

Browning of paper

Fluorescence changes accompanying the discolouration of artificially aged paper

The occurrence of fluorescence in association to brown stains on paper, for instance, around iron-gall ink writings, at the tideline in water stains, and on foxing stains is a frequently observed phenomenon in paper conservation practice. In order to investigate the relationship between fluorescence and discolouration of paper, changes in the fluorescence of two additive-free papers submitted to artificial ageing have been monitored both visually and spectroscopically, and the results were compared with the information obtained from the UV-VIS reflectance spectra of the same samples by using colour analysis. Both papers exhibited an increased overall fluorescence upon artificial ageing, which shows a single emission maximum at 445–450 nm (excitation wavelength = 365 nm). The increase in fluorescence intensity and the discolouration of the test papers occurred simultaneously. Whereas for one of the papers (cotton cellulose) no change in the spectral distribution of fluorescence was observed, the formation of new fluorescent species was indicated in the fluorescence spectrum of the other sample (bleached chemical pulp), which displayed a perceptible shift in chromaticity. The visually observed colour changes in the fluorescence of the investigated papers under ultraviolet illumination may therefore be attributed to a combined effect of discolouration and fluorescence. The changes in intensity and colour of fluorescence of paper observed in this study reproduce, to some extent, the changes in fluorescence associated to iron-gall inks, wet-dry interfaces, and foxing agents, which are reported in the literature. These results support the idea of a possible common underlying mechanism to explain the fluorescence of paper under the influence of different degrading factors.

Fluoreszenzerscheinungen sind oft beobachtete Phänomene in Verbindung mit Verbräunungen auf Papier, beispielsweise um Eisengallustintenschriftzüge, an den Rändern von Wasser- und auf Schimmelflecken. Um die Beziehung zwischen Fluoreszenz und Papierverfärbung zu untersuchen, wurden die Fluoreszenzveränderungen von zwei additivfreien Papieren im Zuge einer künstlichen Alterung optisch und spektroskopisch verfolgt; die Ergebnisse wurden mit den Informationen verglichen, die über UV-VIS-Reflektionsspektroskopie mittels Farbmessung von den gleichen Proben ermittelt wurden. Beide Papiere zeigen einen Anstieg der Fluoreszenz bei einem Emissionspeak bei 445–450 nm (Anregungswellenlänge = 365 nm). Der Anstieg der Fluoreszenzintensität und die Verfärbung der Testpapiere durch die künstliche Alterung verlaufen gleichzeitig. Während bei einem der Papiere (Baumwollcellulose) keine Veränderung in der spektroskopischen Verteilung der Fluoreszenz beobachtet werden konnte, bildete sich bei der anderen Probe (gebleichter Zellstoff) eine neue fluoreszierende Substanz im Fluoreszenzspektrum, die sich auch in einer Farbverschiebung zeigt. Die bei den untersuchten Papieren optisch unter ultraviolettem Licht beobachtete Farbveränderung der Fluoreszenz könnte deshalb eine Folge eines kombinierten Effekts aus Verfärbung und Fluoreszenz sein. Die in dieser Studie beobachtete Intensitäts- und Farbveränderungen spiegeln bis zu einem gewissen Grade die Fluoreszenzveränderungen im Zusammenhang mit Eisengallustinten, Naß-trocken-Grenzflächen und Schimmelflecken wider, die in der Literatur beschrieben werden. Diese Ergebnisse unterstützen die Theorie eines möglichen gemeinsamen Mechanismus zur Erklärung der Papierfluoreszenz durch verschiedene Abbaufaktoren.

The development of brown stains on the surface of paper is almost invariably an undesired alteration to paper objects of intrinsic value. Discolouration has explicit aesthetic implications to those objects, and it indicates that chemical changes have occurred. Examples of brown discolouration are the irregularly shaped stains generally referred to as “foxing”, iron-gall ink corrosion brown discolouration developing around ink lines and migrating into the paper upon ageing, and the permanent brown lines (tidelines) formed at the interface between wet and dry areas due to the local action of water evaporating from paper.

A common feature associated to brown stains, irrespective of their origin, is that discoloured areas frequently fluoresce when examined under ultraviolet radiation. Enhanced fluorescence has been observed in all the three examples mentioned before: in connection with foxing stains (Hey et al 1992: 244; Cain and Miller 1982: 54; Gallo and Pasquariello 1989: 139; Choisy et al 1997: 131), around iron-gall ink writings (Reißland 1997; Baker 1983: 159), and at the tideline in water stains (Hutchins 1983: 57; Dupont 1994; Eusman 1995; Hofenk de Graaff 1994: 21). Fluorescent areas are usually lar-

ger than the visibly stained areas, and in some cases fluorescence occurs without any visible discolouration. Fluorescence seems to appear before discolouration, to increase in intensity and change colour from white-blue to green-yellow as degradation proceeds. When discolouration has developed into dark brown stains, fluorescence can no longer be observed. The common pattern in these observations suggests a common underlying mechanism at some level for all these cases.

