The development of micro-analytical methodologies for the characterisation of the condition of paper

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Abstract

A research project has been initiated at the Netherlands Institute for Cultural Heritage (ICN) with the aim of developing micro-analytical methodologies for the characterisation of the condition of paper. The development of a comprehensive set of testing methods specifically dedicated to the investigation of paper-based objects having intrinsic value is a recognised need by the paper conservation community. Existing standardised testing methods for paper have been developed by the papermaking industry, and their application to original paper objects is almost invariably prohibitive due to the large amount of test specimens required. Paper properties considered of relevance to characterise its condition have been critically identified, with special emphasis on permanence. A number of chromatographic, spectroscopic, thermal, and microscopic techniques have been selected and will be evaluated concerning their applicability to assess the chosen properties using reduced amounts of sample. Such evaluation will be based on artificial ageing of representative standard reference papers, and on comparison of the obtained results with those of related standardised testing methods. It is believed that after refinement and validation the successfully tested methodologies will provide a basis for conservation policies devoted to safeguard our informational heritage.

Zusammenfassung

Am Niederländischen Institut für Denkmalpflege (ICN) wurde ein Forschungsprojekt mit dem Ziel gestartet, mikro-analytische Methoden zur Zustandsbeschreibung von Papier zu entwickeln. Die Entwicklung einer umfassenden Testreihe speziell für die Untersuchung von wertvollen Papierobjekten wird von der Gemeinde der Papierrestauratoren offensichtlich benötigt. Die bestehenden standardisierten Papiertestmethoden wurden von der Papierherstellungsindustrie entwickelt, aber ihre Anwendung an originalen Papierobjekten verbietet sich fast immer wegen der großen Menge an benötigtem Testmaterial. Die Eigenschaften des Papieres, welche als ausschlaggebend für die Zustandsbestimmung gelten, wurden, mit dem Schwerpunkt auf Alterungsbeständigkeit, genauestens bestimmt. Eine Anzahl von chromatographischen, spektroskopischen, thermischen und mikroskopischen Techniken wurde ausgewählt und werden bezüglich ihrer Anwendbarkeit, die gewählten Papiereigenschaften an kleineren Mengen Testmaterial zu bewerten, ausgewertet. Diese Bewertung beruht auf der künstlichen Alterung von repräsentativen standard Referenzpapieren und auf dem Vergleich der erhaltenen Ergebnisse mit denen der jeweiligen standardisierten Testmethoden. Es wird angenommen, daß nach Verbesserung und Anerkennung der erfolgreich geprüften Testmethoden eine Grundlage für eine Restaurierungspolitik, die sich dem Schutz unseres Kulturerbes widmet, geschaffen wird.

Introduction

The paper conservation community has long been aware of the lack of appropriate testing methodologies for the characterisation of the condition of paper artefacts having intrinsic value. Appropriate methodologies, in this context, should include the following features:

1. assess paper properties which are relevant to the evaluation of its condition, preferably allowing the detection of paper degradation at early stages;
2. allow micro-analytical or non-invasive investigations, being applicable to original paper artefacts;
3. repeatability and reproducibility.

The condition of paper is primarily defined by its chemical composition and structural features. Environmental interactions and internal reactions causing chemical decay and structural failure alter the condition of paper with time. Their detrimental effect on the condition of paper depends on the extent of environmental stress it has been exposed to and on its intrinsic ability to resist alteration. The latter is influenced by compositional and structural elements of paper, and it usually decreases as degradation proceeds. Acidity, traces of transition metal ions (e.g. Fe^{2+}, Fe^{3+}, Cu^{2+}), and lignin, for instance, have been recognised as destabilising internal factors that promote the degradation of paper [1-9]. Structural features such as the porosity of paper [10], fibre morphology, and the accessibility of cellulose [11,12] have also a significant influence on the deterioration of paper by determining the extent of interaction with external reactants.

