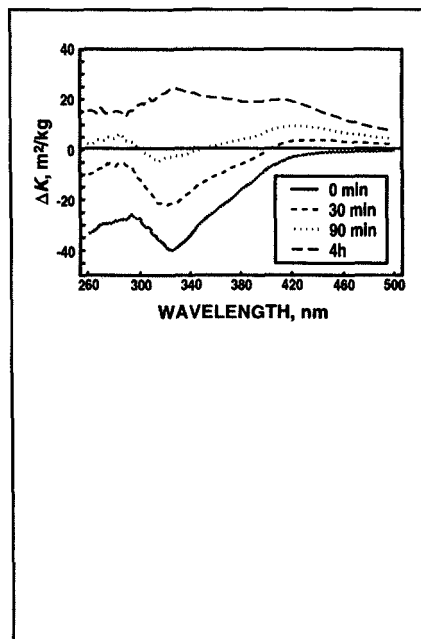
Kubelka-Munk theory

Figure 2 shows the essential elements of the Kubelka-Munk model of absorption and scattering; more detailed information is available in Kortüm (1). The sample is a composite of layers capable of both diffusely scattering and absorbing radiation. A key assumption of the theory is that the absorbers, or chromophores, are uniformly distributed throughout the layers. There is one radiation flux due to incident light, and a second flux, in the opposite direction, is due to scattering. Reflectance is the ratio of the reflected flux to the incident flux at the sample surface. This model leads to two coupled differential equations, which can be solved given appropriate boundary conditions. For the boundary condition that the sample be sufficiently thick such that no incident radiation passes through, the solution is the Kubelka-Munk remission function given in Eq. 1A.

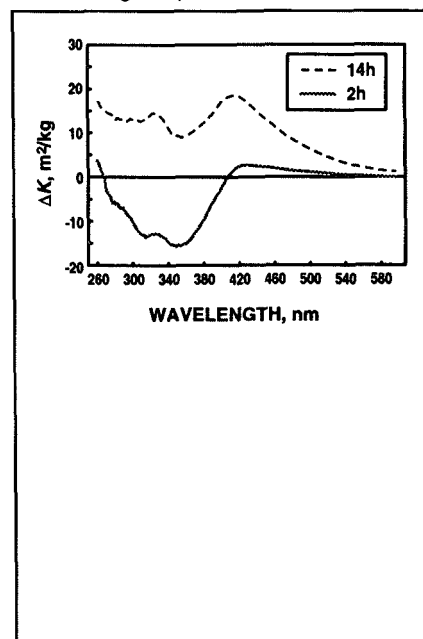
4. A: Absorption spectra of bleached spruce TMP after irradiation with near-UV light. B: Absorption-difference spectra of the samples in A ($\Delta K = K_{\text{irradiated}} - K_{\text{unirradiated}}$).



5. Absorption-difference spectra of peroxide-bleached spruce TMP after reduction with sodium borohydride and subsequent irradiation with near-UV light ($\Delta K = K_{\text{reduced, irradiated}} - K_{\text{unreduced, unirradiated}}$).



6. Absorption-difference spectra of bleached spruce TMP after treatment with ascorbic acid and subsequent irradiation with near-UV light ($\Delta K = K_{\text{irradiated}} - K_{\text{unirradiated}}$). A near-UV absorbing chromophore is photobleached during the first 2 h of light exposure.



Calculating ΔK from R_{∞}

If the scattering coefficient is known and remains constant, the remission function allows us to calculate a spectrum of the change in absorption coefficient (ΔK vs. wavelength) from measured spectra of R_{∞} .

$$\Delta K = S[(K_1/S) - (K_2/S)] = S[f(R_{\infty})_1 - f(R_{\infty})_2] \quad (3A)$$

If the scattering coefficient is not known, a quantity proportional to chromophore concentration can be obtained from Eq. 3b.

$$PC = f(R_{\infty})_1 - f(R_{\infty})_2 \quad (3B)$$

In studies of pulp discoloration, this is commonly referred to as the post-color number, PC.

Two conditions must be met if Eq. 3A is to give a true change in absorption coefficient.

