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Development of a methodology using methylene blue to quantify the amount of UV-screen applied and to determine the homogeneity of application on paper

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Abstract

Methylene blue was employed as a model molecule to develop a methodology to quantify the incorporation of compounds into the paper matrix. Quantification was based on absorption and fluorescence measurements of the solutions employed to transfer methylene blue into paper. This methodology is suitable to quantify amounts which correspond to less than 1% of the paper weight, and it can be applied when testing the inhibition efficiency of UV-screens against the photoyellowing of paper made from mechanical pulp. In addition, two application methodologies were compared to establish which one led to a more homogeneous distribution of methylene blue in paper. The 'dip method' led to a more even distribution than the 'drop method'.

Keywords: Methylene blue; UV-screen; Homogeneity

1. Introduction

Different approaches have been developed to minimize the yellowing in mechanical and chemimechanical pulps and paper. One of these approaches is the use of light absorbing compounds (UV-screens) [1–3], which act as 'sunscreens' by blocking the light normally absorbed by lignin. Since the photochemistry of lignin is responsible for the formation of yellow products [4], the absorption of light by another compound in principle leads to a decrease of the yellowing of paper. The normal procedure to test inhibitors is to determine how the loading, i.e., the amount of inhibitor applied on paper, affects the extent of yellowing. Loadings are generally established by weighting the paper before and after application of the UV-screen, and the amount of screen on the paper corresponds to the difference between the two weights [2]. In order to be commercially viable, the loadings of the inhibitor will have to be small. However, since the errors in the weights of paper are typically 1%, any loadings below this value cannot be determined accurately by weighting the samples. An alternate method involves slowly dropping a concentrated solution of the inhibitor onto the paper sample and evaporating the solvent ('drop method'). This method assumes that all the inhibitor was incorporated into the paper. However, it leads to non-homogeneous distribution of the inhibitor.

Quantification of the amount of inhibitor by diffuse-reflectance, which is a direct spectroscopic method, is problematic since the scattering coefficient of the paper is not easily determined [5], and cannot be assumed to be the same in the presence and absence of the UV-screen. In addition, quantitative measurements using diffuse-reflectance are difficult for highly absorbing samples [6,7], which is the condition encountered when UV-screens are applied to mechanical papers, since both the screen and lignin absorb in the same region of the spectrum. A second important aspect when determining the efficiency of a UV-screen, is to establish the homogeneity of its distribution in paper, since aggregation will influence the screen's photophysics.

We employed methylene blue (MB) to develop a methodology to quantify the amount of screen applied to paper and to explore the distribution homogeneity. MB absorbs in the visible region of the UV–Vis spectrum. In this respect, MB cannot be employed as a UV-screen to inhibit the yellowing of paper, but it is instrumental to compare absorption and fluorescence measurements of the solutions used to load MB onto the paper with diffuse-reflectance measurements of the paper containing MB. In addition, MB forms dimers at moderate concentrations and oligomers at high concentrations in solution, and the absorption of its monomeric and

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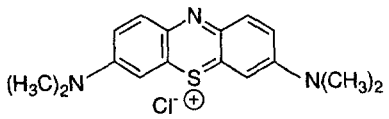
dimeric forms are different [8–13]. This property was employed to establish the degree of dimerization when MB is incorporated into paper.



(MB)

2. Experimental

2.1. Materials

The bleached thermomechanical (BTMP) or bleached chemithermomechanical (softwood BCTMP) pulp handsheets (basis weight of approximately 200 g/m^2) were prepared by Paprican (Pointe Claire, QC Canada). Methylene Blue (MB, 3,7-*bis* [dimethylamino]-phenazothionium chloride·3H_2O, Sigma), and methanol (ACP, spectragrade) were used as supplied. 2,4-Dihydroxybenzophenone (DHBP, Aldrich) was recrystallized from diethyl ether/petroleum ether and water was deionized with a SYBRON (Barnstead) system.

2.2. Equipment

UV–Vis absorption measurements were obtained with Varian spectrophotometers (Cary 1E or Cary 5) at room temperature, using cells with pathlengths of 1.000 cm or 0.100 cm. The measured absorbance values were always kept below 2.5. All absorbance values presented have been normalized to a 1.0 cm pathlength.

