Photoyellowing of mechanical pulp

Part 1: Examining the wavelength sensitivity of light-induced yellowing using monochromatic radiation

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ABSTRACT *Paper made from mechanical pulp was exposed to monochromatic radiation to determine the wavelength sensitivity of light-induced yellowing. Both photoyellowing and photobleaching were observed as wavelength-dependent phenomena in the wavelength range of 260–600 nm. Transition from photoyellowing to photobleaching was observed between 340 nm and 400 nm. The increase in yellowness per available photon decreased logarithmically with the wavelength of exposure. Results also suggest that the change in yellowness index is a linear function of light intensity at a given wavelength.*

KEYWORDS
Brightness
Chromophores
Color reversion
High yield pulps
Lignins
Optical properties

High-yield pulps such as groundwood, thermomechanical pulp (TMP), chemithermomechanical pulp, and neutral sulfite pulps can potentially replace at least some of the chemical pulp used in paper products ranging from tissues and towels to printing paper. A major obstacle to the wider use of such pulps, both bleached and unbleached, is their tendency to undergo thermal and light-induced yellowing.

Most of the literature on the subject of yellowing is devoted to thermal yellowing of pulps (*1*). However, light-induced yellowing of high-yield pulps has been studied extensively (*2–11*). Yellowing and the associated brightness reversion of paper occur because chromophoric groups in the lignin absorb radiation, primarily ultraviolet (UV) radiation in sunlight (or other source), leading to several photoreactions. The mechanisms and the kinetics of these reactions are not fully understood.

Yellowing of paper

It is generally believed that the near-UV light absorbed by lignin leads to the formation of alpha-carbonyl compounds as well as *ortho-* and *para-*quinonoid structures (*3, 9-11*). Carbonyl groups absorb radiation in the wavelength region of 300–400 nm and are readily photo-excited to the triplet state. Abstraction of phenolic hydrogen by triplet carbonyl groups yields phenoxy radicals, which subsequently react with ground-state oxygen to form quinones (*6-8*).

An alternative mechanism, proposed by Gellerstedt *et al.* (*4, 12*), suggests that the formation of colored photoreactive products occurs via the quenching of the carbonyl triplet by ground-state oxygen. Singlet oxygen formed as a result of such quenching then oxidizes the phenolic hydroxyl groups to the *o-* and *p*-quinones. In both mechanisms, the generation of a carbonyl triplet state followed by energy transfer to a phenolic group plays a key role.

The yellowing reaction brought about by these mechanisms is likely to be wavelength dependent, proceeding rapidly under irradiation with short-wavelength UV light. Such a wavelength dependence has been demonstrated by Andtbacka *et al.* (*2*) and Nolan *et al.* (*11*). The former workers showed that a specific region of the sunlight spectrum (380–400 nm) was more active in promoting yellowing in both unbleached and bleached

