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An alternative to foxing?

Oxidative degradation as a cause of cellulosic discolouration

The role of environmental conditions, and particularly of relative humidity (RH) in the chemical deterioration of paper artefacts was examined in relation to library and archival storage in a humid climate. The investigation focussed on the chemical response of the paper artefact in a fluctuating moisture content, as a possible explanation for the causation of cellulosic staining, that redefines the evidence of foxing. The mechanisms of deterioration experienced in the humid climatic conditions of southeast Africa were investigated in the relationship between a fluctuating equilibrium moisture content of the paper substrate and the formation of cellulosic staining. The total water content of the atmosphere under fluctuating humid climatic conditions was replicated under experimental conditions, and the moisture content of the sample monitored in an attempt to plot the causation of cellulosic staining in the inconstant rate of diffusion from the paper surface. The chemical mechanisms of oxidative deterioration were quantified by the measurement of the formation of degradation products under controlled laboratory conditions. The experimental sample was subjected to a simulation of ambient conditions to monitor the formation of degradation products, determined by photographic, chemical and spectral analysis, compared with the empirical evidence of damage encountered under humid conditions. In the identification of the predominant oxidative mechanism of deterioration under humid climatic conditions, the investigation enables an assessment of risk that precedes and ultimately precludes costly conservation intervention.

Untersucht wurde die Rolle von Umweltbedingungen, im Besonderen die der relativen Feuchtigkeit (RH) bei der chemischen Zersetzung von Papier unter dem Aspekt der Lagerung in Bibliotheken und Archiven in feuchtem Klima. Die Untersuchung konzentrierte sich auf die chemische Antwort von Papierobjekte auf einen schwankenden Feuchtigkeitsgehalt als mögliche Ursache für Flecken auf der Zellulose, was eine Neubeurteilung von „Foxingflecken" zur Folge hätte. Die Beziehung der Zersetzungsmechanismen, die unter den feuchten klimatischen Bedingungen Südostafrikas auftreten, zu schwankenden Feuchtegehalten im Papiersubstrat und der Fleckenbildung auf Zellulose wurde untersucht. Der Gesamtwassergehalt der Atmosphäre unter schwankenden klimatischen Bedingungen wurde unter experimentellen Bedingungen nachgestellt und der Feuchtegehalt der Proben beobachtet, um die ungleichmäßige Diffusion zur Papieroberfläche als Ursache für Fleckenbildung nachzuweisen. Die chemischen Mechanismen des oxidativen Verfalls wurden durch Messung der Menge an Abbauprodukten unter Laborbedingungen quantifiziert. Die Proben wurden simulierten Umweltbedingungen unterworfen, um die Bildung von Abbauprodukten zu beobachten, deren Menge durch fotografische, chemische und spektroskopische Analysen bestimmt und mit den empirischen Daten von Schäden, die unter feuchten Bedingungen auftreten, verglichen wurden. Durch die Identifizierung des oxidativen Zersetzungsmechanismus als unter feuchten klimatischen Bedingungen vorherrschendem ermöglicht die vorliegende Untersuchung eine Einschätzung der Risiken, die kostspieligen Restaurierungsmaßnahmen vorausgehen, und kann sie schließlich vermeiden.

The preservation and conservation of library and archival materials is focussed on the life expectancy of paper artefacts. The study of the effects of climatic conditions on the complex mechanisms of deterioration has aimed to ensure the optimum environmental conditions for preservation, based on the chemical kinetics of the Arrhenius plot of accelerated ageing tests (Feller 1973; Thomson 1964).

The deterioration noted in a humid subtropical climate in southeast Africa is typified by areas of brown discolouration, loosely termed foxing and commonly associated with biodeterioration. With a growing awareness of common sites of discolouration, matched by the lack of evidence of universal damage by fungal contamination which might be anticipated in a warm humid climates, alternative causation was sought for the formation of areas of brown discolouration.