UV–Vis diffuse-reflectance spectra were obtained with the Cary 1E using an integrating sphere (Varian #00 10046400, ID-73 mm, with barium sulfate as internal coating). Nylon disks supplied by Dr. J.C. Scaiano (University of Ottawa) (ISO brightness 97.21 (sample) and 97.27 (reference), measured at Paprican, Pointe Claire, QC Canada) were used as references to obtain the baseline for the diffuse-reflectance measurements. Handsheet samples were placed on top of 30 Whatman filter paper (#1) sheets and the smooth side of the handsheets was exposed to the incident beam. Since the paper surface is not homogeneous, three measurements exposing different areas of the handsheet were obtained for each sample. The Kubelka–Munk remission function ($F(R)$) was employed to convert reflectance measurements into 'absorbance' spectra which are related to chromophore concentrations [5]. Great care must to be taken in order to obtain absolute concentrations from paper samples measured by diffuse-reflectance [5–7]. For this reason, we did not attempt to obtain absolute concentrations, but we employed $F(R)$ values to measure relative concentrations of MB incorporated into paper.

Fluorescence measurements were performed with a PTI QM-2 spectrofluorimeter. The excitation and emission slits were set to a value that corresponds to a bandpass of 2.0 nm. MB samples in solution or when incorporated into paper were excited at 610 nm. In both cases, a front-face geometry was employed by using a triangular cell for the solution experiments, or by mounting the paper sample on a triangular solid holder that ensures a 45° angle between the excitation beam and the detection optics.

2.3. Methods

Paper samples of different sizes were cut from handsheets (4 cm × 4 cm, 2.25 cm × 4 cm and 0.6 cm × 12.5 cm, weighting approximately 0.30 g, 0.18 g, and 0.15 g, respectively). These samples were soaked in methanol for 20 min and air dried for 24 h in order to remove impurities.

2.3.1. Application of screens

Two different methods were employed to apply the screens (MB or DHBP) onto the paper samples: (1) Drop method: The screen solution containing the amount to be applied on paper was dropped on the smooth side of the handsheet samples by using a micropipette. Application started in the center and proceeded to the margins of the sample in a spiral fashion. The volumes applied were 2.0 ml and 1.125 ml for 4 cm × 4 cm and 2.25 cm × 4 cm samples, respectively; except when screens in methanol were applied to 4 cm × 4 cm samples when only 1 ml was employed. (2) Dip method: Samples were immersed in screen solutions for a given amount of time. After removal of the paper sample, the volume of the solution is smaller due to absorption of water by the paper. The initial volume was re-established by the addition of solvent before the absorption and fluorescence measurements were performed. In the case of 2.25 cm × 4 cm samples a calibrated 200 ml Erlenmeyer flask was employed, whereas the 0.6 cm × 12.5 cm samples were dipped into 10 ml volumetric flasks. For both methods the samples which were treated with methanolic solutions were dried for at least 12 h, whereas the samples treated with aqueous solutions were dried for at least 48 h.

2.3.2. Screen incorporation kinetics

The samples (4 cm × 4 cm or 2.25 cm × 4 cm) were immersed in 100 ml Erlenmeyer flasks containing MB. At specific times, aliquots of 0.5 ml and 0.15 ml were removed for the solutions containing 0.05 mM and 0.5 mM MB, respectively. The former were diluted by 1:4 and the latter by 3:40 before the absorption was measured. These dilutions were necessary to obtain MB concentrations amenable for absorption measurements. The volumes retrieved as aliquots were small, and no correction for the volume decrease was performed.

3. Results

MB was chosen as the model compound to develop the quantification methodology for additive incorporation into paper samples because it absorbs in a region of the spectrum (Fig. 1A), where there is no interference from the absorption of paper. The monomer–dimer equilibrium for MB in aqueous solutions has been well established [13], and these species have absorption maxima at 664 nm and 610 nm, respectively. Due to the presence of dimerization, the MB absorption values do not follow the Beer–Lambert law and calibration curves were established (inset Fig. 1A). The fluorescence spectrum of MB is fairly broad with a maximum at 680 nm. The same emission spectra are obtained when exciting MB at 610 nm or 660 nm, suggesting that either the dimer and monomer have the same emission spectra or only one species, e.g., the monomer emits.