End-use requirements for paper products that do not include preservation purposes are mostly related to their durability qualities, i.e. the ability of paper to perform well during intensive usage [13]. Characterisation of the condition of paper in such cases is focused on the assessment of physical properties that are pertinent to the intended use of the material. Contrarily, the condition of paper artefacts having intrinsic value and which are intended to be preserved for extended periods of time without extensive handling is better characterised by its chemical stability, or permanence [14]. The slow but continuous chemical changes undergone by cellulose and other paper constituents result, after a certain time, in loss of

macroscopic properties having a direct effect on the usability of paper as such.

Due to the intimate relationship between the chemical stability of paper and the retention of its mechanical properties, the latter have been extensively used as an indicator of the former [14]. Artificial ageing experiments designed to assess the permanence qualities of paper strongly rely on monitoring the retention of its mechanical properties in hostile environments [15]. The onset of mechanical failure, however, seems to happen only after considerable chemical decay. The physical strength of paper is well retained until the degree of polymerisation (DP) of cellulose decreases to low critical levels [1]. Early stages of degradation or menace, therefore, cannot be evaluated by means of existing testing methods assessing mechanical properties. Additionally, the amount of test specimen required for the evaluation of mechanical properties of paper according to existing standardised methods is certainly prohibitive when it comes to investigating original objects of intrinsic value (See Table 1). Mechanical properties also do not provide information on the nature of the operating degradation mechanisms, which is of interest for the design of chemical preservation treatments.

Table 1. Amount of test specimen required by existing standardised methods to assess mechanical properties of paper

PAPER PROPERTY	TEST METHOD	SAMPLE REQUIREMENT[1]
Folding Endurance (MIT Tester)	TAPPI T511 om-88	10 (15x150mm) test strips per direction of paper
	ASTM D2176-89	10 (15x150mm) test strips per direction of paper
Folding Endurance (Schopper Type Tester)	TAPPI T423 om-89	10 (15x100mm) test strips per direction of paper
Tensile Strength	TAPPI T494 om-88	10 (25x >180mm) test strips per direction of paper
	ASTM D828-87	10 (15x250mm) test strips per direction of paper
	DIN 53 112	10 (15x250mm) test strips per direction of paper
Zero-Span Tensile Strength	TAPPI T231[2]	6x >15mm test strips[3]
Bursting Strength	TAPPI T403 om-91	10 (62x62mm) test specimens per side of paper
	ASTM D774-87	10 (64x64mm) test specimens per side of paper
	DIN 53 113	5 (100 cm_) test specimens per side of paper
Internal Tearing Resistance[4]	TAPPI T414 om-88	10 (53x63mm) test specimes per direction of paper
	ISO 1974:1990(E)	10 (50x63mm) test specimes per direction of paper
	DIN 53 128	10 (50x65mm) test specimes per direction of paper

1 Minimum amount of test specimen required
2 Standard method for testing pulp test sheets
3 Minimum amount of test strips not specified
4 Each test specimen consists of a set of several pieces of paper piled together

Sources:

TAPPI Test Methods 1992-1993, TAPPI Press, Atlanta (1992).

1991 Annual Book of ASTM Standards, Vol. 15.09 - Paper; Packaging; Flexible Barrier

Materials, Business Copy Products, ASTM, Philadelphia (1991).

DIN-Taschenbuch 118, Papier, Pappe und Zellstoff 1, Beuth Verlag GmbH, Berlin – Köln (1991).

DIN-Taschenbuch 213, Papier, Pappe und Zellstoff 2, Beuth Verlag GmbH, Berlin – Köln (1986).

ISO 1974 International Standard (Third edition 1990-02-01).

Characterisation of the chemical environment of cellulose within the paper provides information on the degree of risk associated to known degradation reactions such as acid-catalysed hydrolysis, alkaline degradation, and iron(II)-catalysed oxidation [1,16-18]. The reactivity of cellulose towards oxygen in different chemical environments, for instance, can be evaluated to estimate the ability of paper to resist auto-oxidative degradation. The possibility of detecting earlier stages of alteration and of gaining information on the ongoing degradation mechanisms by identifying and quantifying reaction products of cellulose and other paper components is also a significant advantage of assessing chemical properties of paper.