Paper conservators often use UV-light as a diagnostic tool that is simple and generally available. The interpretation of visually observed fluorescent patterns seen under UV-illumination, however, is not straightforward. Is it generally true that fluorescent areas always develop into brown discolourations? Can further conclusions be drawn from observations of specific perceived fluorescence colours?

There is still a lack of knowledge on the precise mechanisms causing the phenomena, both on the chemistry level and on the spectral level. This paper is the first in a series where the question of a common mechanism for fluorescence preceding browning will be investigated. In this study two types of additive-free papers were subjected to accelerated

ageing conditions. The changes in fluorescence and the discolouration of the test papers were assessed visually and checked against their spectral behaviour. At this stage no attempt has yet been made to elucidate the chemical structures of possible fluorescent species involved. Instead, this study aims at describing the fluorescence and browning phenomena at a spectroscopic level. Consistency of spectroscopic data is checked against visual observations with colour calculations.

Theory

Fluorescence

Fluorescence is a process of absorption and re-emission of light by molecules. Typically a photon with higher energy (shorter wavelength) will be absorbed by a molecule, in a process called excitation. By internal processes a part of the energy acquired by the molecule in the excitation process will be dissipated. As a result, when the excited molecule re-emits light to return to its original (ground) state, most or all of the re-emitted light will be shifted to lower energies than the absorbed light. For a single fluorescent molecule the spectral distribution of emitted light is related to the chemical structure of that molecule, and it is independent of the excitation wavelength. The fluorescence intensity will depend on the concentration of the fluorescent molecule, as well as on the excitation wavelength. Besides that, one should be aware of the fact that fluorescence is generally much more sensitive to the molecular environment than light absorption processes. The sensitivity of fluorescence to the environment is a consequence of the relatively long time a molecule stays in an excited state before de-excitation (typically 10^{-9} to 10^{-8} s). On this timescale several processes can occur such as collisions with other molecules and local changes of the conformation of molecules, which lead to de-excitation without the emission of light (Cantor 1980: 433).

Colour perception

A general situation of an individual in a darkened room judging the intensity and colour of fluorescence is depicted in fig. 1. Light from an UV-lamp is shone onto the object and surroundings. The light is characterised by a spectral distribution that is ideally a narrow wavelength band of UV-radiation.

The intensity and spectral distribution of the fluorescent light which is returned from the object to the eye depends on the types and concentrations of fluorescent species present in the object, on the spectral characteristics of the light source, and on the efficiency of the fluorescence process.

An accurate visual assessment of the colour of fluorescence is not straightforward. One reason that is hard to avoid is the interpersonal variation of colour judgement at relatively low intensities. On the other hand there is often a lack of precise information about various aspects of the surroundings that can affect the colour judgement. The spectral distribution of the light that reaches the object is simply unknown or might contain also some visible light. This is either due to

non-ideal filters used in the lamp or due to the presence of fluorescent materials in the darkened room. In case the spectral distribution of the light that reaches the object contains a visible component, the reflectance properties of the object will influence the perceived colour of fluorescence.

Although straightforward it is perhaps not always realised that protective glasses used by the observer will block certain parts of the fluorescence spectrum and distort the perceived colour to a larger or smaller extent. The spectral distribution that finally reaches the eye now is determined by the product of the fluorescence spectrum and the transmission curve of the glasses.

The colour perception induced by the spectral distribution of the fluorescent light emitted by the object can be expressed in terms of the CIE-chromaticity co-ordinates (x, y), and the CIE-luminance Y . (See for example Judd 1975).

Experimental

Test samples

Two papers manufactured respectively from cotton linters and from a bleached chemical wood pulp have been used as test specimens in order to compare the ageing behaviour of paper made from these two different fibre sources.

Whatman No.1 qualitative filter paper, a cotton linters paper with high α -cellulose content (87 g/m²; ash content 0.06 %), and an additive-free bleached sulphite softwood pulp paper (78 g/m²; ash content 0.45 %, manufactured by Papierfabriek Schutt, Heelsum, The Netherlands) were used in this study without further purification. Chemical characterisation of the latter by infrared spectroscopy showed the presence of hemicelluloses (Havermans et al 1994: 62).

Artificial ageing experiments

The test samples were submitted to artificial ageing at 90 °C and cycling relative humidity between 80% and 35%, each step having a duration of 3 hours. Ageing periods of 0, 3, 6, 12, and 18 days have been used in this study. The samples (15 x 15 cm) were hung inside a Vötsch Industrietechnik VC 0020 programmable climate chamber, and a Heraeus TSI software was used to program the ageing cycles.