Mechanisms of deterioration

Three primary mechanisms of paper deterioration driven by excessive relative humidity (RH) were considered: biological deterioration, mechanical stress and chemical reactivity. Biological deterioration, immediately associated with humid conditions, has been thoroughly investigated and reported in the conservation literature — notably without consensus on the role of fungal infestation in the causation of foxing (Arai et al. 1990; Arai 1984; Beckwith et al. 1940; Press 1976; Szczepanowska 1986). More recent research on mechanical stress has called for a relaxation of environmental control, fuelling the debate on the efficacy of standard environmental recommendations (Mecklenburg, Tumosa 1995; Michalski 1996; Reilly 1996; Schultz, 1995). Ironically, a conference sponsored by the Conservation Analytical Laboratory of the Smithsonian Institute and the National Centre for Preservation Technology and Training in September 1997 was unable to achieve its goal of clarifying the issue of guidelines for collections environments (Tumosa et al. 1998). The recommendations of that conference called for the determination and quantification of the risks to library and archival collections by environmental settings and fluctuations. Encouraged by that challenge, an investigation was made of the role of chemical reactivity in paper deterioration prevalent under ambient humid conditions.

Chemical reactivity

The relative importance of chemical reactivity had not been

previously assessed in the context of environmental control, and an investigation was conducted into a possible chemical response of paper objects to the seasonal environmental fluctuations in humidity experienced in the sub-tropical climate, focussed on the role of oxidation at the wet/dry interface in the deterioration of paper.

The free radical mechanism, initiated in the decomposition of hydrogen peroxide formed at the wet/dry interface, and the complex chain reactions in which the presence of trace metals is significant, was considered. The study of the yellowing of paper conducted by the paper manufacturing industry was examined, in which the formation and consumption of measurable amounts of hydrogen peroxide were reported to be formed on the ageing of paper, and that the peroxide content increased during moist ageing to values higher than those noted in dry ageing (Kleinert, Marraccini 1963; Kleinert, Marraccini 1966; Marraccini, Kleinert 1962).

The role of autoxidation in the deterioration of library and archival collections was recognised in the formation of a brown line at the wet/dry interface, and the early investigations in the textile industry applied to this study in the capillary action of repeated wetting and drying as induced by fluctuating environmental conditions. The chemistry of the browning reaction, known as the Maillard reaction, was found to be linked to the moisture content in the dehydration of foodstuffs, and in the components of discoloured areas, linked also to the biochemical formation mechanism of foxing (Arai et al. 1988).

The theory of paper ageing, textile dyeing and the dehydration of foodstuffs has been applied to the conservation of paper in the discolouration noted in mounted works housed in storage boxes, and the tideline formation resulting from local treatment procedures. Peroxide formation was confirmed in the analytical investigation of the degradation products extracted from the brown line. The absorption of excessive water vapour in humid conditions, and the desorption kinetics of a seasonal decrease in RH was proposed to cause an oxidative reaction, equivalent to the formation of brown line at the wet/dry interface, as the predominant factor in the causation of deterioration, previously obscured by the accepted explanation of biological deterioration in a humid climate.

The lack of consensus on causative factors in the evidence of foxing was examined, and the role of both fungal infection and metal-induced catalysis found to be associated with hydrogen peroxide formation (Szczepanowska 1986; Williams et al. 1977). The role of the oxidative mechanism can be determined therefore as a precipitating factor in the causation of foxing.

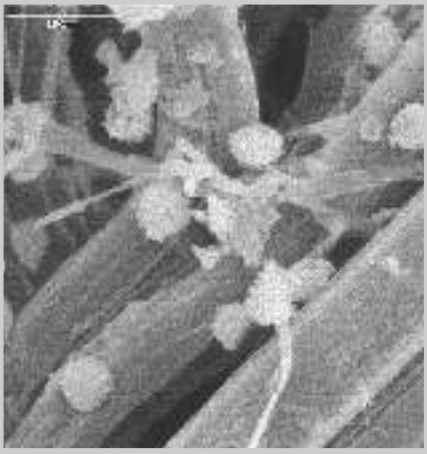
A further assessment of the chemical interaction of air, water and cellulose was included in the investigation of the crystalline/amorphous ratio of the cellulose structure. The modification of the fine structure of the cellulose in response to the fluctuating moisture content of the material was proposed as a measure of paper permanence, in the determination of a chemical mechanism of oxidative degradation in relationship between storage environment and paper substrate.