Up-to-date micro-analytical methods offer an attractive range of possibilities to assess various chemical properties of paper. The intention of this work is to evaluate a selected number of those techniques with respect to their usefulness to characterise the condition of paper using reduced amounts of sample, with emphasis on permanence. It is believed that this will lead to the elaboration of a set of complementary testing methodologies for the investigation of paper artefacts having intrinsic value.

Discussion

The working structure devised for the development of micro-analytical methodologies to characterise the condition of paper is depicted below (See Scheme 1).

Pre-selection stage

The initial step to be considered in order to characterise the condition of paper for preservation purposes consists in identifying paper properties that are fundamentally related to its permanence qualities. Because the two major general degradation mechanisms of paper under ordinary storage conditions are the acid-catalysed hydrolysis and the auto-oxidation of cellulose, paper properties providing information on its intrinsic stability towards those degradation modes or on the extent to which such degradation reactions have already occurred are of relevance.

Hydrolytic scission of cellulose glycosidic bonds is strongly promoted by the presence of acids within the paper, and its immediate result is a reduction in the degree of polymerisation of cellulose. Acidity and the average DP of cellulose are therefore relevant parameters directly related to the degradation of paper.

Characterisation of the chemical environment of cellulose with respect to acidity is usually carried out by measuring the hydrogen-ion activity in aqueous extracts of paper, i.e. the pH of those extracts. The concept of 'pH of paper' – referring to the pH of aqueous extracts of paper - has been widely used as perhaps the most important indicator of permanence. Standardised methods to measure the pH of aqueous extracts of paper under controlled conditions are available. Quantification of the acidity of aqueous extracts of paper by titrimetric determination of acid milliequivalents is also possible according to standard procedures. A limitation of those test methods to the study of objects having intrinsic value is again the relatively high amount of test specimens required (e.g. 2 x 1.0g of paper for pH measurements and 2 x 5.0g of paper for the determination of acidity according to TAPPI test methods [19-21].

An alternative standardised test measures the surface pH of paper using a flat combination electrode immersed in a drop of water applied onto the surface of paper [22]. This xrocedure does not require the cutting of the paper or extensive soaking, and it is therefore referred to as non-destructive. The development of brown discoloration where wet-dry interfaces occur within the paper due to water-cellulose interactions, however, is a well-known degradation type observed for locally wetted cellulosic materials [23-25]. The brown lines formed in those areas have been shown to become insoluble with time, permanently altering the appearance of paper.

The above mentioned methods rely on a single extraction procedure whereby acidic constituents of paper are only partially extracted. The total acidity of paper, therefore, cannot be estimated. Additionally, the intrinsic acidity of paper can be wrongly estimated when water is used to extract it. Such a deviation arises from the fact that the measured acidity in aqueous extracts results from a combination of the concentration of hydrogen ions originally present within the paper plus those generated from paper components undergoing acid reaction with water such as aluminium sulphate [26].

The development of suitable methodologies to evaluate the acidity of paper artefacts on a microscale has been discussed by Wouters [27]. This includes miniaturisation of existing pH measuring procedures for aqueous extracts of paper under rigorously controlled conditions, evaluation of non-instrumental methods based on micro-sensors, multiple extractions, meas-urement on non-aqueous solvent extracts, and discrimination of pH-establishing components present in the extracts of paper. The latter will provide information on the nature of the dissolved compounds in the extract contributing to acidity - mineral acids, organic acids, and other chemical species such as inorganic salts. The discrimination of individual components of aqueous and organic extracts of paper can also provide useful information on the ongoing degradation mechanisms, and on the extent of degradation undergone by different paper components – cellulose, hemicelluloses, and lignin.