1. The distribution of chromophores must be uniform in both samples.
2. The process contributing to the change in absorption coefficient must not alter the scattering coefficient.

These conditions are not always met. For example, it would be difficult to compare the absorption coefficients for pulps produced under different refining or grinding conditions, since refining and grinding clearly affect scattering. Alkaline-peroxide bleaching can also affect scattering, since the alkali swells the pulp fibers, which can affect their surface morphology and scattering. Finally, measurements of the change in absorption coefficient cannot be obtained in this way for optically thick samples that have been discolored by light, since the chromophore concentration in an irradiated sample is no longer uniform.

Calculating ΔK from the reflectance of transmitting sheets

We can avoid these problems if, instead of using the remission function K/S , we calculate K and S individually. Two methods of calculating K and S are described here.

Method A. In Method A, scattering coefficient S is calculated from direct measurement of reflectances R_0 and

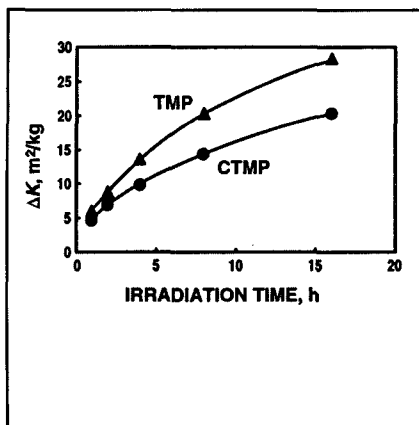
R_{∞} . A single transmitting sheet is suspended over a nonreflecting black background,¹ and the reflectance R_0 is recorded. Next, a sufficient number of sheets are stacked so that no further change in reflectance occurs upon addition of another sheet, and the reflectance R_{∞} is recorded. In principle, R_{∞} also could be measured from a brightness handsheet. In practice, this is less desirable because the extent of inter-fiber bonding is probably not the same for a single opaque sheet as for a stack of thin sheets. The scattering coefficient S is calculated from Eq. 4, where b is the basis weight of the sheet.²

$$S = (1/b) [R_{\infty} / (1 - R_{\infty}^2)] \ln \{ R_{\infty} [(1 - R_{\infty} R_0) / (R_{\infty} - R_0)] \} \quad (4)$$

¹A standard black cavity, as described in CPPA standard E.2.

²The Kubelka-Munk theory uses the thickness of the sample here. Van den Akker showed that, for paper, basis weight can be used instead without loss of mathematical rigor (2).

7. Change in absorption coefficient at 420 nm for peroxide-bleached spruce TMP and CTMP during irradiation with UV light. The light-absorption coefficient at 420 nm increases more rapidly for TMP than for CTMP.



8. Comparison of photoyellowing for CTMP and TMP

	TMP	CTMP
Absorption of near-UV light	Light is strongly absorbed only into the first few surface layers (more near-UV photons per unit volume)	Light is less strongly absorbed in surface layers; near-UV photons penetrate further into the sample
Distribution of photochemically formed chromophores	Stronger absorption of near-UV photons at the sample surface leads to a higher concentration of photochemically formed chromophores in a thin surface layer	Lower concentration of photochemically formed chromophores in surface layers, but the yellowed layer is thicker

The absorption coefficient K is calculated from Eq. 1B, a rearrangement of Eq. 1A.

$$K = S[(1 - R_{\infty})^2 / 2R_{\infty}] \quad (1B)$$

Method B. Method B involves measurement of R_0 as well as R_w , the reflectance of a single sheet over a white backing of reflectance $R_{w.b.}$. The value of R_{∞} for the sample is then calculated from Eq. 5. (The Appendix at the end of this article discusses the source for Eq. 5.)

$$R_{\infty} = \frac{R_0 + (1/R_w)}{[(R_0 - R_w + R_{w.b.}) / (R_{w.b.} R_0)] + R_w} \quad (5)$$

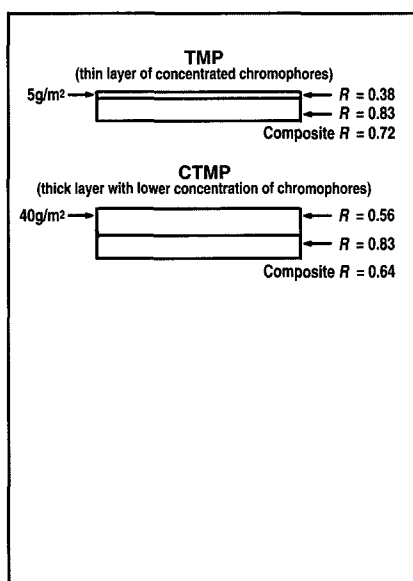
S and K are then calculated from Eqs. 4 and 1b, as in Method A.