1 Typical situation of an object examined under UV-illumination by a conservator.

Visual examination under UV-illumination and daylight

Visual examination of the test samples was carried out under daylight and in a dark room under ultraviolet illumination at 365 nm. The ultraviolet source used was a Spectroline® Super High Intensity black light lamp model SB-100/F (Spectronics Corporation, USA). For eye protection protective glasses were used whose transmission spectrum was characterised by UV-VIS spectroscopy.

UV-VIS reflectance spectroscopy

UV-visible reflectance spectroscopy was carried out by using a Perkin-Elmer UV/VIS/NIR spectrometer equipped with an integrating sphere. Infinite thickness reflectance measurements were performed on piles containing about 11 layers of paper.

Fluorescence spectroscopy

Surface fluorescence spectra were acquired using a Shimadzu RF-5001PC fluorescence spectrometer. All spectra were re-

corded with excitation at 365 nm, which is the nominal wavelength for commercially available ultraviolet lamps commonly used for examination by conservators. Since only the visible fluorescence emission is of interest to the paper conservation practice, emission spectra were recorded in the region 380-700 nm. The samples were clamped using an emission-free quartz plate, and the measuring geometry was chosen in order to minimise scattering (excitation using an incident angle of 53° with respect to the sample surface; fluorescence measuring angle of 37° with respect to the sample surface). Filters were used on both the exciting and the emission sides in order to remove stray light in the exciting light (U-340) and to remove scattered light from the sample surface (UV-39).

Results and discussion

Visual examination under UV-illumination and daylight

Examination of the artificially aged papers under daylight showed a slight, gradual discolouration as a result of ageing. Discolouration of Whatman No.1 was less discernible than that experienced by the chemical pulp paper. Although the difference in colour between the unaged and the 18 day-aged papers was clearly perceptible, differences between consecutive ageing intervals were not so easily observable in both cases.

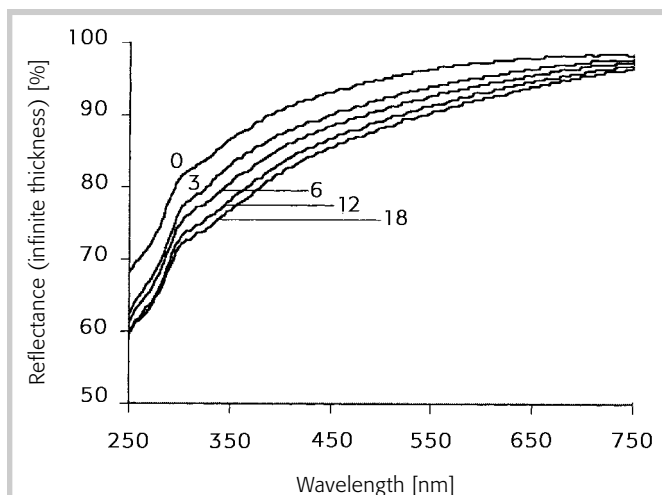
Examination of the test samples under ultraviolet radiation at 365 nm and through protective glasses showed that the fluorescence of the chemical pulp paper changed from a low-intensity bluish colour to a more intense, greenish (3 and 6 days aged) and later on yellowish colour (12 and 18 days aged) in the course of artificial ageing. Whatman No.1 filter paper exhibited a very faint bluish initial fluorescence that developed into a somewhat more intense and yellowish emission at the end of the ageing treatment. Unlike the chemical pulp paper, no clear intermediate stage in the development of fluorescence could be observed for Whatman No.1.

UV-VIS reflectance spectroscopy

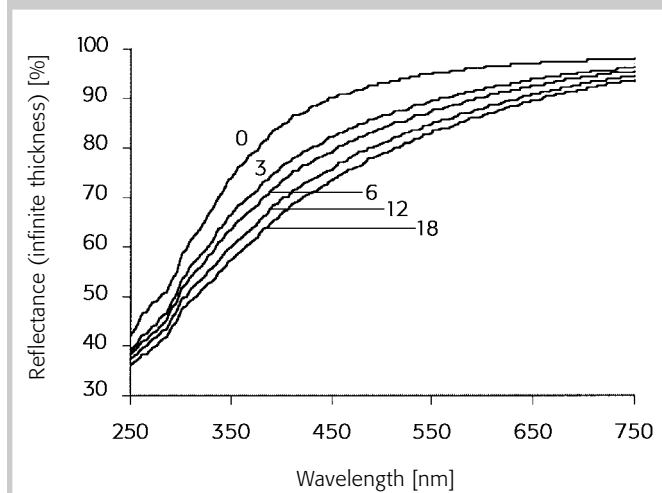
UV-visible reflectance spectra of the test samples are shown in fig. 2 and 3. For both types of paper the reflectance decreases with ageing, showing the highest change during the first three days of artificial ageing.

