Methodology and Criteria for the Evaluation of Paper Conservation Interventions. Literature Review.

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Introduction

The evaluation of various paper conservation interventions has been the subject of many publications. It is remarkable though that very few studies have been published focusing on the methodology of the evaluation itself. In this article, we review the relevant literature in order to establish the principles, theoretical and practical, of the methodology and the criteria used for the evaluation of paper conservation interventions.

The basic underlying principle of every such study is the notion that a conservation treatment should slow down the deterioration process. On that ground, the comparison of the deterioration rates of the most important mechanical, chemical and optical properties of paper before and after the treatment would demonstrate the suitability of the treatment under study. There are certainly other criteria of minor importance, but they will be presented and discussed later.

The most important references concerning organized attempts for the establishment of a methodology for the evaluation of paper conservation interventions come from the industry. The FMC Corporation suggested the following methods for the evaluation of mass deacidification systems[1, 2]:

- Evaluation based on chemical properties: pH, alkalinity, brightness, yellowness, copper number
- Evaluation based on physical properties: measurement of tensile properties, folding endurance and tearing resistance. A numerical criterion of effectiveness is also introduced, based on the improvement of folding endurance and the effect of accelerated ageing.

From the conservation field, Bansa[3, 4] suggests that any attempt to evaluate the effectiveness of a treatment should be limited to the comparison of few of the most important paper properties of untreated and treated samples after accelerated ageing, the temperature and relative humidity of ageing being unimportant. On the basis of convenience, he recommends a dry oven operating at 105°C. He considers that from the mechanical properties of paper, tensile post fold is the most suitable for use in an evaluation procedure.

The evaluation of a paper conservation intervention can be seen as a comparative study of permanence evaluation, where the permanence of the treated and the untreated paper are compared. From this point of view, the discussion of the methodology of paper permanence studies is relevant to the present review. Browning[5] presents the methodology and the chemical and physical tests that can be used for the estimation of paper permanence.

The existing methods for the evaluation of paper properties originate from the paper industry, where they are used for quality control, research for new products and improvement of quality, yield and performance. With very few exceptions, no methods have been developed specifically for the evaluation of the suitability of paper conservation treatments. This happens for a number of reasons:

- Paper conservation is a relatively new scientific field involving a limited number of scientists. Until recently, paper conservation was a craft based on secret recipes, practiced by highly skilled but empirically educated individuals. Dedicated scientific journals appeared at early seventies. On the contrary, paper industry has a long history and the industrial associations have standardized the quality control processes. Scientific journals date from the beginning of the 20th century.
- Industrial paper production is subject to the market laws and competition. Industry must fund research and quality control in order to maintain high quality standards

and develop new competitive products. Contrariwise, paper conservation is practiced by individuals or public organizations, with limited economics.

A critical question concerns the paper properties that should be included in an evaluation study and the most suitable methods for their measurement. The properties should help create a complete picture of the sample condition and the methods must be sensitive to the changes induced by the treatment. On the other hand, economy of time and resources are equally important and the number of tests should be kept to a minimum. Wilson and Parks[6] recommended the following tests for detecting changes during the ageing of paper, but they think that they are not particularly useful for identifying the nature of the change: folding endurance, tearing strength, elongation, tensile energy absorption, alkali solubility, copper number and viscosity. They also suggested the following tests for determining "what happens" during the ageing of paper: zero span tensile strength (fibre strength), wet tensile (crosslinking), pH (generation of acid), alkali solubility (some chain scission and alkali sensitivity), functional group content (oxidation), molecular chain length distribution (chain scission and randomness of chain scission) and peroxide formation and oxygen sorption (oxidation). They think that tensile and bursting strength are "somewhat useful". Most of these methods appear in paper conservation evaluation and permanence studies and will be reviewed in the following pages.

In order to meet the targets of this review, the following topics are relevant and will be discussed in this article:

- Accelerated ageing: theoretical principles, most common methods, standards and conditions (temperature and relative humidity).
- Experimental setup: sample selection and preparation, planning of the experiments.
- Methods for the evaluation of paper properties: established methods already in use, various methods that have been sparingly used and methods that have never been used but have the potential to evolve and apply to specific problems of the evaluation.
- Criteria of effectiveness of the intervention.