Manifestation of cellulosic discolouration

Initial investigation by stereo microscopy revealed that some forms of staining, in particular those with dark concentrated centres were clearly visible, while the diffuse blotches were scarcely discernible from the substrate. No evidence of fungal growth could be identified at 100x magnification. Scanning electron microscopy did reveal minimal evidence of microbiological infestation in the presence of only isolated hyphae at some sites. Proliferation of the Aspergillus niger fungal species identified was not evident (Berjack 1995 – Fig. 1). If microbiological degradation was the primary operative mechanism of paper deterioration, a more significant infestation could have been anticipated. Similar findings are reported by other authors (Hey et al. 1988). This, it has been argued, is related to the change in optimum conditions for sustained growth (Florian 1994).

The minimal evidence of microbiological infestation clearly bore no correlation to the widespread discolouration present. Cellulosic discolouration commonly encountered under humid conditions, in the form of diffuse areas of brown staining, gives a blotched appearance rather than the spotted effect associated with foxing (Fig. 2).

Such diffuse staining can be anticipated in the margins of books, predominantly but not exclusively adjacent to the exposed edges — the head, the tail and fore-edge of bound volumes. Tears and dog-eared corners were noted to be consi-



1 Scanning electron micrograph (SEM) of an area of cellulosic discolouration, revealing isolated hyphae.

2 Digital image of cellulosic staining at a resolution of 600 dpi.

stently associated with cellulosic discolouration, as were the frontispiece and illustration pages bearing graphic artistic techniques and the leaves adjacent to impervious coated paper used for photographic printing techniques.

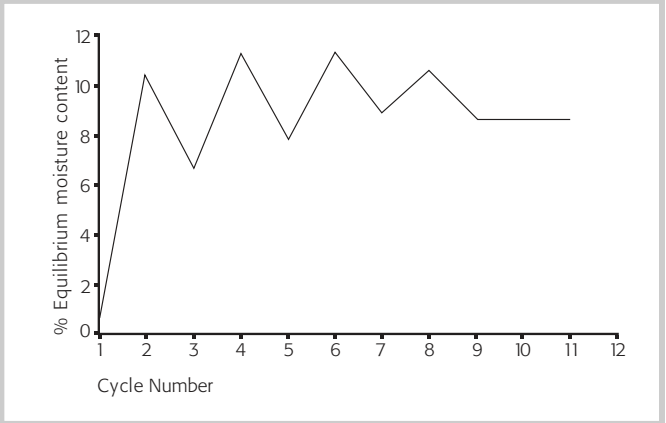
The discolouration is commonly evidenced in highly porous papers, comprising for example, the illustration pages of books which lack surface sizing for better graphic printing. The smooth, hard surface of the clay coated pages is consistently unaffected, in comparison to the heavy staining of adjacent soft-sized and rough surfaced leaves. Notable is the three dimensional penetration of staining through adjacent leaves, diminishing over a number of leaves distanced from the coated paper insertion. The role of microbiological infestation was further questioned in this transudatory appearance of staining.

A similar effect was noted in the discolouration accordant with brush strokes of laminate adhesive on artworks apparently laid down unevenly. Glued lamination is commonly practised in commercial picture framing to prevent inevitable cockling and surface planar distortion of works on paper displayed under humid conditions. Routine conservation delamination of artworks led to a noted correlation of discolouration with air bubbles in unlaminated areas, giving rise to further dissatisfaction with the traditional explanation of microbiological infestation, and prompting the need for further investigation of the browning phenomenon.

The ephemeral nature of much rare archival material frequently dictates the necessity for protective enclosure for loose leaves. Of particular concern is the cellulosic discolouration noted on documents stored in partially-filled boxes, while those stored in close-fitting enclosures seem unaffected. The implications for microclimate preservation strategies were pertinent to this investigation, but had been investigated elsewhere (Hofenk de Graaf 1994).

Chemical reactivity in a humid environment

The manifestation of damage, was believed to be of direct environmental causation. The increased chemical reactivity of cellulosic materials in a humid climate, generated in the relatively high equilibrium moisture content, is coupled with the seasonal fluctuation in the sharp drop in humidity in the short winter months.