Fluorescence spectroscopy

The fluorescence emission spectra of the investigated paper samples at different ageing times are shown in fig. 4 and 5. The series of spectra for both papers show an initial fluorescence under illumination with ultra-violet light at 365 nm at the start of the accelerated ageing treatment. This is believed to be due to either processing or natural ageing (Toner and Plitt 1962: 681). Fluorescence increases rapidly in the beginning of the ageing treatment and then levels off or decreases slightly for longer ageing periods. The spectra for the two types of paper have both their single emission maximum at



2 UV-VIS reflectance spectra of Whatman No.1 artificially aged for 0, 3, 6, 12 and 18 days. A decrease in reflectance upon ageing is seen.



3 UV-VIS reflectance spectra of the chemical pulp paper artificially aged for 0, 3, 6, 12 and 18 days. A decrease in reflectance upon ageing is seen.

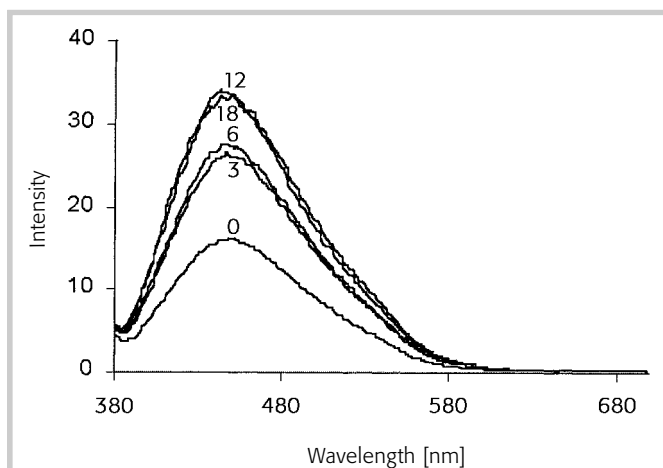
445-450 nm. The position of this maximum coincides relatively well with the emission maxima at 420-440 nm reported by Choisy et al (1997: 131) for a large number of foxing stains using an excitation source at 365 nm.

The overall line shapes of the fluorescence emission spectra for the ageing series of both Whatman No.1 and the chemical pulp paper all show a remarkable similarity. The normalised spectra shown in fig. 6 and 7 indicate that, for Whatman No.1, no significant change in the shape or position of the emission spectrum during ageing has occurred, whereas the chemical pulp paper shows a small but systematic development of a shoulder at the right hand side of the emission band. The presence of one main single emission band with a constant line shape for the two papers during the whole ageing process strongly indicates that the observed fluorescence in both cases is caused by one major single fluorescent structure. The development of a shoulder in the fluorescence spectrum of the chemical pulp paper suggests the formation of secondary fluorescent species. A possible explanation for the

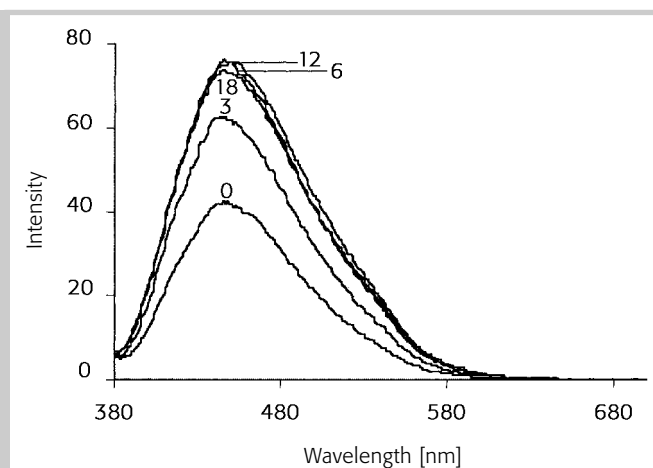
formation of secondary fluorescent species within this paper could be the presence of hemicelluloses or even some residual lignin (Tylli et al 1992: 117). However, by comparing the normalised fluorescence spectra of the chemical pulp paper with those of Whatman No.1, it was observed that the latter contain an identical shoulder, which indicates that the changes in fluorescence of the chemical pulp paper are not related to the presence of residual wood constituents.