The degree of polymerisation of cellulose is a very important property for a quantitative evaluation of cellulose degradation. A number of methods are available for the determination of average degrees of polymerisation of cellulose [28]. Viscosity measurements are the most commonly used technique for practical applications. It relies on the relationship between DP and the viscosity of cellulose solutions. Especial solvent systems for cellulose or derivatisation of the polymer followed by dissolution in a suitable organic solvent are required to assess the average DP of cellulose. Existing standardised methods are based on the measurement of the efflux time of dilute cellulose solutions in a capillary viscometer or on the falling ball method. The amount of test specimen required ranges from 250mg to 1875mg of moisture-free pulp according to TAPPI standard test methods [29,30].
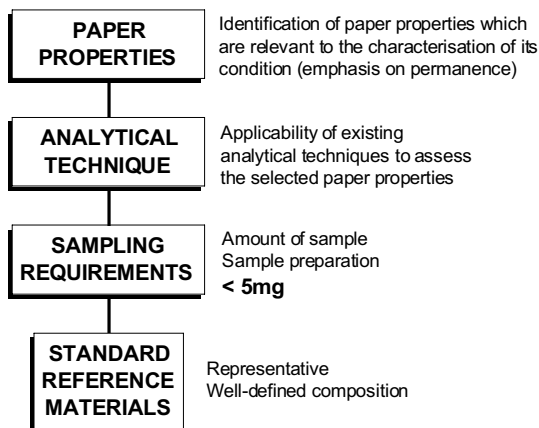
Size-exclusion Chromatography (SEC) is a more recent technique that can be applied to evaluate the DP of cellulose. Because it is based on the transport velocity of cellulose chains having different lengths through a porous medium, SEC permits the determination of the molecular weight distribution of cellulose. Molecular chains of different lengths constituting the polydisperse cellulose polymer are separated, and the analysis provides a distribution curve showing the relative concentration of the different molecular fractions as a function of their degree of polymerisation. This is a relevant information to the understanding of the progress of degradation in terms of chain scission. Methodologies using both derivatised and non-derivatised cellulose are available [31-33], which require sample amounts in the range of 10-50mg and may be further miniaturised with the introduction of micro-columns. Packed-Capillary columns, for instance, allow for a reduction in the amount of test specimen by a factor or 100 to 1000 in comparison to conventional packed columns.

Unlike the straightforward degradation mechanism of acid-catalysed hydrolysis, the oxidation of cellulose due to its interaction with atmospheric oxygen is a much more complex chemical process including several simultaneous and consecutive reaction steps. Generation of carbonyl and carboxyl functional groups across the molecular chains, ring opening, cross-linking, and chain scission yielding different oxidised fragments result from the auto-oxidation of cellulose.
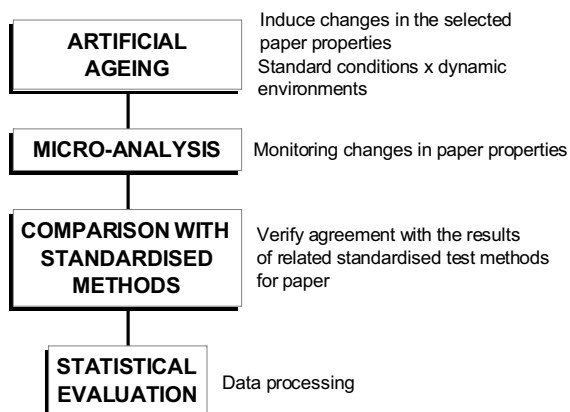
A significant factor influencing the oxidative degradation of cellulose is the catalytic action of transition metal ions such as iron, copper, and manganese. The content of such species within the paper is therefore an important property related to its oxidative stability. Recent standardised methods for the quantitative determination of iron, copper, and manganese in paper make use of atomic absorption spectroscopy (AAS). This extremely sensitive technique is based