I. Yellowness index for paper samples exposed to monochromatic radiation — constant exposure time at each wavelength[a]

Wavelength, nm	Brightness		Yellowness index[b]		Total photons/cm² × 10^{-18}		Yellowing efficiency ($\Delta YI/10^{18}$ photons cm^{-2})		L		a		b	
	U[c]	P[d]	U	P	U	P	U	P	U	P	U	P	U	P
Non-irradiated	56.00	43.64	26.86	41.59	...	...	...	...	83.24	83.17	(0.4)	0.9	8.1	20.7
260	44.56	35.29	31.84	53.69	12.5	15.1	0.398	0.801	78.09	79.64	(0.20)	1.4	13.7	25.4
	46.48	33.43	34.04	52.82	21.3	8.8	0.337	1.276	78.42	78.01	(0.10)	1.4	12.4	24.1
280	42.26	32.38	36.86	53.76	50.7	60.8	0.197	0.200	79.61	77.23	(0.40)	1.6	16.7	24.5
	39.93	31.98	38.78	54.94	79.9	38.8	0.149	0.344	76.68	76.28	(0.20)	1.7	15.0	24.9
300	34.48	31.26	43.65	55.20	109.0	92.5	0.154	0.147	74.86	76.22	0.80	2.0	19.5	25.4
	36.82	30.45	45.47	56.09	177.0	148.0	0.105	0.098	76.65	75.64	0.70	2.0	18.6	25.6
320	36.67	25.47	44.28	57.87	153.0	149.0	0.114	0.109	77.72	70.81	0.90	2.0	21.7	24.6
	37.97	29.55	48.22	57.00	237.0	232.0	0.090	0.066	77.93	74.94	0.20	3.0	19.5	26.7
340	36.08	26.34	42.80	59.52	164.0	162.0	0.097	0.111	75.75	71.99	1.30	3.2	19.0	25.7
	30.30	30.52	46.58	57.28	251.0	246.0	0.079	0.064	79.45	76.17	0.50	2.4	18.0	25.9
400	43.87	35.21	24.43	49.46	154.0	144.0	(0.016)	0.055	76.60	77.55	(0.70)	2.3	8.5	21.4
	46.85	35.27	25.27	49.65	206.0	187.0	(0.008)	0.043	78.05	77.43	(0.50)	2.1	8.2	21.5
500	51.95	36.48	21.39	48.71	297.0	154.0	(0.018)	0.046	79.93	78.29	(1.50)	0.2	7.5	22.5
	54.13	37.24	21.18	41.94	378.0	385.0	(0.015)	0.001	81.17	78.46	(1.30)	(0.2)	7.7	20.9
600	50.49	36.04	23.47	49.26	256.0	262.0	(0.013)	0.029	82.69	78.69	(0.90)	0.3	9.6	22.7
	52.94	36.23	25.38	49.45	316.0	324.0	(0.005)	0.024	80.63	79.36	(0.70)	(0.9)	8.6	23.1

[a]Negative values are in parentheses
[b]Mean of several values taken at different places in the same sample
[c]U = samples not exposed to polychromatic light before beginning experiment
[d]P = samples pre-exposed to polychromatic light.

spruce stone groundwood pulp. Nevertheless, the spectral sensitivity of light-induced yellowing of paper, particularly paper made from high-yield pulps, has not been widely explored. The report presented here is the first attempt to study the wavelength dependence of the yellowing phenomenon by exposing paper to monochromatic radiation in the UV-visible range.

The dependence of the yellowing reaction on light intensity is a related question that also is not addressed in the literature. Lebo *et al.* (*13*) studied the formation of *o*-quinonoid structures in white spruce refiner mechanical pulp upon exposure to light. Their results suggested that the amount of quinonoids formed is a function of the irradiance at a given wavelength. At 315 nm, approximately 0.08 moles of quinonoids were formed per mole of photons absorbed. The report presented here addresses the issue of light intensity and its effect on color reversion.

Control of yellowing and photoreversion is of obvious interest to the paper industry. To prevent yellowing of lignocellulosic materials, a practical approach would be to identify the wavelength range responsible for the yellowing and then control absorption of radiation at these wavelengths. This would discourage the formation of excited transient species and the subsequent photoreactions that cause yellowing.

Action spectrum

The wavelength dependence of a light-induced process such as photoyellowing or photobleaching can be conveniently expressed in terms of an action spectrum. (An action spectrum plots change in the magnitude of a light-dependent physical or mechanical property as a function of wavelength.) The data needed to generate an action spectrum for paper (or any other polymer) can be obtained by exposing the material to identical irradiances at different wavelengths for a specific interval of time and then determining the extent of reaction using an appropriate technique. Thus, the nature of a specific action spectrum depends on the absorption properties of the paper, the nature of the specific degradation process monitored, and the efficiency of the photodegradation process. It does not, however, depend upon the light source used to irradiate the sample.

In the laboratory, an action spectrum is obtained by exposing the material of interest to (a) a narrow band of radiation isolated from the dispersed spectrum of light (usually a xenon source) (*14*, *15*) or (b) a spectral line isolated from a discrete source spectrum (e.g., mercury vapor lamp) (*16*). In either case, the irradiance is a function of wavelength, as the source will not produce equal intensities at all wavelengths. This can be taken into account by varying the time of exposure at each wavelength so that a constant irradiance is obtained at each wavelength. Alternatively, the ratio of the extent of yellowing (or other reaction) to the incident intensity can be plotted, provided that the duration of the exposure at each wavelength is the same. The latter approach, however, assumes that yellowing has a linear dependence on light intensity at these wavelengths.

The present study was undertaken to determine the wavelength-dependence of yellowing in newsprint. Identification of the spectral sensitivity of paper will help in understanding the reversion characteristics of TMP under different light conditions. This, in turn, may suggest approaches for

controlling the phenomenon, leading to increased use of high-yield pulps.