The 'drop' and 'dip' methods were employed to incorporate MB into the handsheets. Visual inspection of the samples shows that the 'drop' method clearly leads to a very non-homogeneous distribution of MB on the handsheets (Fig. 2). The differences observed are not due to the amount of MB incorporated, since more MB was incorporated for sample C than for sample B. Diffuse-reflectance spectra were measured for both methods and $F(R)$ values were obtained for measurements at different positions of the handsheets with respect to the incident beam. Due to the heterogeneity of paper, the $F(R)$ values always vary for repeat measurements with one sample. In the case of the 'dip' method, this variation is between 6 to 10%, and for the 'drop' method it is approximately 15%.

The $F(R)$ spectra obtained from diffuse-reflectance measurements for samples prepared by the 'dip' (Fig. 1B) and 'drop' methods are similar. It is worth noting, that the relative amount of dimer with respect to monomer is much smaller

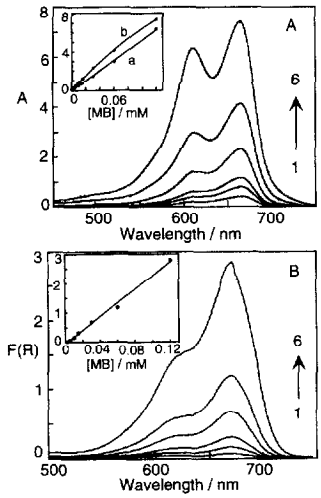


Fig. 1. MB absorption spectra in water (A) and $F(R)$ spectra for paper samples (B) dipped in aqueous solutions (1 to 6: [MB] = 5, 10, 15, 30,60, and 120 μM) Insets: (A), dependence of the absorption of the dimer (a, 610 nm) and monomer (b, 664 nm) on the MB concentration; (B), dependence of F(R) measured at 670 nm on the MB concentration.

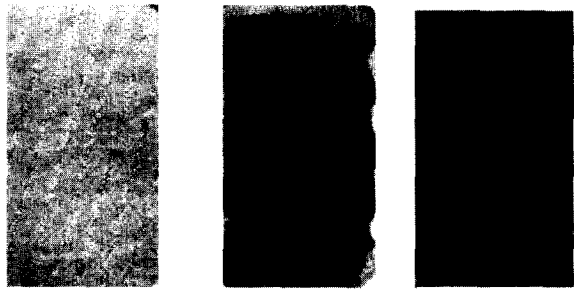


Fig. 2. Comparison of MB applied by the drop (B) and dip methods (C) with a blank handsheet sample treated only with water (A).

for MB incorporated into paper than in water. A significant increase of the dimer-to-monomer MB absorbance ratio ($A_{610\,nm}/A_{660\,nm}$) is observed in water, where this ratio varied from 0.45 to 0.88 for MB solutions with concentrations between 2.5 μM and 100 μM MB, respectively. In contrast, the ratio for paper samples ($F(R)_{618\,nm}/F(R)_{670\,nm}$) is fairly constant. This ratio varied between 0.46 and 0.52 for samples prepared by dipping the paper into aqueous solutions with MB concentrations between 2.5 μM and 100 μM MB, and it varied between 0.48 and 0.55 for samples prepared over the same concentration range with the 'drop' method. A linear relationship is observed between the $F(R)$ values at 670 nm and the concentration of the solution into which the paper samples were dipped (inset Fig. 1B).

The need to develop a spectroscopic method to quantify the amount of screen incorporated into paper was established by the fact that fluctuations of at least 1% for the weight of paper samples stored over a period of days are frequently observed. These weight differences are probably related to changes in ambient humidity and preclude the precise determination of the amount of screen incorporated at low loadings. The industrial method for determining the moisture in paper is to maintain paper samples at 105°C until a constant weight is achieved [14]. In principle, this method could be applied when testing the efficiency of screens. However, this method is more time-consuming than the method described in this work, and paper samples of at least 2 g have to be employed.