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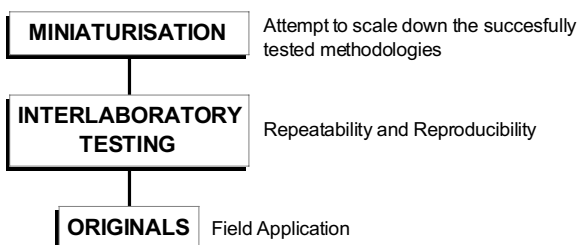
1. Pre-Selection Stage

PAPER PROPERTIES — Identification of paper properties which are relevant to the characterisation of its condition (emphasis on permanence)

ANALYTICAL TECHNIQUE — Applicability of existing analytical techniques to assess the selected paper properties

SAMPLING REQUIREMENTS — Amount of sample. Sample preparation **< 5mg**

STANDARD REFERENCE MATERIALS — Representative. Well-defined composition

2. Experimental Tests

ARTIFICIAL AGEING — Induce changes in the selected paper properties. Standard conditions x dynamic environments

MICRO-ANALYSIS — Monitoring changes in paper properties

COMPARISON WITH STANDARDISED METHODS — Verify agreement with the results of related standardised test methods for paper

STATISTICAL EVALUATION — Data processing

3. Refinement and Validation

MINIATURISATION — Attempt to scale down the succesfully tested methodologies

INTERLABORATORY TESTING — Repeatability and Reproducibility

ORIGINALS — Field Application

Scheme 1. Working structure for the development of micro-analytical ethodologies to characterise the condition of paper.

on the phenomenon of selective absorption of ultraviolet or visible radiation by free atoms in a hot gas sample. The amount of excitation radiation absorbed by each element is proportional to its concentration in the measurement solution, and the detection limits for many elements are better than 1 ppb. Although the amount of test specimen required according to the standard procedures for paper is relatively high (e.g. 10.0g according to TAPPI T266 om-88 and 30.0g according to DIN 54 363), micro-sampling can be considered without analytical restrictions. Utilisation of a graphite furnace to atomise the sample, for instance, requires the injection of only 20-50mL of dilute measurement solutions, and biological samples can be directly introduced in the furnace tube where they are ashed or chemically pre-treated as part of the analytical procedure [34]. Paper samples of 0.05-0.8 mg have already been successfully analysed for iron and copper without any preparation by means of this technique [35].

The stability of paper to resist auto-oxidation can be evaluated by assessing the reactivity of cellulose in different chemical environments under high oxygen pressures and at elevated temperatures. Oxygen uptake, chemiluminescence, and thermal analysis may be used to characterise the resistance of paper to undergo oxidative degradation. Those techniques have been used to study the oxidative stability of organic materials in general, and the analysis is usually based on determining the time before auto-oxidation reaches its autocatalytic stage – the oxidation induction time (OIT). The auto-oxidation of organic materials is characterised by an initial stage in which the reaction is very slow (induction time) followed by an autocatalytic stage where the reaction rate increases exponentially. Materials having high oxidative stability exhibit longer induction times, whereas a lower stability is indicated by shorter OIT's.

Oxygen consumption by cellulose measured as a function of time provides information on the reaction rate of oxidation. Measurements using 1g paper samples have been successfully carried out by Hernadi in relatively short times within the temperature range of 130°C-190°C [12]. Application of this methodology on a micro-scale should therefore be considered to the investigation of original paper objects.

Chemiluminescence analysis (CL) is a very sensitive technique that allows the investigation of oxidative reactions at very early stages, long before degradation can be assessed by any physical property [36]. It is based on the emission of low levels of light by organic materials undergoing oxidation, which can be related to the rate of reaction. The chemiluminescence of cellulose has been investigated using paper surfaces of 1cm^2 [37], and it has been verified that samples as small as 0.25cm^2 can be satisfactorily analysed[1]. The development of a CL-imaging technique allowing the visualisation of light emission by means of a slow scan CCD camera offers the advantage of simultaneous multiple analysis using very small samples [38]. This is being further developed and will lead to the introduction of a CL-microscopy technique. Additionally, the possibility of investigating the progress of oxidation in real time under heterogeneous conditions is a welcome feature to help understanding the degradation of paper.