Results and discussion

Samples were exposed to monochromatic radiation in two modes:

- Constant exposure time at each wavelength
- Constant number of photons at each wavelength.

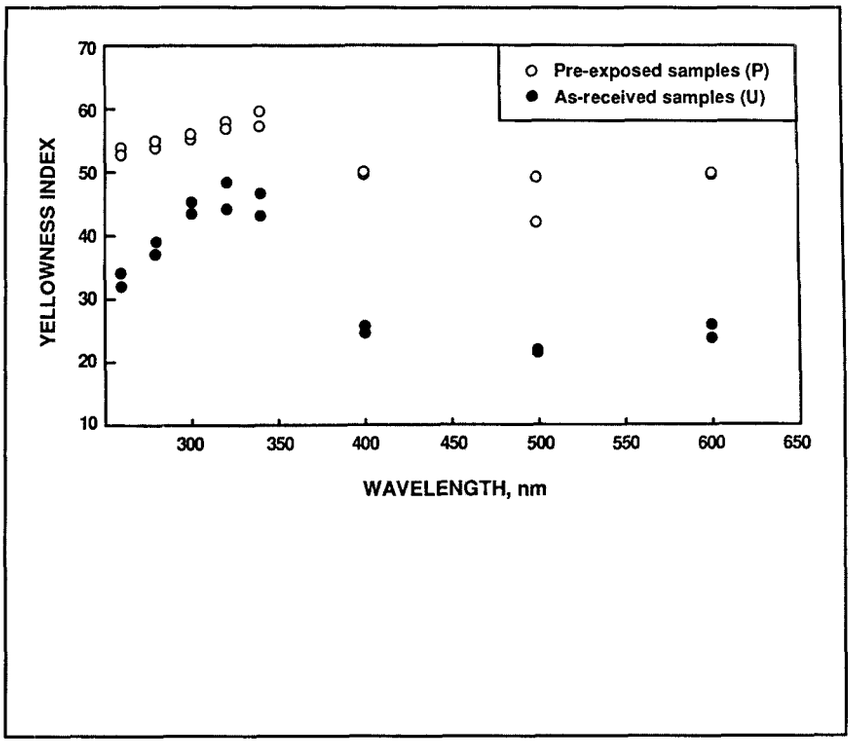
Constant exposure time at each wavelength

Table I gives the mean yellowness index (YI) at the various wavelengths for the two sets of samples used in the constant-exposure-time experiments. (Set P, pre-exposed to polychromatic light before exposure to monochromatic light; Set U, unexposed before exposure to monochromatic light.) Exposure to monochromatic radiation of wavelengths 260 nm to 340 nm resulted in increased yellowing for both sets of samples. The extent of yellowing observed is a function of both the efficiency of yellowing at a given wavelength and the irradiance obtained at that wavelength. The relative intensities of light at different wavelengths used in the study were about the same as that in a filtered xenon source, which closely simulates terrestrial sunlight. Since the irradiance at different wavelengths was not the same, the changes in YI are not direct measures of the efficiency of photoreaction. The yellowing observed at the longer wavelengths within the 260-340-nm near-UV region was higher than that observed for the shorter wavelengths within this region. However, this is a result of the much higher irradiances obtained at the longer wavelengths. Shorter-wavelength radiation is well known to be particularly efficient in bringing about discoloration and weakening of lignocellulosic materials (*2, 11*).