The 'dip' method was employed for the development of the quantification methodology, because of the more homogeneous distribution of MB when this method is applied (Fig. 2). Absorbance and fluorescence experiments were employed to quantify the amount of MB incorporated from measurements of the aqueous solutions before and after immersion of the paper samples. This method assumes that any decrease for the absorbance or emission intensities is due to transfer of MB from the solution to the paper. Besides absorbing MB, the paper also retains a significant amount of water. For this reason, the total volume was kept constant by the addition of water after removal of the paper samples. Calibration curves were established by fitting the monomer

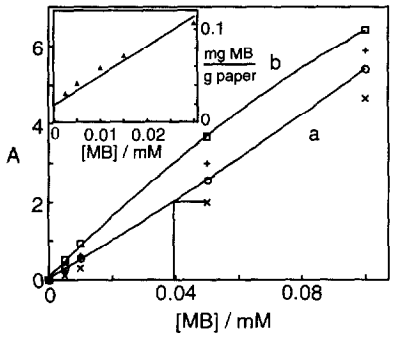


Fig. 3. Calibration curves (solid lines) for the absorbance of the MB dimer (a) and monomer (b) aqueous solutions before the addition of paper samples (0.6 cm×12.5 cm). (×) and (+) correspond to the dimer and monomer final absorption values after removal of the paper samples and replacement of the absorbed water (see Section 2). The amount of MB absorbed is obtained from the difference between the concentration into which the paper sample was dipped and the concentration when the final absorption value is projected onto the calibration curve (e.g., solid vertical line). The inset shows the amount of MB incorporated when dipping the samples into solutions with different MB concentrations for an experiment performed at lower MB concentrations than in the main figure.

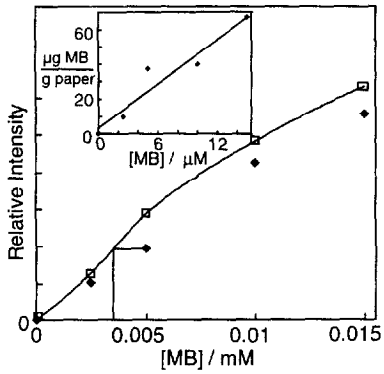


Fig. 4. Calibration curve (solid line, □) for the fluorescence emission intensity at 683 nm of MB solutions before the addition of paper samples. (◆) corresponds to the fluorescence intensity values after removal of the paper samples and replacement of the absorbed water (see Section 2). The amount of absorbed MB is calculated as described in Fig. 3. The inset shows the amount of MB incorporated by dipping samples into solutions with different MB concentrations.

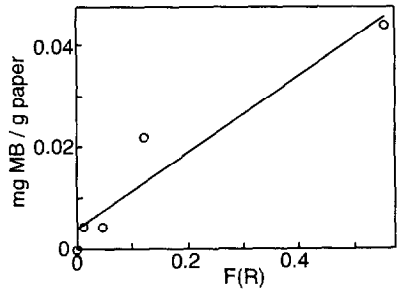


Fig. 5. Correlation of the amount of MB incorporated into paper samples calculated from solution absorbance measurements with the $F(R)$ values obtained at 667 nm.

(664 nm) and dimer (610 nm) absorbances to polynomial equations (Fig. 3, solid line). Paper samples were dipped for 1 min into MB solutions at different concentrations. Short immersion times were employed to ensure that no saturation of the paper samples occurred. The absorbance values after removal of the paper were lower than the absorbances before immersion of the samples, indicating that MB was transferred to the paper. Since the absorbance values for MB do not follow the Beer–Lambert law, the final concentration of MB in water was extrapolated from the calibration curve (Fig. 3). The mass of MB transferred to the paper is calculated from the difference between the MB concentrations before and after the paper was dipped into the solution. Similar mass values were obtained from the monomer or dimer calibration curves for the concentration range studied. The amount of MB incorporated per gram of paper increases as the paper samples are immersed into more concentrated solutions (inset Fig. 3).

Fluorescence provides an alternate method to quantify the amount of MB incorporated. A triangular cell was employed for the fluorescence measurements because the absorption of MB is quite high at the excitation wavelengths (610 nm). The dependence of the emission intensity with MB concentration does not follow a linear relationship, since at low concentrations the samples do not have a high enough absorbance to assure front-face excitation and consequently an inner filter effect may be occurring. In addition, the dimer-to-monomer ratio will be concentration-dependent, and different emission quantum yields for these species will lead to non-linear behavior. The same extrapolation procedure was employed as described for the absorbance measurements (Fig. 4).