Colour analysis

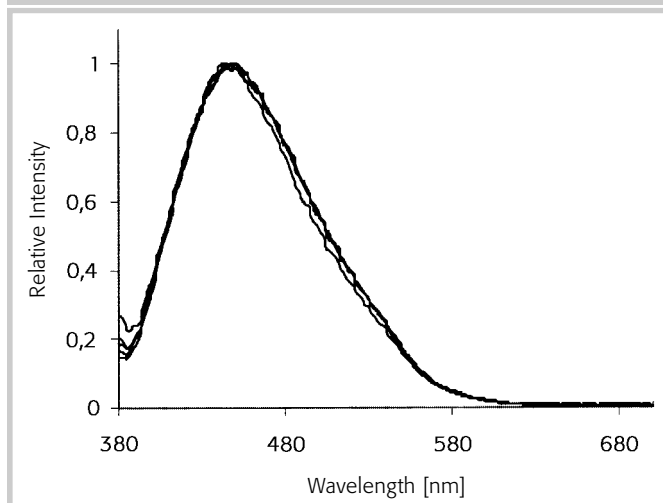
The results of the visual examination can be compared to the spectral information contained in the UV-VIS reflectance spectra and the fluorescence spectra by means of colour analysis. The CIE-chromaticity co-ordinates (x, y) have been calculated for the UV-VIS reflectance spectra using as illuminant standard daylight D65 (Nassau 1998: 45). The chromaticity co-ordinates for the fluorescence were calculated both directly from the spectra and after correcting for the transmission of the protective glasses used by the observers. The results for



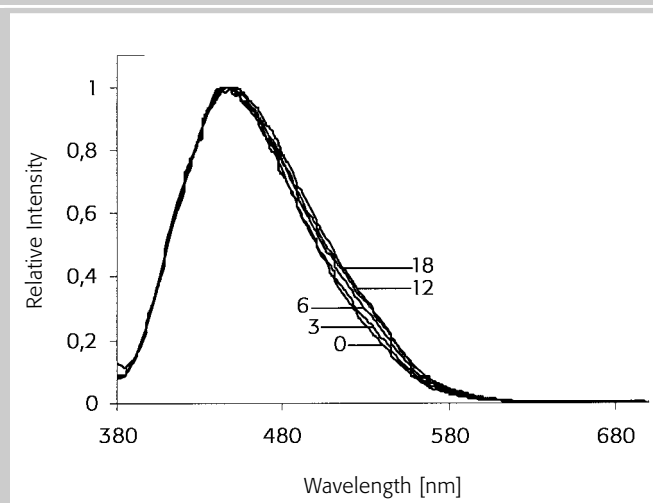
4 Fluorescence emission spectra of Whatman No.1 artificially aged for 0, 3, 6, 12 and 18 days. Excitation wavelength: 365 nm. An increase in emission intensity upon ageing is seen



5 Fluorescence emission spectra of the chemical pulp paper artificially aged for 0, 3, 6, 12 and 18 days. Excitation wavelength: 365 nm. An increase in emission intensity upon ageing is seen.



6 Normalised fluorescence emission spectra of Whatman No.1 artificially aged for 0, 3, 6, 12 and 18 days. Excitation wavelength: 365 nm. No change in the spectral distribution upon ageing is seen.



7 Normalised fluorescence emission spectra of the chemical pulp paper artificially aged for 0, 3, 6, 12 and 18 days. Excitation wavelength: 365 nm. The development of a shoulder at the right hand side of the emission band upon ageing is seen.

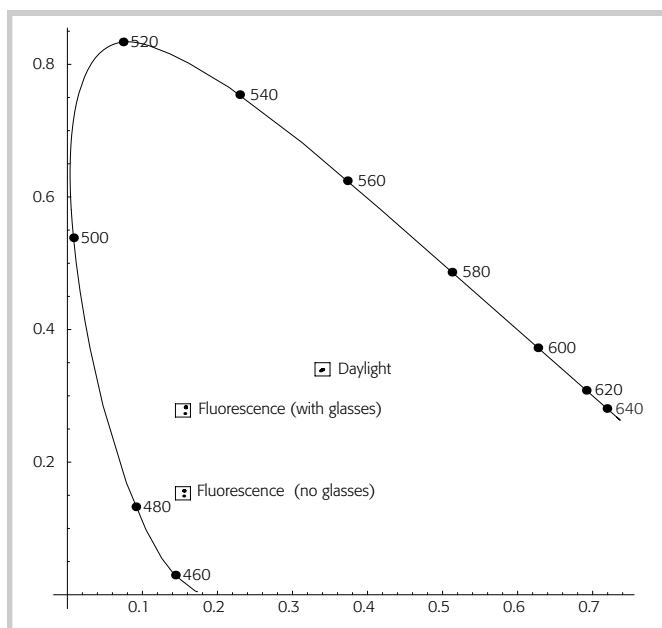
Whatman No.1 and the chemical pulp paper are plotted in the CIE-chromaticity diagrams shown in fig. 8 a and 9 a. The chromaticity co-ordinates of the subsequent spectra are relatively close. Trends for the data points are better observed in the detail plots (fig. 8 b-d and 9 b-d).