3 Moisture regain.

Other environmental factors capable of initiating similar or concurrent oxidative reactions are acknowledged, but have been adequately treated elsewhere in the conservation literature (Feller 1973; Hon 1979; Lee et al. 1989; Lee 1989; Whitmore, Bogaard 1994). The psychrometric properties of atmospheric moisture reveal more clearly, the role of temperature and humidity in the causation of the oxidative mechanism under humid conditions. An excessive volume of water vapour present in a warm humid climate is adsorbed by the porous substrate, added to which is the periodic and localised capillary condensation of water following the excursions to dewpoint on a slight decrease in temperature. Condensed water in the paper is associated with the formation of cellulosic discolouration and is considered a predictor of chemical stability in storage (Vitale, Erhardt 1993: 507).

The transport of water vapour through the porous substrate in the increased rate of sorption and desorption by an increased water vapour permeability is associated with the formation of a brown line at the wet dry interface. The difference in the rate of desorption processes in surface evaporation and the diffusion of water vapour through the pores is explained in terms of the slow rate of air circulation in a humid environment. The disruption in transudational and lateral flow resulting from environmental fluctuations create points of moisture accumulation in the fibrous moisture reservoir of a bound volume. Such points of moisture accumulation have been ascribed to the formation of localised areas of cellulosic discolouration, and the spotted appearance has given rise to the generalisation of the phenomenon, known as foxing.

The absorbent capacity of cellulose in the paper substrate was found to be related to the effect of psychrometric properties of the atmosphere on the equilibrium moisture content of the substrate. The response in moisture regain varied on subsequent cycles of sorption and desorption, governed by the phenomenon of hysteresis, and measured in the change ratio of the crystalline/amorphous fraction. The limitation of chemical reactions in cellulose to the hydroxyl (OH) groups that are accessible to vapour diffusion provides a measure of the structural modification undergone in the formation of functional groups associated with oxidative degradation.

A clear understanding of this mechanism of autoxidation of cellulose is hampered by the complexity in the variety of basic repeating molecular units and the variety of forms in which cellulose occurs, which govern the degree of crystallinity and the moisture content of the material (Tryon, Wall 1961). Chemical reactivity is thus a function of the degree of crystallinity of the cellulose structure (Angibeau et al. 1985; Bertoniere, Zeronian 1987; Tasker et al. 1994).

The amorphous/crystalline ratio was monitored in the chemical modification of the cellulose, evidenced in the formation of functional groups, and measured against moisture regain, the diminishing sorption behaviour in response to repeated exposure to humid environmental conditions (Fig. 3).

Analytical investigation

An analytical procedure was developed to induce evidence of brown staining that might conform to the development of a wet/dry interface in response to humid environmental conditions. The investigation comprised two stages:

> the pre-conditioning of paper samples in simulation of ambient environmental conditions in climate controlled environment;

> a comparison of the formation of oxidative degradation products found in stained areas of naturally aged samples with those resulting from the induced staining procedure in stage 1.

The aim of the analytical investigation was to gather evidence in support of the relationship between the oxidative degradation mechanism and the unfavourable environmental conditions experienced in a humid climate.

Naturally aged samples with evidence of typical cellulosic staining were selected for comparison with samples prepared for artificial ageing and subsequent dynamic climate ageing in simulation of common physical features that might form sites of moisture accumulation and under fluctuating environmental conditions thus constituting a wet/dry interface. Such defects were considered to include the previous tideline formation resulting from water staining that might be accidental or as a result of localised conservation treatment. The application of paste and glue, representing the intentional introduction of moisture, was included as a potential source of damage as confirmation of the empirical evidence of uneven staining of artworks laid down unevenly to a backing board. Physical defects in the form of tears and dog-eared corners were introduced to the sample to represent areas that might hinder the transudational capillary flow. Such defects had formed the basis of the empirical evidence of an alternative causation of areas of staining in the bookstock.

Following the dynamic simulated climate ageing procedure, comparisons were made between both naturally and artificially aged sample groups to determine the formation of similar oxidative degradation products as a result of the climate ageing process. The most appropriate spectroscopic techniques for this task proved to by FTIR, using a DRIFT attachment. The formation of similar functional groups exhibited in the absorption bands in the region 1730-1720 cm^{-1} in both naturally and artificially aged paper samples was found in tentative evidence of a chemical mechanism of oxidative degradation resulting from exposure to fluctuating humid conditions. However, the acknowledged difficulty of measuring old paper successfully by FTIR spectroscopy, which detects only large changes of order, dictated the additional comparison of oxidative degradation by means of chemical reagents, as recommended by experienced researchers (Daniels 1995; Eusman 1995).