In Eqs. 4 and 5, R_0 is the reflectance recorded over a perfectly nonreflecting background. However, black cavities have a finite reflectance, typically 0.2–0.4%. While this is a small deviation, the true value of R_0 can be determined from Eq. 6, where R_b is the reflectance measured over a cavity of reflectance $R_{b.b.}$. (Derivation of Eq. 6 is given in the Appendix.)

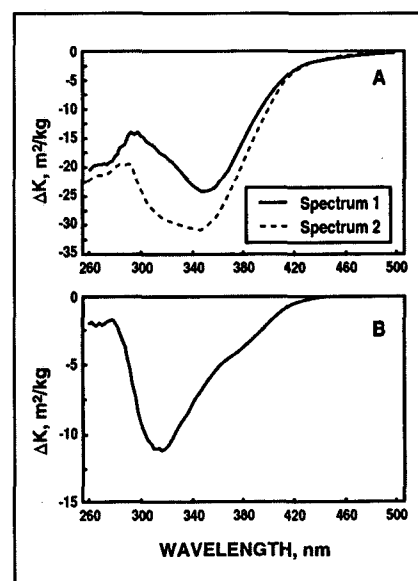
$$R_0 = \frac{R_b R_{w.b.} - R_w R_{b.b.}}{R_{w.b.} - R_{b.b.} - [R_{w.b.} R_{b.b.} (R_w - R_b)]} \quad (6)$$

While these methods generate separate values for absorption and scattering coefficients, they do not eliminate

8. Brightness (R_0) of composite sheets of TMP and CTMP (equal initial brightness) after photoyellowing. The TMP sheet, with its thin, strongly absorbing yellow layer, has a higher brightness than the CTMP sheet, where the yellowed layer is thicker but absorbs less strongly.



9. A: Absorption-difference spectra of peroxide-bleached TMP ($\Delta K = K_{\text{bleached}} - K_{\text{unbleached}}$). Spectrum 1 corresponds to normal peroxide-bleaching practice, while Spectrum 2 is observed after long reaction times. The result shows that a different chromophore is slowly removed during the extended reaction time. B: Spectrum obtained by subtracting Spectrum 1 from Spectrum 2. Result shows that maximum absorption loss occurs at about 320 nm.



the dependence of K upon S ; i.e., K depends on S by the remission function (Eq. 1B), and S must be calculated before K . Care is therefore required in comparing values of K for two pulps with different scattering coefficients.

Intuitively, we expect a change in scattering coefficient to cause a change in R_{∞} rather than K . This is easily confirmed for a given pair of samples, as shown in Fig. 3, which shows the absorption spectra of bleached soft-

wood TMP and bleached softwood CTMP (chemithermomechanical pulp). In the near-ultraviolet (UV) region, the CTMP has a significantly lower absorption coefficient. To confirm that this arises from a reduction in the concentration of chromophores, and not from an artifact of the lower scattering coefficient of CTMP, we calculated an absorption spectra from Eq. 1B, using R_{∞} for the TMP sample but S for the CTMP. The resulting spectrum is virtually identical to the TMP sample, as seen in Fig. 3. This confirms that the reduction in absorption coefficient observed for CTMP results from a lower concentration of chromophores.

Sample requirements

The magnitude of the difference between R_0 and R_{∞} depends upon the amount of light transmitted through the sample and affects (a) the precision of the values of K and (b) the short-wavelength limit to which we can measure in the ultraviolet. The lignin in mechanical pulps absorbs strongly at near-UV wavelengths ($\lambda < 400$ nm). If a sheet is too thick, very little light will penetrate to the backing and, through normal measurement error, situations may arise where R_0 equals or exceeds R_{∞} . Either situation leads to singularities in Eq. 4. Thus, R_0 must be measured using the thinnest sheets possible.