The calculated chromaticity co-ordinates for both the daylight reflection of Whatman No.1 and the chemical pulp paper do show a small but systematic shift towards the yellow region of the diagram. The magnitude of this colour shift for Whatman No.1 is close to a just noticeable chromaticity difference as reported by MacAdam (1942: 247). The magnitude of the shift towards the yellow is larger for the chemical pulp

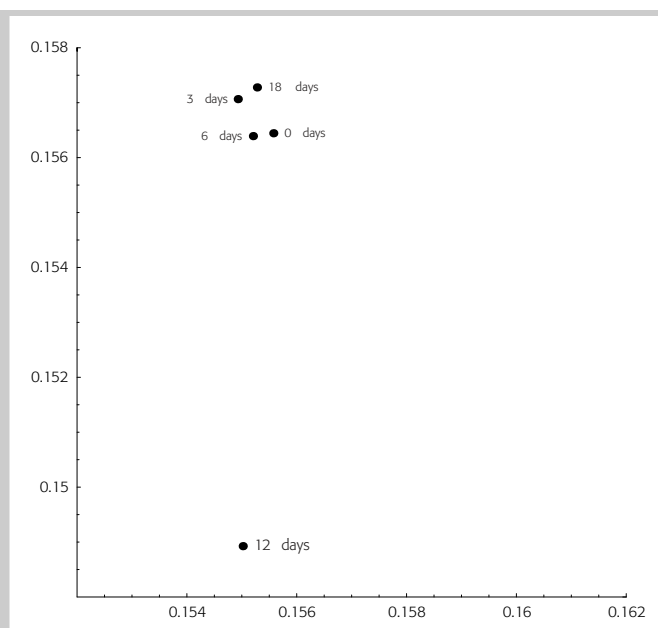
paper data points. The trend for calculated chromaticity co-ordinates is consistent with the observed discolouration.

The calculated chromaticity co-ordinates for the fluorescence of Whatman No.1 are scattered a bit, but do not show any systematic trend. This fact is in contradiction with the visually observed colour shift from faint blue to yellow. Possibly the yellowing of the paper, as evidenced by the UV-VIS spectra, became visible under UV-illumination due to the presence of some blue light generated by the UV-source. The colour calculations from the fluorescence spectra assume a pure UV-source and do not take into account this non-ideality.

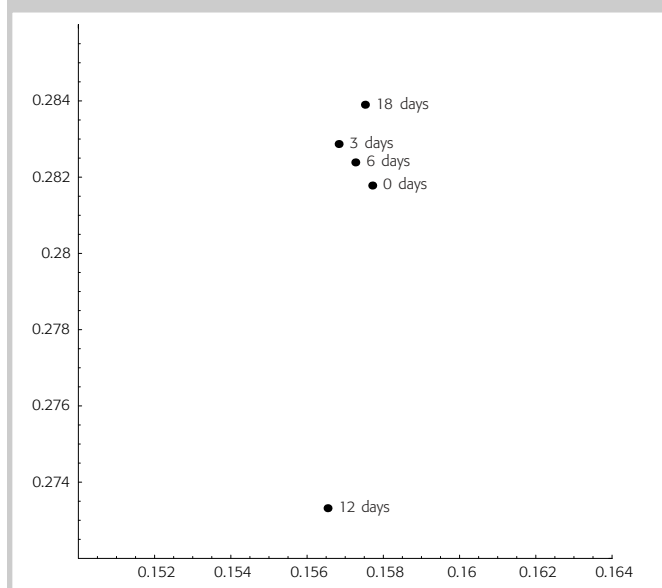
The calculated chromaticity co-ordinates for the fluores-



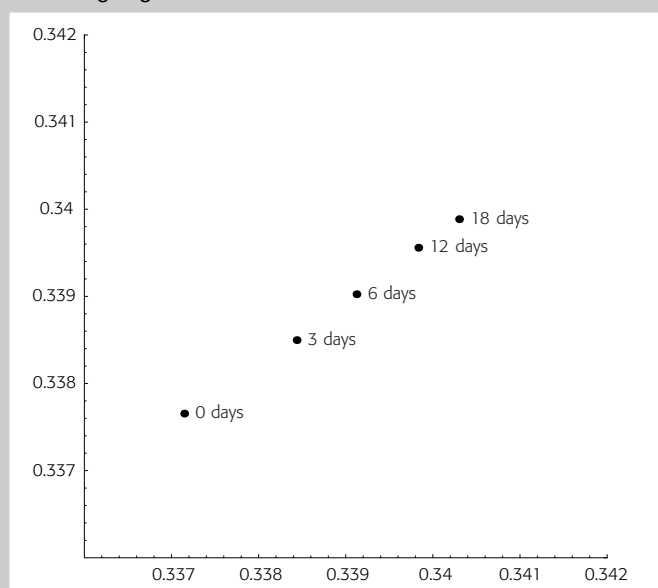
8 a Overview of the CIE-chromaticity diagram for Whatman No.1 paper showing the chromaticity co-ordinates at different ageing times and different illuminations.