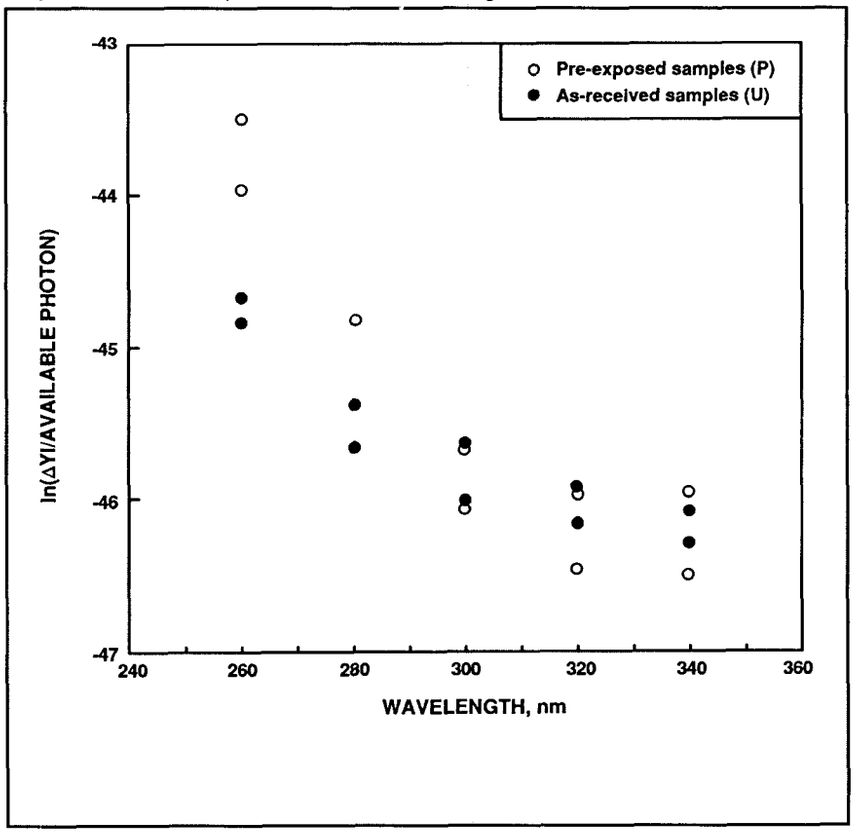
Figure 1 shows that, under the same exposure conditions, the change in yellowing for the fresh samples (Set U, previously unexposed) is significantly higher than for the pre-exposed (and thus preyellowed) samples (Set P). It is also apparent that the YI values for the pre-exposed samples are less dependent on wavelength than the unexposed samples.

The efficiency of light-induced

1. Yellowness index as a function of wavelength for samples subjected to constant exposure time at each wavelength



2. Log yellowing efficiency (ΔYI/available photon) as a function of wavelength for samples subjected to constant exposure time at each wavelength



II. Yellowness index for paper samples exposed to monochromatic radiation — constant irradiance at each wavelength*

Wavelength, nm	Photon irradiance, 10^{15} photons/cm²/s	Irradiation time, min	Yellowness index	L	a	b
280	1.23	45.16	43.78	81.53	(0.16)	22.59
	0.97	57.27	42.76	81.44	(0.12)	21.93
	1.53	36.31	41.75	81.33	(0.13)	21.45
	1.94	28.63	42.14	81.55	(0.12)	21.68
300	2.32	23.94	47.11	81.21	0.08	24.40
	2.04	27.23	44.10	81.02	(0.04)	22.64
	3.30	16.83	44.77	81.50	(0.13)	23.13
	3.80	14.61	43.17	81.39	(0.05)	22.26
320	3.31	16.83	42.85	81.67	(0.05)	22.02
	3.18	17.47	43.31	81.35	(0.25)	22.38
	4.78	11.62	42.43	81.33	(0.03)	21.64
	5.08	10.93	41.40	81.53	(0.25)	21.55
340	4.32	12.86	38.20	81.75	(0.07)	19.29
	4.24	13.10	40.02	81.88	(0.22)	20.49
	6.31	8.80	38.22	81.64	(0.06)	19.45
	6.37	8.73	37.43	82.00	(0.26)	19.34
400	4.08	13.61	22.33	83.16	0.78	10.50
	3.64	15.26	23.50	82.94	0.76	11.07
	4.56	12.18	23.88	83.15	0.85	11.31
	5.24	10.60	22.43	83.26	0.82	10.58
500	5.81	9.56	18.51	84.06	(0.52)	9.18
	5.85	9.49	18.79	84.20	(0.49)	9.39
	7.45	7.45	18.84	83.96	(0.35)	9.31
	7.43	7.47	18.14	84.20	(0.77)	9.90
600	5.10	10.89	22.26	84.22	(0.34)	11.10
	5.21	10.66	21.80	84.36	(0.45)	10.90
	6.78	8.19	21.54	84.24	(0.50)	10.81
	6.43	8.64	22.10	84.30	(0.40)	11.05

*Negative values are in parentheses. Unexposed samples had and average YI=19.3 units.

yellowing at a given wavelength can be expressed in terms of the change in YI units per available photon of light at a relevant wavelength. Note that this is not a quantum efficiency, since the irradiance refers to the available photons rather than the photons actually absorbed by the paper. The efficiency was calculated as shown in Eq. 1.

$$\text{Yellowing efficiency} = (YI_i - YI_0)/\text{total photons at each wavelength} \quad (1)$$

where

YI_0 = YI of nonirradiated sample

YI_i = YI of sample after irradiation.