The $F(R)$ values at 670 nm were measured for the samples employed in the quantification studies described above. For both, the solution absorbance and solution fluorescence measurements, a fairly linear relationship was observed for the calculated mass of MB incorporated and the magnitude of $F(R)$ (Fig. 5). This relationship is expected from the assumption that a decrease in the absorbance or fluorescence values is related to the transfer of MB to the paper. The use of fluorescence of paper samples to quantify the amount of MB was also explored. Although the MB emission is observable for the paper samples, no quantification of the relative amounts incorporated was possible because the emission intensities varied significantly for different positions of the same sample in the spectrometer. Clearly, fluorescence is much more sensitive to the microheterogeneity of the paper matrix than diffuse-reflectance measurements.

The incorporation kinetics of MB into the paper samples was studied to establish at which MB level saturation occurred. Paper samples were kept in 0.05 mM or 0.5 mM MB solutions, and small aliquots from these solutions were collected at different time intervals to establish how much MB had been transferred. In the case of the more diluted solution, all the MB is transferred from the aqueous phase to the paper (Fig. 6). Although we do not know the kinetic order for MB transfer, the experimental data points were fitted to an exponential function. The half-life values were determined to be 98 min and 162 min for the 4 cm×4 cm and

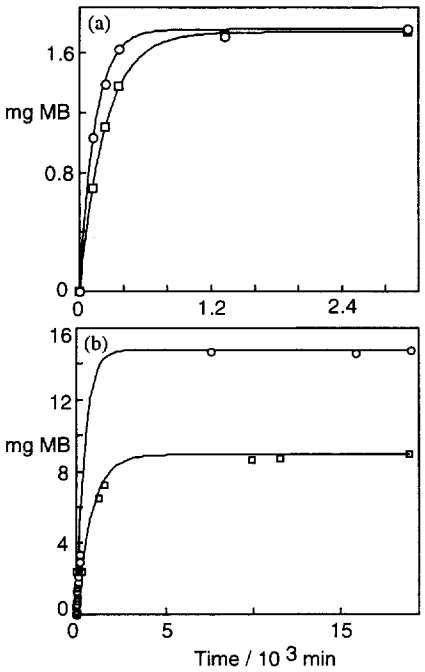


Fig. 6. Mass of MB incorporated into paper samples (O, 4 cm×4 cm, □ 2.25 cm×4 cm) when dipped into aqueous MB solutions (A, 0.05 mM; B,0.5 mM). The solid line corresponds to a fit of the experimental data to an exponential function.

2.25 cm×4 cm samples, respectively. The smaller half-life for the larger sample is expected considering that it has a larger surface area, and incorporation should be faster. The incorporation kinetics is also in agreement with the small amounts of MB that were determined to be taken up during the first minute of immersion (insets Figs. 3 and 4). Therefore, our assumption is valid that quantification was performed with no saturation of the paper with MB. When the incorporation kinetics was repeated with a different handsheet sample, longer half-lives were observed (210 min and 640 min for the large and small paper samples, respectively), suggesting that the uptake kinetics is very dependent on the handsheet structure. For this reason, the uptake kinetics can only be employed for qualitative studies. Saturation of the paper samples with MB was observed when the samples were immersed into the more concentrated MB solutions (Fig. 6). The half-lives (310 min and 580 min, for the 4 cm×4 cm and 2.25 cm×4 cm samples, respectively) also correlate with the sample size. The saturation amount of MB is directly related to the surface area of the sample, since the ratio of MB incorporated for the large and small samples (1.7) is the same as the ratio for the surface areas (1.8).

The quantification methodology developed for MB was also tested for dihydroxybenzophenone (DHBP), since this molecule has been used as a UV-screen to inhibit the photoyellowing of paper [1,2] and it is frequently employed as a standard to test the inhibition efficiency of new compounds. DHBP was transferred to paper from methanolic solutions, because it is not very soluble in water. The incorporation of this neutral molecule is much slower than observed for MB,

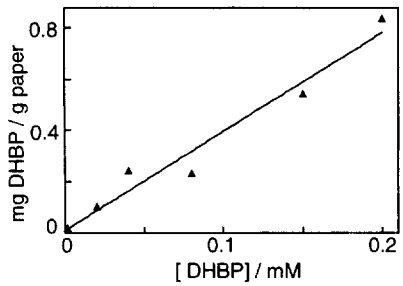
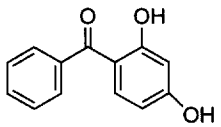


Fig. 7. Amount of DHBP incorporated by dipping samples into methanolic DHBP solutions at different concentrations.