We used sheets with a basis weight of 10 g/m². The sheets were made using the procedure described in the Appendix to this report. Sheets of this grammage allow measurements to 260 nm with a standard deviation of about 5–10%. At wavelengths greater than 300 nm, where the absorption is not as strong, the standard deviation drops to about 1–2%.

The use of 10-g/m² sheets also eliminates the problem of nonuniform chromophore concentrations in sheets subjected to light-induced yellowing. Because the 10-g/m² sheets are illuminated evenly throughout their thickness, the concentration of chromophores remains uniform. This is verified when the same reflectance is mea-

sured from both sides of an irradiated sheet.

A potential problem with using sheets of such a low basis weight is that formation may be nonuniform over the area probed by the spectrophotometer beam. The impact of anomalous measurements is lessened by averaging the results from four to six samples.

Presentation of spectra

Figure 4 shows absorption spectra for bleached TMP before irradiation and after irradiation ranging from 1 h to 8 h. The spectra in Fig. 4A are presented as K vs. λ and show a featureless, increasing absorption with irradiation time. In general, the spectra are much more informative when presented as difference spectra of ΔK vs. λ ($\Delta K = K_{\text{irradiated}} - K_{\text{unirradiated}}$), as seen in Fig. 4B. The apparent absorption maxima are easily identified from the difference spectra.

Data measurement and handling

A UV-visible spectrophotometer equipped with an integrating sphere is required to measure reflectance spectra. This accessory is widely available. If determination of absorption-difference spectra is to be used as a routine research tool, a personal computer is necessary to do the tedious calculations of K and S , and to do the averaging and subtraction of spectra that are required to generate difference spectra. Modern UV-visible spectrophotometers are capable of transmitting data as ASCII characters to an attached computer. The appropriate data manipulation can be done in a spreadsheet program (e.g., LOTUS 123, Excel, etc.) or using simple software written by the user.

Application examples

The prime justification for determining absorption spectra is that this technique can reveal information that is not accessible from a simple brightness measurement. In this final section, we present examples from our own work

and the recent literature to illustrate the utility of this technique.

Effect of borohydride reduction on light-induced yellowing of mechanical pulps

Aromatic ketone groups have long been thought to be the main sensitizer of light-induced yellowing in high-yield pulps. However, extensive reduction (with sodium borohydride) of carbonyl groups to the corresponding nonabsorbing benzyl alcohols has almost no effect on the rate of brightness loss (3). On the basis of brightness measurements only, it is quite reasonable to conclude that perhaps aromatic ketones are not important to yellowing, and this suggestion has been made (4). However, when the complete absorption spectrum is considered, it is clear that such a simple interpretation is premature.

Figure 5 shows the absorption-difference spectra of peroxide-bleached, black spruce thermomechanical pulp that was exhaustively reduced with sodium borohydride to remove carbonyl groups and then irradiated. The difference spectra were determined by subtracting the absorption spectrum of an unirradiated sample of peroxide-bleached pulp. Treating bleached TMP with borohydride reduces aromatic ketones, resulting in the loss of an absorption maximum at about 330 nm. When the reduced samples are irradiated, this absorption recovers rapidly. After irradiation for about 4 h, the difference spectrum was identical to that of an irradiated, unreduced sample of bleached TMP.

The rapid recovery of the absorption at 330 nm and the fact that, after sufficient irradiation, both reduced and unreduced samples have identical difference spectra suggest that aromatic carbonyl groups are formed in a light-driven reaction. If this is so, then simple borohydride reduction is an inadequate test for the importance of aromatic carbonyl groups in photoyellowing.

Photobleaching of mechanical pulps by ascorbic acid

Several researchers have observed that ascorbic acid is among the more-effective inhibitors of light-induced brightness reversion. Beyond the general knowledge that ascorbic acid is an effective antioxidant, little is known about its mechanism of action.