Figure 2 is a semilogarithmic plot of the data on yellowing efficiency. For both sets of samples, the efficiency of yellowing dramatically decreases with increasing wavelength of the irradiating light. The efficient yellowing caused by shorter-wavelength UV radiation, observed in the present study, is also consistent with the reported UV-visible absorption characteristics of lignin. A good light absorber, lignin shows a strong absorption at about 280 nm; the absorbance rapidly decreases and flattens out in the 300-400-nm region (*17*).

Figure 2 covers the wavelength interval of 260 nm to 340 nm. (Yellowing was not observed at longer wavelengths.) The action spectrum for the yellowing of pulp shows a slight curvature. In the wavelength range relevant to terrestrial sunlight ($\lambda \geq 300$ nm and above), only three data points are available. Nevertheless, the data appear to be linear, particularly in the case of fresh (Set U) samples. A regression line fitting the data for Set U samples in the wavelength interval of 280–340 nm yielded a gradient of 0.011 (correlation coefficient 0.94 and 0.99 for the duplicate set of Set U samples). The gradient of a log-linear action spectrum is a valuable single parameter characterizing the monochromatic wavelength sensitivity of the material (*14, 15*). A gradient for the pre-exposed samples (Set P) was not calculated because of the high degree of scatter in the data.

Photoyellowing was less dependent on wavelength for Set P, and the changes in YI upon exposure were smaller than for Set U at wavelengths >300 nm. This must be attributed to surface chemical changes resulting from the pre-exposure of Set P samples to white light.

Proposed mechanisms for photoyellowing of lignin involve demethoxylation (*9, 18*) and the generation of phenoxy radicals (*6, 10*). The smaller concentration of reaction sites (possibly the phenolic groups) in the lignin fraction of pre-exposed samples was probably responsible for the diminished yellowing response when the pre-exposed samples were subsequently exposed to monochromatic light. In addition, the polychromatic-light-induced yellowing and the associated chemical changes in the Set P samples produced various chromophores that altered the light-absorption characteristics of the samples, and this may have contributed to the altered yellowing behavior as well.

Exposure to monochromatic radiation also had a significant effect on brightness. The lowest brightness for the Set U samples occurred following exposure at 340 nm, with brightness approximately 40% lower than the control. For Set P, the lowest brightness occurred at 320 nm, where brightness was about 37% less than the control. Irradiation at 400 nm, 500 nm, and 600 nm bleached the Set U samples and increased brightness. Irradiation at 600 nm, however, decreased brightness slightly.

Constant irradiance at each wavelength

Table II provides data on the change in yellowing for a set of samples exposed to the same number of photons per unit area at each wavelength. Because the spectral distribution of the polychromatic source was nonuniform, the time of exposure at each wavelength had to be adjusted to obtain a constant irradiance at each wavelength. (Adjustments were based on measured irradiance.) The samples used in this experiment were

not pre-exposed to polychromatic light, so the data can be directly compared with data for Set U samples in Table I and in Figs. 1 and 2.

The action spectra in Fig. 2 (constant exposure time at each wavelength) are based on efficiencies of yellowing, expressed as the change in YI per unit photon. This measure of efficiency assumes that the change in YI upon exposure to light of a specific wavelength is a linear function of the intensity (or photon flux), at least for the range of intensities used in this study. Data on intensity dependence of yellowing of mechanical pulps are not reported in the literature.

The second experiment, based on exposure to equal photon counts at each wavelength, avoids the need for such an assumption. Since the number of photons at each wavelength was the same, a direct plot of ln(ΔYI) vs. wavelength yields the action spectrum depicted in **Fig. 3.**