and the paper samples were immersed for 30 min in order to transfer a similar amount of DHBP as the amount of MB incorporated within 1 min of immersion. Although paper samples pre-treated with methanol for 20 min were employed, we observed that more impurities which absorb in the same spectral region as DHBP were extracted after immersion of the samples into methanol in the absence of DHBP. For this reason, the absorbance spectra in the presence of DHBP were corrected for the absorbance due to the compounds extracted from the paper by subtracting the absorbance of a methanolic solution not containing DHBP into which a paper sample was immersed. The amount of DHBP transferred to the paper samples was calculated from the corrected absorption values at 350 nm, and varies linearly with the DHBP concentration of the immersion solutions (Fig. 7).



(DHBP)

4. Discussion

In the past, the 'drop' method has been preferred when a property of the paper was investigated as a function of loading of an additive, because it can be assumed that all the mass of the additive has been transferred to the paper matrix. However, the distribution of the added compound on the paper is very non-homogeneous as shown for MB in Fig. 2. This non-homogeneity is problematic when testing UV-screen compounds, since the photophysics of these molecules can be significantly altered upon aggregation. The non-homogeneous distribution is probably related to the different swelling of the paper for the point of application of the solution and its surroundings and the different diffusion rates of MB through the paper matrix. It is worth noting that the non-homogeneity is not apparent from diffuse-reflectance measurements. The variation of the $F(R)$ values for samples B and C in Fig. 2 are very similar, and the concentration gradients apparent in sample B were not detected in these meas-

urements probably due to the fact that a fairly large area is used to collect diffuse-reflectance data and an average reflectance is measured. These results show that fairly constant diffuse-reflectance values cannot be used to ascertain a homogeneous distribution of incorporated additives.

One of the objectives in choosing MB was its property to form dimers in solution, so that the dimerization in solution could be compared with that in the paper matrix. In this respect, a cationic molecule was chosen since its propensity for aggregation would be small because the paper matrix is negatively charged. The dimer-to-monomer equilibrium constant is very high in water (4.7×10^3 M^{-1}) [13]. For both application methods, a significant decrease of the relative dimer concentration was observed when MB was incorporated into paper, suggesting that the interaction of MB with the paper matrix is stronger than with itself. This result parallels the incorporation of MB into Nafion films [15], which are also negatively charged. In the case of Nafion, the equilibrium constant for dimer formation was determined to be 3.4 M^{-1}, which is three orders of magnitude smaller than observed in water [15]. The ability of paper to inhibit dimer formation will depend on the availability of negative sites in the paper matrix. The dimer-to-monomer ratio does not vary significantly for the different MB concentrations employed, suggesting that there are enough negative sites on the paper to which the MB monomer can bind electrostatically. Indeed, this result is in agreement with the fact that the samples were only immersed for one minute and the amount of MB incorporated is very far from the saturation limit. At this low incorporation regime the dimer-to-monomer ratio are similar for the 'drop' and 'dip' methods. However, this situation may be different at higher loadings, when for the 'drop' method saturation may occur in the more concentrated MB regions. In addition, the fact that the dimer content does not increase for the 'drop' method does not exclude aggregation at low loadings for a neutral or negatively charged compound, since these would not interact with the negative sites of the paper matrix. These results suggest that the introduction of a positive charge in the structure of UV-screen molecules minimizes aggregation.

The quantification of the incorporation of small amounts of additives cannot be achieved by weighting samples. The quantification is also not easily performed via diffuse-reflectance spectra for highly absorbing samples [5–7], which will be the case when UV-screens are employed since these inhibitors have to absorb in the same spectral region as lignin in order to be effective. For these reasons, we developed a method in which the amount of additive transferred to the paper is quantified from difference of the absorbance or fluorescence measurements of the solutions into which the paper samples were immersed. Absorbance measurements have the advantage over fluorescence that they do not depend on the particular settings of the spectrometer, such as intensity of the irradiation source and slit widths. In this respect, the calibration for absorption measurements only has to be performed a few times, whereas for fluorescence a calibration curve has to be acquired before every experiment. However, fluorescence has the advantage of being much more sensitive than absorption, and it can be employed when very low concentrations of additives are incorporated into paper.