The difference spectra in Fig. 6 show that during the first two hours of irradiation of ascorbic acid-treated bleached TMP, a chromophore absorbing at about 350 nm is photobleached (3). We have not positively identified this chromophore, but we speculate that it may be a stilbene. The existence of an additional chromophore that absorbs in the near-UV region is another indication that the mechanism of yellowing of mechanical pulps is more complicated than current models suggest.

Rate of brightness reversion in TMP vs. CTMP

It is well known that bleached CTMP loses brightness faster upon irradiation than bleached TMP, and this has been interpreted as evidence of a faster reaction rate in CTMP (5). In fact, the rate of chromophore formation is faster in TMP (6), and the more-rapid brightness loss of bleached CTMP results from a physical effect rather than a chemical effect.

Figure 7 shows that the increase in the absorption coefficient (at 420 nm), and therefore the increase in chromophore concentration, is more rapid in TMP. To understand the apparently contradictory behavior of absorption coefficient and brightness (an R_∞ measurement), we must examine the absorption spectra of TMP and CTMP, shown earlier in Fig. 3. CTMP has a lower absorption coefficient in the near-UV region, a consequence of its lower chromophore concentration. The implications of these differences in absorption for the distribution of photochemically formed chromophores in optically thick sheets are summarized in Table I.

The different distribution of chromophores in CTMP can result in faster brightness loss, even when the rate of chromophore formation is slower. Figure 8 shows the brightness calculated for two composite samples (7), each consisting of a yellowed surface layer on top of a brighter lower layer. The lower layer has the same brightness in both cases. Sample A, which corresponds to the case of bleached TMP, has a thinner yellowed layer with a higher concentration of chromophores (i.e., lower R_∞ for the yellowed layer³) than does sample B, which corresponds to the bleached CTMP. Even though sample A has a higher concentration of chromophores in the yellowed layer, the brightness of the composite is higher than that of the CTMP sample. This occurs because the reflectance depends not only on the total amount of chromophores produced (a chemical effect), but also on how these chromophores are distributed in the sheet (a physical effect). It is this physical effect that causes the faster brightness reversion of CTMP.

This example clearly shows why measurements of infinite reflectance, for example PC number, cannot probe the rates of surface chemical changes such as light-induced yellowing. The central assumption of the Kubelka-Munk theory—the uniform distribution of absorbers—no longer holds, so the remission function (Eq. 1A) is no longer proportional to the concentration of chromophores. Even the simple correlation of higher R_∞ with higher chromophore concentration is not warranted without other supporting evidence.

Peroxide bleaching of mechanical pulp

The absorption-difference spectra in Fig. 9 were obtained after treating bleached TMP of black spruce with large excesses of hydrogen peroxide

(8). Spectrum 1 in Fig. 9A was observed after reaction times comparable with commercial bleaching practice. The spectrum corresponds to the loss of coniferaldehyde end groups and quinones (9), as has been previously reported. Spectrum 2 was observed after long reaction times (up to 300 h) and differs from Spectrum 1 at wavelengths < 400 nm. Clearly, a different chromophore is being removed in a very slow reaction.

The nature of the chromophore being removed in this slow reaction is seen more easily by subtracting Spectrum 1 from Spectrum 2, as seen in Fig. 9B. The position of the absorption loss ($\lambda_{\max} \approx 320$ nm) is similar to that observed after borohydride reduction, suggesting that aromatic ketones are being oxidized.

Note that no brightness changes occur during the slow phase of bleaching, since aromatic ketones do not absorb at 457 nm. Thus, this change would have been missed if brightness were the only experimental measurement. Although this slow chemical change does not affect brightness, it does effect the color of the pulp. Since the tail of the absorption continues beyond 400 nm, its presence affects the whiteness of the pulp, which is usually reflected in the b parameter of the L, a, b color coordinates.

The kinetics of peroxide bleaching of mechanical pulp from *E. regnans* (10, 11) and *P. radiata* (12) has been studied recently by determining changes in absorption coefficient. Because this quantity is directly proportional to the concentration of chemical species, the authors were able to propose several mechanisms and to develop further experiments to distinguish between them. They concluded that peroxide bleaching is best described by a model involving an equilibrium between chromophoric and leukochromophoric groups, in addition to reactions that destroy chromophores.