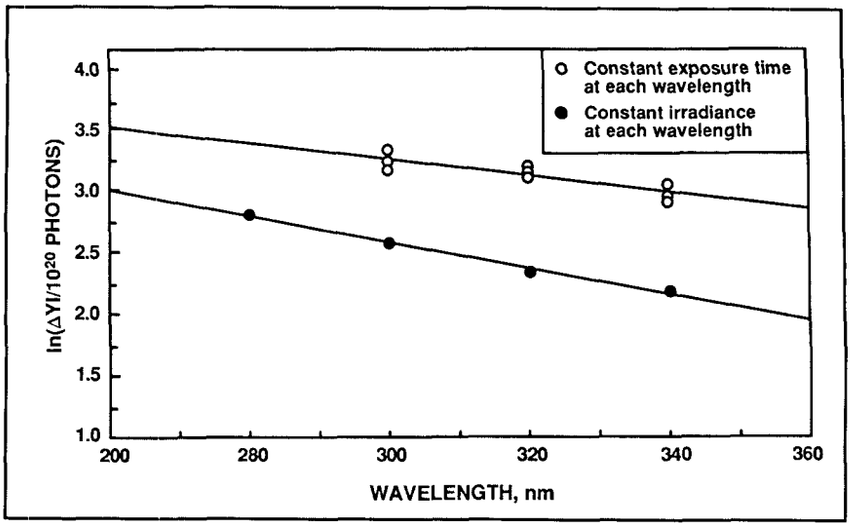
The gradients obtained using data from Table II (constant irradiance at each wavelength) can be compared with gradients obtained using data from Table I (constant time exposure at each wavelength). If the gradients turn out to be the same, this would suggest that the change in yellowness is a linear function of light intensity within the studied wavelength region. **Table III** compares the values of intercepts and gradients from both modes of exposure.

Paper has a high scattering coefficient compared with a clear plastic film. Thus paper is likely to undergo yellowing exclusively at the surface layer (*19*). The yellowing observed in the samples was localized at the exposed surface. Measurement of the reverse or back surface of the exposed samples showed that the YI for the reverse surface was only slightly higher (1-2 units) than unexposed control samples in the wavelength region of 280-400 nm. At the higher wavelengths, the changes in yellowness in both surfaces is small and about the same. However, the bleaching observed in the 500-nm exposure is obtained on both front and reverse surfaces, suggesting that the longer-wavelength radiation is able to filter through the paper. The sample exposed at 500 nm showed a YI of 18.57 units on the exposed surface and 18.15 units on the reverse surface.

YI is a specific function of the tristimulus X,Y,Z parameters of the paper. The tristimulus values can be used to calculate the parameters L,a,b, which indicate the placement of the "yellow" color obtained within a standard color space. Parameter L is a direct measure of the lightness of surface color, and its value ranges from zero (black) to 100 (white). Relatively small changes in L at the different wavelengths suggest that the increased discoloration obtained at the shorter wavelengths is not merely a change in lightness alone.

Figure 4 shows a plot of parameters a vs. b for as-received samples exposed to the same number of photons per unit area at different wavelengths. The dark circle represents the control sample. Samples exposed to shorter-wavelength radiation of <400 nm showed higher values of parameter b, while parameter a remained unchanged from the control. This suggests that exposure at these wavelengths shifted the paper toward the yellow region of the color space. Data on color measurements show the parameter a to be less sensitive to wavelength of exposure than parameter b. This suggests a change in hue with change in wavelength of exposure. Thus the differences in yellowness between the samples cannot be completely explained on the basis of different concentrations of the same chromophore. At wavelengths >400 nm, where bleaching was seen, the samples showed a change in parameter a toward the greenish region of the color space. Interestingly, the samples exposed to 400-nm radiation, where the transition from yellowing to bleaching was observed,