Supporting evidence was found in the corresponding fluorescence, which proved difficult to quantify. Conclusive evidence was found in the mutually concordant results of reagent staining using both methylene blue and the peroxide reagent, Naphtorin, indicating an oxidative reaction by free radicals resulting from hydrogen peroxide formation and decomposition under fluctuating environmental conditions (Fig. 4).

4 Concordant reagent staining by methylene blue (top) and Napthorin (below).

Conclusions

Evidence of cellulosic discolouration in the form of spotted blemishes at localised points of moisture accumulation, which could be directly related to foxing, was not successfully induced in this study, due possibly to a limitation of the experimental technique. It was not possible in the environmental control chamber to simulate the slow circulation of air in the ambient humid climate. On the basis of the preceding investigation however, the relationship between chemical and biological mechanisms of cellulosic discolouration cannot be discounted.

That foxing does not stem from mould growth alone, is supported by anecdotal evidence of the disruptive role of ventilation. Varied air circulation rates on the stability of surface layers of air surrounding organic artefacts act in restricting mould growth. Scott has reported on the validity of high ventilation rates in the control of available moisture in the paper substrate as supported by the lack of evidence of universal damage by fungal rot which might otherwise be anticipated in warm humid climates (Scott 1994).

The induced formation of cellulosic discolouration in the form of tidelines at the wet/dry interface and general evidence of moisture accumulation adjacent to a non-porous surface was noted. The significant inference made from these findings is that atmospheric oxidation is unequivocally related to cellulosic discolouration in a surface reaction, as suggested by earlier research (Marraccini, Kleinert 1962).

The nature of measured degradation was clearly oxidative. The degree of degradation visible as staining was qualitatively evaluated by reagent staining. The correlation of the evidence of the formation of oxidative degradation products with the evidence of cellulosic staining noted in library and

archival collections afforded the following conclusions:

- A chemical mechanism of paper degradation is evidenced in the oxidative reaction at the wet/dry interface, following the condensation and evaporation kinetics induced by cycling RH, and driven by temperature fluctuations in diurnal and seasonal ranges.
- The absorption of excessive water vapour is increased in a humid climate by an increased RH or by decreased temperature, where the dewpoint is reached. The vapour concentration rate at the surface, or at the atmospheric interfaces results in chemical deterioration by oxidation similar to that noted at the wet/dry interface, and accounts for the staining evident in paper that has no fungal basis, similar to the tidemark effect noted at the wet/dry interface.
- Sites of moisture accumulation which constitute a wet/dry interface are associated with evidence of cellulosic discolouration by the mechanism of the brown line formation as a surface phenomenon, as a vapour barrier to the transudational force of capillary action.
- Sites of moisture accumulation which constitute a wet/dry interface can be identified in physical defects , such as tears and dog-eared corners of books, at areas of uneven adhesive lamination, in local aqueous conservation treatments, and in impervious archival storage containers or adjacent to impervious surfaces.

The formation mechanisms of foxing has revealed a growing recognition of multiple factors in the causation of cellulosic staining, promoting an understanding of the nature and processes of degradation, which may operate simultaneously and probably sequentially. This investigation was directed rather, towards the role of oxidation at the wet/dry interface as found at the surface boundary layer as a unique phenomenon separate from and preceding the biological deterioration traditionally associated with paper deterioration in a humid climate. The phenomenon is thought to precipitate the causation of foxing. The non-proliferation of anticipated fungal growth can be assessed as a result of either inappropriate environmental conditions, or of chemical modification of the substrate, which in the limited response to atmospheric moisture, no longer sustained fungal activity.

The aim of this study was to investigate the causation of cellulosic discolouration in the increased chemical reactivity evidenced in the phenomenon of autoxidation at the wet/dry interface at the paper surface under humid conditions. The significance of an autoxidative phenomenon was demonstrated as a function of the unstable moisture content of materials similar to that of oxidation at the wet/dry interface, and previously obscured by the accepted explanation of biological deterioration. Analysis of the experimental work indicates an oxidative mechanism of degradation in response to the condensation and evaporation kinetics induced by cycling relative humidity driven by fluctuations in diurnal and seasonal ranges of temperature and humidity, which has profound relevance for recommended levels of in library and archival storage environments, beyond the boundaries of humid climatic regions.

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