³ R_∞ does not imply that the surface layer is infinitely thick. Calculation of the brightness of the composite requires the reflectance of a sample that is analogous to the surface layer but is infinitely thick (7).

Summary

Spectra of absorption coefficients in the UV-visible region can reveal much more information about the chemistry occurring in wood fibers during pulping, bleaching, and yellowing than can standard measurements of brightness. With the advent of inexpensive personal computers, measurement of absorption spectra can become a routine research task. **□**

Literature cited

1. Kortüm, G., *Reflectance Spectroscopy*, Springer-Verlag, Berlin, 1969, p.107.
2. Van den Akker, J. A., *Tappi* 32(11): 498(1949).
3. Schmidt, J. A. and Heitner, C., *J. Wood Chem. Technol.* 11(4): 397(1991).
4. Ek, M., Lennholm, H., and Iversen, T., *Nordic Pulp Paper Res. J.* 5: 159(1990).
5. Gellerstedt, G., Hammar, L.-Å., Olsson, A., *et al.*, *Svensk Papperstid.* 88(6): R68(1985).
6. Heitner, C. and Min, T., *Cellulose Chem. Technol.* 21: 289(1987).
7. Scallan, A. M., *J. Pulp Paper Sci.* 11(3): J80(1985).
8. Holah, D. G. and Heitner, C., *J. Pulp Paper Sci.*, 18(5): J161(1992).
9. Reeves, R. H. and Pearl, I. H., *Tappi* 48(2): 121(1965).
10. Wright, P. J. and Abbot, J., *J. Wood Chem. Technol.* 11(3): 349(1991).
11. Wright, P. J., Ginting, Y. A., and Abbot, J., *J. Wood Chem. Technol.* 12(1): 111(1992).
12. Abbot, J. and Ginting, Y. A., *J. Pulp Paper Sci.* 18(3): J85(1992).
13. Kubelka, P., *J. Opt. Soc. Am.* 38: 448(1948).

The authors thank B. D. Jordan and M. O'Neill for assistance in setting up the UV-visible spectrophotometer to measure absorption coefficients. They also thank A. A. Koukoulas for a thorough review of the manuscript.

Received for review July 23, 1992.

Accepted Sept. 5, 1992.

Appendix

Derivation of Eq. 6

Kubelka and Munk derived several equations relating R_∞ , R_0 , R , and R_g (13). R_∞ and R_0 are defined as earlier. R is the reflectance recorded when the sample is suspended over a background of reflectance R_g . Equation 5 is one of these (with $R_w = R$ and $R_{w.b.} = R_g$). Equation A-1 is another:

$$R_0 = (R - R_g) / \{1 - [R_g(2a - R)]\} \quad (\text{A-1})$$

where

$$2a = R_\infty + 1/R_\infty$$

Setting R_b = sheet reflectance over the black backing means that $R_{b.b.} = R_g$. Substituting for $2a$ from Eq. 5 yields Eq. A-2.

$$R_0 = \frac{R_b - R_{b.b.}}{1 - \{R_{b.b.} [R_w + \frac{R_0 - R_w + R_{w.b.}}{R_0 R_{w.b.}} - R_b]\}} \quad (\text{A-2})$$

Solving Eq. A-2 for R_0 gives Eq. 6 of the text. Note that if $R_{b.b.} = 0$, Eq. 6 reduces to $R_0 = R_b$.

Preparation of low-basis-weight sheets

Low-basis-weight sheets were prepared by filtering an appropriate amount of pulp onto a 74- μm Teflon™ mesh placed over the wire screen of a British sheet machine. Care should be taken to ensure that there are no air bubbles trapped under the mesh. The

mesh and the sheet are placed on a kraft blotter (mesh-side down). A Whatman filter paper (18.5-cm diam.) is placed on top, and the sheet is pressed manually with three or four passes of a couch roll. After peeling the Teflon mesh from the sheet, the sheet and filter paper are placed sheet-side down on a stainless steel plate and are pressed for 5 min at 0.34 MPa. After air drying, the sheet is carefully separated from the filter paper with the aid of a spatula and cut into squares (ca. 5 cm x 5 cm). For determination of the basis weight, the area of each sheet is measured accurately using precision calipers.