Oxidative reactions accelerate the rate of degradation of cellulose during pyrolysis in air at temperatures up to 300°C [39]. Because oxygen still plays a major role in enhancing the degradation of cellulose at such high temperatures, application of thermal analysis to study the oxidative stability of paper materials seems justifi-

able. Differential Scanning Calorimetry (DSC) measures the heat flow from or to the sample as it undergoes exothermic or endothermic processes, respectively. Organic materials being thermally degraded under oxygen or air undergo oxidative breakdown. Because oxidation is a highly exothermic reaction, this is clearly evidenced in a DSC thermogram. DSC has found wide application in the industry as a fast and simple means of assessing the oxidative stability of synthetic polymers, and of screening antioxidants to improve the long-term performance of polymers. A standardised test method for the determination of OIT of polyolefins makes use of DSC analysis (ASTM D3895). Paper samples of 1-5 mg can be analysed by DSC under static air allowing the determination of thermo-oxidative induction times [40].

Identification and quantification of products yielded by hydrolytic and oxidative degradation of paper components evidence the extent to which degradation has occurred. It also provides a means of detecting paper degradation at earlier stages.

Cello-oligosaccharides, cellobiose and monosaccharides have been identified in aqueous extracts of degraded paper, evidencing the depolymerisation of cellulose [25,41]. Degradation products including uronic acids [25], hydroxymethylfurfural [4], L-malic acid [41], oxalic acid [42], and acetic and formic acid [43] have also been identified in extracts of paper, showing that oxidative reactions of cellulose and hemicelluloses have taken place. The presence of water-soluble lignin degradation products in paper extracts confirms the contribution of lignin to the development of acidity within the paper, and their relative concentrations may be considered an indicator of the extent of oxidative degradation of paper (See Table 2).

LIGNIN DEGRADATION COMPOUNDS	CONCENTRATION [g.L^{-1}]
Vanillic Acid	800
Vanillin	90
Ferulic Acid	40

Table 2. Some lignin degradation products identified in aqueous extracts of paper [43].

Volatile compounds are also yielded by degradation reactions of paper. Analysis of the headspace of naturally aged books has shown, for instance, the presence of more than 140 constituents [44]. The analysis of headspace samples offers the advantage of being non-invasive to the paper, which is a desirable feature concerning the investigation of original objects.

Chromatographic techniques have been used to separate the individual components of paper extracts or headspace samples. High-performance Liquid Chromatography (HPLC) allows the separation of complex mixtures in liquid phase and a highly accurate and precise quantitative analysis [45]. A liquid sample containing the compounds to be analysed is injected in the chromatographic column, through which a mobile phase consisted of a suitable solvent or solvent mixture is continuously pumped. The individual components are separated as the mixture is transported through the column. As the separated analytes leave the column they are monitored by a detector, whose response is proportional to their concentration. The possibility of using small injection volumes (mL range) and the high sensitivity of available detectors make

HPLC a suitable method for the analysis of extracts of minute paper samples.

Gas chromatography (GC) is based on the same principle used in HPLC with the major difference that a gas is used as the mobile phase to transport volatile analytes or their derivatives through an appropriate column. Active samplers that pump headspace samples into trapping systems can be used to collect volatile degradation products of paper. A more practical alternative is the use of passive samplers onto which those compounds are adsorbed in relatively short time periods. Solid Phase Microextraction (SPME) [46,47] seems to be a suitable method to sample small headspace volumes inside books, archival boxes, etc. It uses specially coated fibres that can extract volatile compounds from air samples by exposure for a few minutes. The analytes remain on the fibre until they are thermally desorbed inside the gas chromatograph, and concentrations in the ppb range are well detectable. The fibres are reported to have a useful life of 50-100 extraction-desorption cycles, and the analytes can be stored on the fibre itself for later analysis.