8 b Detail of the CIE-chromaticity diagram showing the chromaticity co-ordinates of Whatman No.1 under UV illumination at 365 nm. No clear correlation with ageing is seen.



8 c Detail of the CIE-chromaticity diagram showing the chromaticity co-ordinates of Whatman No.1 under UV illumination at 365 nm after correction for the transmission characteristics of the protection glasses used. No clear correlation with ageing is seen.



8 d Detail of the CIE-chromaticity diagram showing the chromaticity co-ordinates of Whatman No.1 paper under D65 daylight illumination. A small but systematic trend towards the yellow is seen.

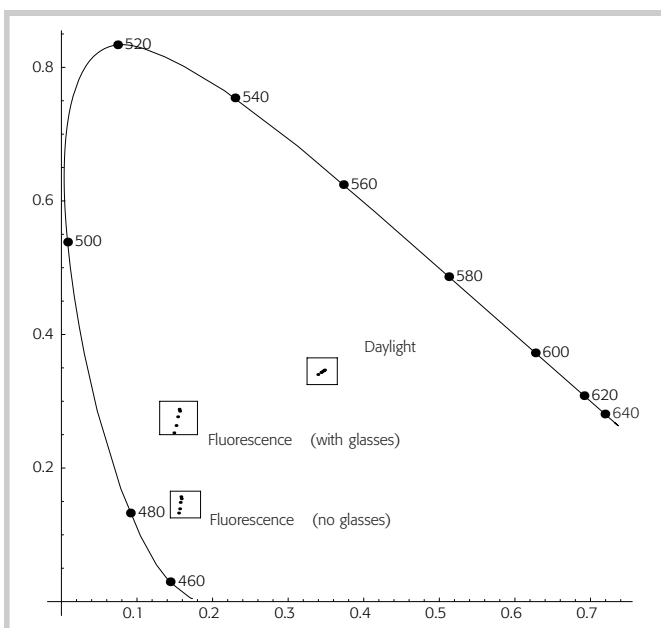
cence of the chemical pulp paper do show a systematic chromaticity shift whose direction is consistent with the visually observed changes from a bluish to green and finally yellow fluorescence. Based on the results obtained from the colour analysis for Whatman No.1 it is expected that the perceived colour changes are not solely due to changes in the fluorescence emission spectrum of the chemical pulp paper, but that they have been enhanced to some extent due to the presence of some blue light.

Conclusions

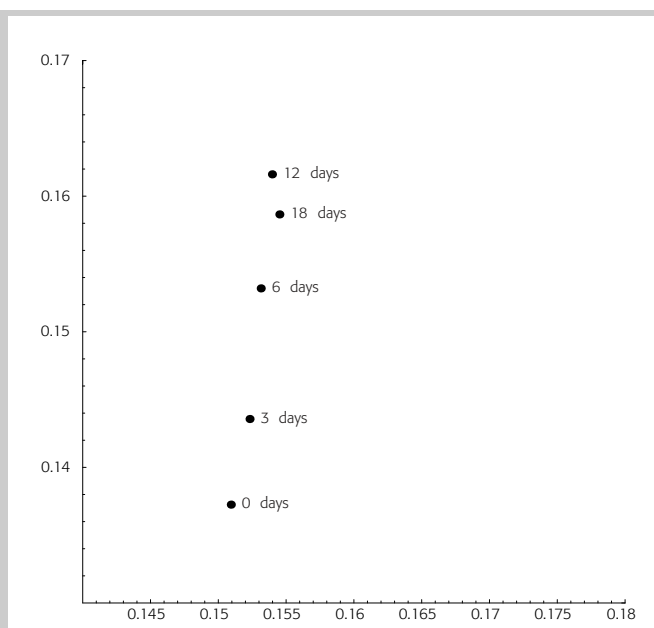
Paper submitted to ageing conditions that promote hydroly-

tic and oxidative degradation of cellulose exhibits increased overall fluorescence, which shows a single emission maximum at 445-450 nm (excitation wavelength = 365 nm). Fluorescence increases rapidly in the beginning of the ageing treatment and then levels off or decreases slightly for longer ageing periods. The initial fluorescence observed for the unaged test samples is believed to result from the processing and natural ageing of cellulose.