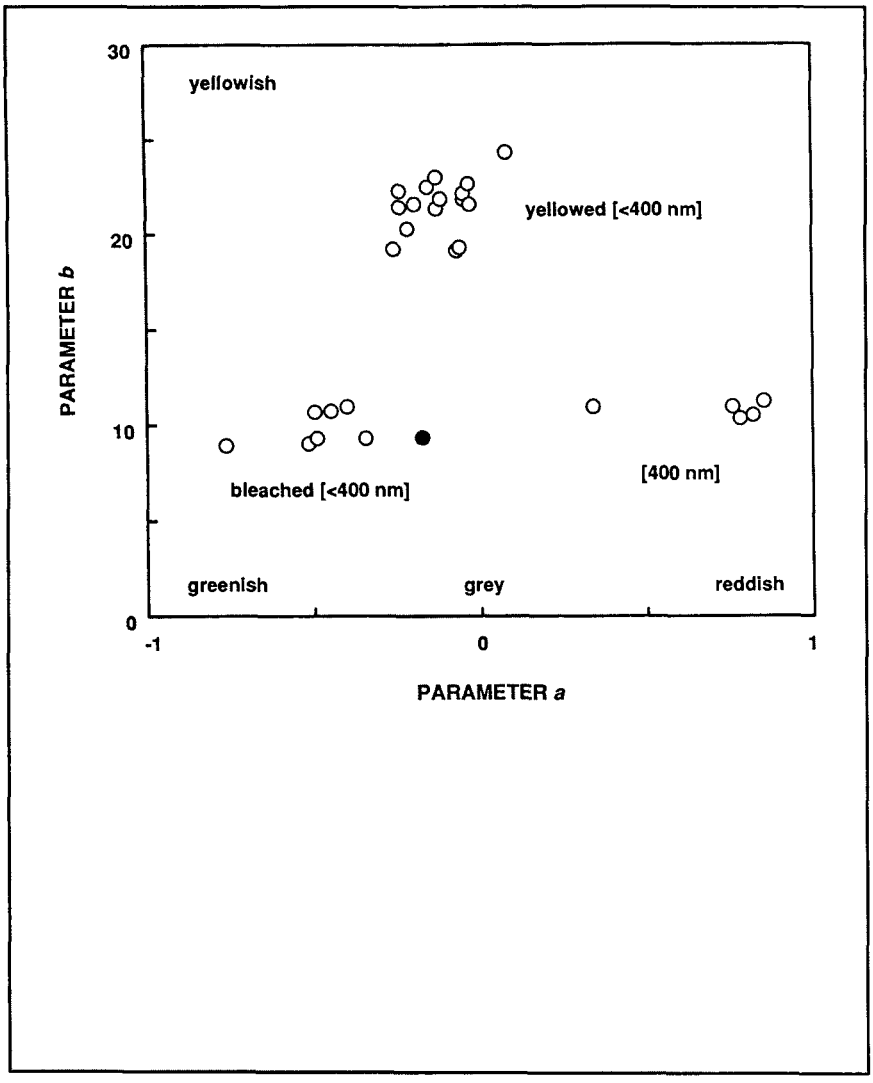
3. Log yellowing efficiency ($\Delta YI/10^{20}$ photons) as a function of wavelength for Set U samples from both experiments



III. Intercept and gradient of action spectra for photoyellowing under constant-photon and constant-duration exposure conditions (Set U samples)

Sample	*Intercept*	*Gradient*	r^2
	Constant-photon exposure (1 x 10^{20} photons)		
1	6.23	0.010	0.99
2	4.58	0.005	0.88
3	5.48	0.007	0.96
4	5.26	0.007	0.94
Average	5.76	0.011	0.99
	Constant-duration exposure (10 h)		
Average		0.011	0.94-0.99

4. Correlation between *a* and *b* parameters for samples subjected to constant irradiance at each wavelength. ● = unexposed control.



showed the *b* parameter at the original level of the control sample, while the *a* parameter increased and moved toward the reddish region.

An interesting observation in Table I (constant exposure time at each wavelength) is the behavior of samples exposed to radiation of wavelengths in the interval 340-600 nm. Especially in the case of the two longest wavelengths—500 nm and 600 nm—paper was not yellowed as a result of exposure. In fact, the Set U samples yielded YI values significantly lower than their initial values for exposures at 500 nm and 600 nm. The data in Table II (constant irradiance at each wavelength) show that the sample exposed to 500-nm radiation also was bleached. Radiation of longer wavelength does not cause yellowing and actually photobleaches the paper by promoting other reactions that destroy existing yellow species. In the pre-exposed, yellowed samples (Set P), the trend is about the same as with the Set U samples. At the longest wavelengths, little or no additional yellowing occurred, although no photobleaching was observed.

Paper produced from unbleached (or partially bleached) mechanical pulps, such as newsprint, contain significant amounts of red-absorbing chromophores that can be bleached by white light (*5*). Photobleaching of pulps has been reported by Andtbacka (*2*) and others (*11*). No acceptable mechanism, however, has been forwarded to explain the light-induced bleaching of pulp.

Conclusion

The yellowing of paper that occurs upon exposure to sunlight is the result of photoreactions induced mainly by the shorter-wavelength near-ultraviolet region of the visible spectrum. The logarithmic nature of the action spectra presented in this report indicates that newsprint is particularly sensitive to the yellowing caused by short-wavelength radiation. Screening by glass and certain types of plastic materials should provide some protection from these wavelengths and the yellowing that they cause.