In the case of MB, calibration curves had to be established for the absorbance measurements because dimerization occurs in aqueous solution. The concentration difference was obtained from extrapolation to the calibration curve of the absorbance after immersion of the paper. In most cases, a linear relationship is expected for the absorbance in solution and the concentrations of screen being applied. However, for each new compound, the linearity of the absorbance measurements with concentration should be confirmed. If this dependence is linear, the amount of compound transferred to paper can be calculated directly from the absorbance readings, provided a molar absorptivity value is known. In addition, it is important to establish a concentration range and immersion time that leads to large enough changes in the absorbance values for accurate measurements to be made. For example, when the absorbance differences are small, the measurements can be improved by increasing the immersion time, decreasing the initial concentration of the additive in solution or by increasing the size of the paper sample. Appropriate conditions will have to be established for each case.

We chose to work at very short immersion times for the incorporation of MB in order to establish if the incorporation did correlate with the concentration of the solution into which the paper samples were immersed. This correlation can only be tested if the paper is not saturated with the dye. Both, the absorption and fluorescence data showed a linear relationship between the amount of MB transferred to the paper and the concentration of the solution into which the paper was immersed. The same linearity was observed for DHBP. Indeed, the correlation for the latter compound is better than for MB. In the case of DHBP, the samples were immersed for 30 min, whereas for MB they were immersed for only 1 min, and the inaccuracy due to small changes in the immersion time will be higher for the latter compound. In other words, since the uptake kinetics has a higher rate for MB than for DHBP, small variations in the immersion time will lead to relatively larger variations of the amount transferred for MB than for DHBP. Finally, it is worth noting that the studies with DHBP were performed with methanolic solutions and the absorbance values had to be corrected for the extraction of impurities from paper that absorb in the same region as DHBP. Caution will have to be exercised when using any solvent other than water, and control experiments should be performed to establish the spectra of the impurities extracted by each solvent. Fluorescence measurements are not suitable for solvents that extract impurities since these can act as quenchers leading to an overestimation of the amount of compound transferred into paper.

The $F(R)$ values for the MB absorption vary linearly with the concentration of the solution into which the samples were immersed (inset Fig. 1B). In contrast to the measurements in solution, a linear relationship is expected for the paper

samples because the dimer-to-monomer ratio does not change significantly over the MB concentration range studied. For this reason, the magnitude of the $F(R)$ value is a measure of the relative amount of MB transferred into the paper. The fairly linear relationship observed for the transferred amount calculated from the absorbance data, and the $F(R)$ values of the paper samples suggests that the quantification methodology is much more precise than the weighting method, where loadings below 1% commonly resulted in errors of 100%.

From the incorporation kinetics, it is apparent that the rate of MB uptake is dependent on the surface area of the samples. When the samples were immersed in diluted MB solutions, all the MB was transferred to the paper showing that paper has a great affinity for the dye, probably due to the electrostatic interactions of the negative groups on paper with the positively charged MB. However, electrostatic interactions are not always the overriding force for incorporation, since anionic dyes have been shown to efficiently interact with cellulose [16]. The incorporation kinetics and the equilibrium conditions will be different for each additive. Saturation was observed when paper samples were immersed in concentrated solutions of MB. The amount of MB transferred corresponds to 5% of the weight of paper samples of different sizes. The half-lives measured for the uptake kinetics were not very reproducible, probably indicating that the structure of the handsheet is important to determine the rate at which the dye is transferred. However, kinetic studies are important when the uptake rate of different compounds is to be compared and to establish the saturation amount for each molecule. For example, the uptake of DHBP is much slower than for MB, since the former is a neutral molecule. Furthermore, the results with MB suggest that for loadings up to 5%, the dye will be homogeneously distributed in the paper matrix but a non-homogeneous distribution may occur at higher loadings.

In conclusion, we showed that absorption and, in limited cases, fluorescence studies of the solutions into which paper samples are dipped can be effectively employed to determine the amount of compound transferred into the paper matrix. It is important to emphasize that the methodology described is applicable for the testing of additives on handsheets, but does not relate to the application technology that eventually will have to be developed for application of these additives in a paper machine. The methodology described will be particularly useful when the effect of low loadings on a property of the paper is being studied. This will be the case for the new generation of UV-screens being developed as inhibitors against the photoyellowing of paper, since any commercially viable compound will have to correspond to a very small component ($<1\%$) of the paper matrix.

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