Although separation and quantification of paper degradation products is readily accomplished by the above-mentioned chromatographic techniques, identification of the chemical structure of unknown analytes requires the use of special detection systems. Mass spectroscopy (MS) is the most common method used in combination with GC or LC that gives information on the chemical structure of the analysed compounds [48]. MS is based on the separation of gas-phase ions generated from the fragmentation of the analytes according to their ratio of mass to charge. Analysis of the mass fragmentation pattern of the analytes provides information on their chemical structure.

Table 3 summarises the paper properties identified as being of relevance to characterise the condition of paper for preservation purposes, and the analytical techniques that may be used to assess those properties using reduced amounts of sample. Concerning sampling requirements an initial maximum amount of 5mg of test specimen has been arbitrarily chosen as a pre-selection criterion to include a given analytical technique in the test programme. Additional aspects of relevance to be considered are the heterogeneity of paper and therefore the representational factor of the sample, and the sampling technique itself when destructive analysis is required. The latter could include, for instance, removal of surface or edge fibres, punching minute holes through the paper, or cutting loose edges. In order to provide consistent results, the development of an adequate micro-sampling tool as part of the analytical methodologies to characterise the condition of paper is an important goal to be achieved.

Table 3. Paper properties and analytical techniques selected to assess the condition of paper on a micro-scale

PAPER PROPERTY	ANALYTICAL TECHNIQUE
Acidity	HPLC, AAS, pH-measuring methods
DP of Cellulose	SEC
Transition Metals Content (Fe, Cu, Mn)	AAS
Oxidative Stability	DSC, CL, Oxygen Uptake
Degradation Products	HPLC, GC-MS

The production of standard reference papers having specified, well known compositions is an essential aspect of this study. Assessment of the applicability of the selected analytical techniques to characterise the condition of paper will be based on measuring changes undergone by reference samples promoted by artificial ageing, and the application of the developed methodologies to original objects will strongly rely on the comparison between the obtained results and those acquired with the standard reference papers. A set of reference papers that are representative of existing types of paper must therefore be carefully defined and manufactured in order to ensure the comprehensiveness of the testing methodologies. Examples of representative reference papers include:

• Gelatine-sized cotton paper with or without fillers (e.g. carbonates, sulphates);
• Groundwood, lignin-containing papers with alum-rosin size;
• Bleached chemical wood pulp papers with alkaline sizing/ filling.

Samples of the same standard reference paper having varying concentrations of transition metal ions and acidified to different extents must also be prepared in order to assess the effect of those parameters on the degradation of paper.

Experimental tests

Except for the transition metals content, changes in the selected paper properties will be induced by artificial ageing the standard reference papers. The aim of artificial ageing experiments is to increase the rate of degradation observed during the natural ageing of paper by submitting it to hostile conditions. It is desired that the rates of individual degradation reactions are increased by the same factor, and that no other reaction is introduced in the system by the artificial ageing conditions [49]. According to Erhard, the extent to which artificial ageing of paper corresponds to natural ageing is not yet clear, and there are no generally accepted standard ageing conditions. Existing standardised methods make use of static 'moist-hot' or 'dry-hot' environments to simulate natural ageing [15,50]. It has been verified, however, that dynamic conditions such as cycling relative humidity (RH) can promote

more drastic changes in paper properties [51]. The generation of free radicals within the paper due to mechanical stresses resulting from changing humidities is a possible reason to such behaviour [52]. If daily or seasonal humidity cycles do influence the natural degradation of paper to the same extent, the use of dynamic ageing conditions has to be considered. Because the hydrolytic degradation of cellulose is very sensitive to moisture [53], and because the ageing process of cellulose has been shown to be RH dependent, artificial ageing will be performed at an elevated temperature (90°C) and under a RH value representative of ordinary storage conditions for paper (50% RH).