A photobleaching reaction occurs at longer wavelengths of 500-600 nm. This suggests that the yellowing that occurs under exposure to white light is the net result of competing yellowing and bleaching processes. Given the low efficiency of the photobleaching process, photoyellowing is likely to be the predominant result when paper made from high-yield pulps is exposed to sunlight. The photoyellowing reaction is primarily a surface reaction with little, if any, shorter-wavelength light penetrating the paper to affect the reverse surface.

Experimental

Paper samples

The newsprint paper used in this study had a basis weight of 48 g/m^2 and was made from unbleached loblolly pine (*Pinus taeda* L.) TMP pulp. The paper was obtained from a commercial supplier.

Pre-exposure to polychromatic light

Two sets of samples were used. One set (Set U) was used as received and irradiated with monochromatic radiation; a second set of samples (Set P) was pre-exposed to white light and yellowed before being exposed to monochromatic radiation. Pre-exposed samples were included in the study to determine the spectral sensitivity of any photobleaching process that might be observed when the samples were exposed to monochromatic light (*2, 11*). Pre-exposure of Set P was conducted with a Spectra Energy 1000-W short-arc xenon lamp equipped with a borosilicate filter. Samples were placed horizontally on a water-cooled black metal plate 68 cm from the light source. The light was reflected via a UV-reflecting

mirror placed at an angle of 135° to the plane of the incident ray. A mean irradiation of 90 mW/cm² (approximately equivalent to terrestrial sunlight at unit air mass) was incident upon the sheet with an approximate variation of about ± 10% in intensity over the entire exposure area. The exposure was carried out at 50% RH and ambient temperature (23 ± 1°C). Immediately after exposure, the sheets were wrapped in aluminum foil and placed in a black envelope.

Irradiation with monochromatic light

Samples were irradiated with monochromatic light using the large spectrograph at the National Institute for Basic Biology in Okazaki, Japan. The spectrograph was equipped with a 30-kW xenon short-arc lamp with water-cooled electrodes. Radiation from this source was filtered through a distilled-water filter to exclude heat and dispersed into a spectrum using double-blazed plane grating with 1200 lines/mm. By placing the samples at an appropriate position on a 10-m-long focal curve, sheets were exposed to monochromatic radiation of the desired wavelength. A set of sharp cut-on filters was used to minimize the incidence of uncontrolled light on the sample. Exposures were generally carried out at 23°C.

Before exposing the sheets to monochromatic light, the equipment was calibrated using a mercury vapor lamp. The stability of the source was continually monitored at preselected wavelengths during the exposure experiments. A long-term variation of less than 2% in the irradiance over the 10-h exposure period was generally obtained with the system. A detailed description of the spectrograph and the instrumentation used to measure irradiance has been previously reported by Watanabe *et al.* (*20*).

Immediately after exposure, the samples were placed in a black paper envelope and stored at ambient temperature. The pre-exposed samples were exposed to monochromatic light within about a week of initial exposure. The yellowness index (YI) for all samples (exposed and control samples) was measured about one week after exposure.

Measurement of yellowness index

YI was determined in accordance with ASTM D1925-70 using an Elrepho 2000 (Ahiba Datacolor) reflectance meter with an integrating sphere. Yellowness and L,a,b values of samples were measured using an X-Rite 968 colorimeter. A standard calibration tile was used as the white standard, whose percent reflectance was measured between wavelength 400 nm and 700 nm against CPPA-calibrated Japanese Opal Glass EW-24, which excluded specular light using a 27-mm aperture with 0°/diffuse geometry. Each sheet was backed by a pile of sheets thick enough to be opaque. For both the opal-glass standard and for the sheets, the UV component of the incident light (98%) was included, but the specular component of the reflected light was excluded during the measurement. YI was calculated based on CIE standard illuminant D_{60} and CIE 10° standard observer viewing (CIE-SO) from the tristimulus values of X, Y, and Z relative to the source C using Eq. 2.

$$YI = [(1.28X - 1.06Z)/Y] \times 100 \quad (2)$$

Several values of YI were obtained from different sections of each exposed sheet. These values were used to obtain a mean value for YI. Reproducibility of measurement was generally better than ±0.50 units.□

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