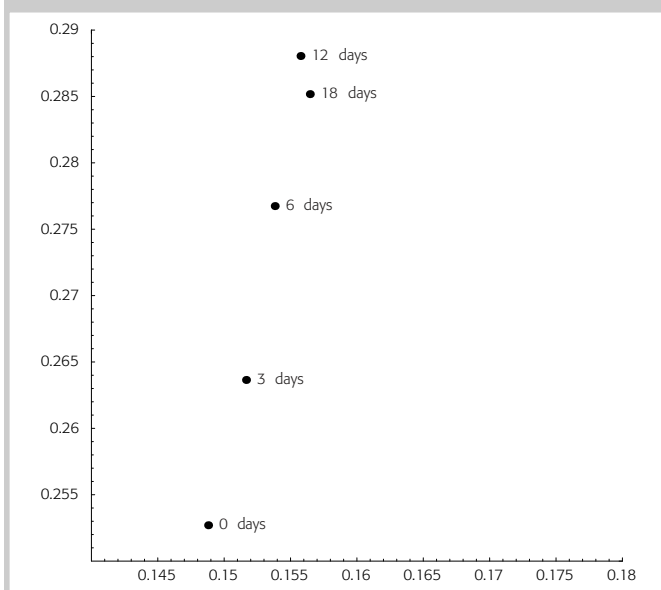
A discernible change in the colour of fluorescence of the chemical pulp paper examined under UV-illumination at 365 nm with protective glasses was observed, which developed from a low-intensity bluish colour into a more intense green



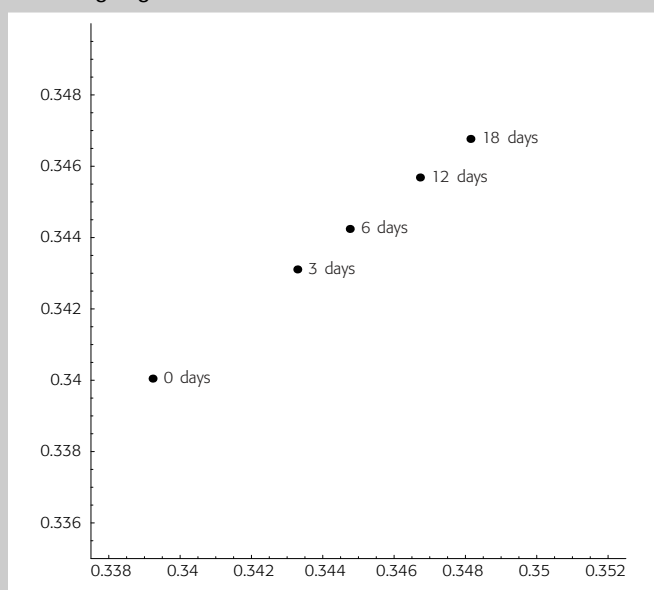
9 a Overview of the CIE-chromaticity diagram for the chemical pulp paper showing the chromaticity co-ordinates at different ageing times and different illuminations.



9 b Detail of the CIE-chromaticity diagram showing the chromaticity co-ordinates of the chemical pulp paper under UV illumination at 365 nm. A clear correlation with ageing is seen.



9 c Detail of the CIE-chromaticity diagram showing the chromaticity co-ordinates of the chemical pulp paper under UV illumination at 365 nm after correction for the transmission characteristics of the protection glasses used. A clear correlation with ageing is seen.



9 d Detail of the CIE-chromaticity diagram showing the chromaticity co-ordinates of the chemical pulp paper under D65 daylight illumination. A systematic trend towards the yellow is seen.

and, later on, yellow colour. This behaviour has been shown to be consistent with the formation of new fluorescent species within the paper re-emitting in the green-yellow region of the visible spectrum. No significant colour change in the fluorescence of Whatman No.1 filter paper could be shown from the calculation of the CIE chromaticity co-ordinates from the emission spectra.

The results of fluorescence spectroscopy show that the reported observations by paper conservators concerning the increase in intensity and the colour change in fluorescence of paper affected by degrading factors such as iron-gall inks, wet-dry interfaces, and foxing agents can be reproduced (at least to some extent) when additive-free paper is submitted to ageing conditions that promote hydrolytic and oxidative degradation of cellulose. The local fluorescence observed in connection to those degrading factors on paper may therefore arise from a locally more extensive degradation of cellulose promoted by them.

The hypothesis that fluorescence precedes brown discolouration in paper could not be verified under the experimental conditions used in the present study. The results showed that fluorescence and discolouration have developed simultaneously in the course of artificial ageing of the test papers, and that only minor changes in the spectral distribution of the fluorescence emission has occurred.

The perceived colour of fluorescence under UV-illumination was found to be affected by the presence of a visible component in the light generated by the lamp. It is believed that the spectral distribution of most ultra-violet lamps used will contain some blue light component. As a result, the colour impression of paper emitting fluorescent light under an ultra-violet lamp will depend on the specific illumination conditions. Perceived colour changes could be due to changes in the fluorescence emission spectrum but could also be due to changes in the reflectance of paper.

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