The selected analytical methods will be used to monitor the changes undergone by the paper samples at regular time periods during artificial ageing. The degradation of paper will be additionally monitored by existing standardised methods providing information on the same properties or other properties related to the permanence of paper. Acidity, DP of cellulose, and transition metal content, for instance, will be determined according to standardised test methods. The copper number of paper, which measures its reducing power, will be used to follow hydrolytic and oxidative changes [54]. A number of mechanical properties such as the zero span tensile strength [55] and the folding endurance of paper [56] as well as brightness [57] will also be assessed. By comparing the results of the micro-analytical methods with those obtained by means of the standardised testing methods it will be possible to verify relations between the selected properties and the overall condition of every standard reference paper.

Refinement and validation

Refinement of the successfully tested methodologies will include further miniaturisation and evaluation of the possibility of using one test specimen for consecutive analysis. Interlaboratory testing will be performed in order to ensure repeatability and reproducibility. Finally, a number of original paper artefacts will be characterised by the developed micro-analytical methodologies as a field test. Characterisation of the condition of a given paper will be possible by applying the methodologies to assess the above mentioned properties. The results can be checked against those obtained for a similar standard reference paper, whose degradation history is known, and a conclusion over the condition of the tested paper can be drawn based on comparison.

Conclusion

The possibility of investigating the condition of original documents, prints and drawings will provide a basis for decision-making: 1) items of intrinsic value identified as being endangered or of poor condition will have high priority for conservation treatments; 2) important information stored on items identified as being of poor condition will have high priority for reformatting (microfilming, etc.). Considering the hundreds of million of paper artefacts stored in libraries, archives, and museums, it is fundamental to have such priorities in order to preserve our paper-based heritage.

Assessment of the efficiency of conservation treatments applied to original paper objects to slow down or arrest degradation is also a significant benefit offered by micro-analytical methodologies.

Additionally, the ability to evaluate the chemical stability of paper will allow a better understanding of the effects of composition on permanence, and a more basic knowledge about the mechanisms of paper degradation will be gained.

Notes

1. Measurement carried out during the demonstration of CL-equipment at the International Workshop on Practical Applications of Chemiluminescence at the Oxidation of Chemical Systems, Slovak Academy of Sciences, Bratislava, 1998.

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Biography

José Luiz Pedersoli Jr. is a chemist with a M.Sc. degree in Polymer Chemistry from the University of Helsinki, Finland (1994). His experience as a paper chemist started at the Finnish Pulp and Paper Research Institute (KCL), where he has worked for a project on the light-induced yellowing of high yield pulps.

In 1993 he was employed by the Finnish National Library to evaluate a simultaneous deacidification-reinforcement conservation treatment for unbound paper items. This research work was carried out at KCL, and it involved a number of analytical techniques for the characterisation of paper, artificial ageing experiments, and standard testing methods. The results obtained were part of his M.Sc. thesis.

In Brazil, Mr. Pedersoli has been involved in several activities in the field of paper conservation. He has taught paper chemistry courses for conservators, and has maintained permanent co-operation with major institutions in Brazil dealing with the preservation of cultural heritage such as the Conservation and Restoration Centre for Movable Heritage – CECOR. He has recently worked as a translator/lecturer for a project devoted to the dissemination of significant literature in the field of preventive conservation in libraries and archives. This project was co-ordinated by the Brazilian National Archives and supported by the Commission on Preservation and Access (USA).

Mr. Pedersoli has also worked as course/laboratory assistant for the 'Scientific Principles of Conservation' courses – ICCROM – held in Brazil (SPC95) and in Rome (SPC96).

Mr. Pedersoli is currently working as a Conservation Researcher at the Netherlands Institute for Cultural Heritage in the field of paper. His activities involve the development of micro-analytical methods to characterise the condition of paper objects of intrinsic value, and investigations into the mechanisms of paper degradation.

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