Table 1 (see appendix) presents a selection of the most important relevant publications of the last 30 years.

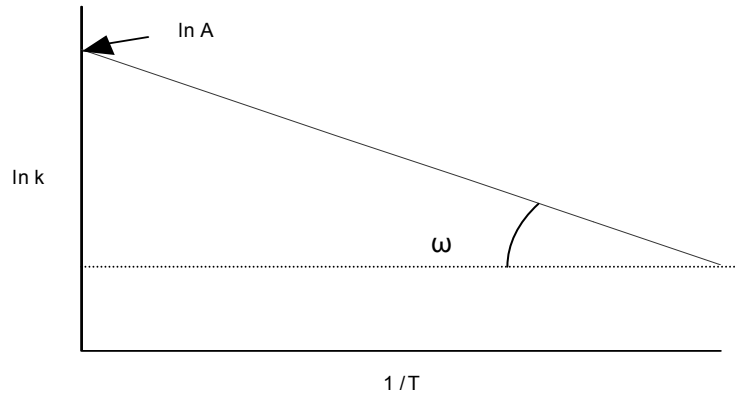
Accelerated Ageing

Theoretical Principles

It has been mentioned in the introduction that the comparison of the deterioration rates of the most important paper properties before and after a conservation treatment demonstrates its effectiveness. The deterioration rates should be estimated at normal environmental conditions, specifically at the conditions that paper is stored in libraries and archives: around 50% RH and 17-21°C. This task presents an insurmountable difficulty: paper deteriorates very slowly under these conditions; some kinds of paper might need many years to present statistically significant changes in some properties when they age naturally. A practically applicable method for the acceleration of the ageing process and logistics for the calculation of the acceleration rate should be introduced, so that experimental results would be produced in a reasonable time span. The scientific principle for such a method lies within the Arrhenius equation, which permits the estimation of the reaction rate at any temperature.

$$k = A \cdot e^{-\frac{E}{RT}} \text{ or } \ln k = -\frac{E}{R} \cdot \frac{1}{T} + \ln A \qquad (1)$$

where k: rate constant, A: frequency factor, E: activation energy ($Kjmol^{-1}$) and T: absolute temperature, T ° (in Kelvin) = t ° (in Celsius) + 273.



There are two distinct stages in a kinetic study implementing the Arrhenius equation[7, 8]. In the first stage the order of the reaction is estimated. In the case of paper, a first order reaction is assumed, which is very often approximated by a zero order at the very beginning of the reaction[9-16]. In the second stage, the rate constant k of the reaction is determined at a number of different temperatures. The rate constant at a given temperature can be found by plotting the concentration of a reactant or a product of the reaction versus the reaction time. The rate constant equals the slope of the plot. By plotting lnk versus the inverse absolute temperature 1/T (Arrhenius plot), E and A can be determined and from them the rate constant k at ambient temperature, say 20ºC can be calculated. This approach, in the case of paper ageing, presents two difficulties:

- Natural paper ageing is the sum of numerous different consecutive or parallel reactions, which are hydrolytic, oxidative or photochemical homolytic or heterolytic in nature. The relative contribution of any of them could be temperature dependent and the order of the reaction could be different at different temperatures. Therefore, the activation energy and frequency factor calculated by the above-mentioned approach must be the apparent values. Zou et al.[15] demonstrated theoretically that this approach is valid in the case of paper ageing

and that the apparent values (which are the weighted means of the values of the partial reactions) thus calculated can be used for permanence predictions.

- There is no way that the concentrations of the reactants or products of paper ageing can be measured. Instead, the following two approaches have been used.
  - The older one involves the measurement of a mechanical or optical property of paper[17, 18, 7, 19-22, 11]. The rationale behind this approach is that chemical changes due to ageing must affect the other properties of paper. This approach is empirical and has been the object of controversy[23], since there is no established relationship between chemical and physical changes during ageing. The derived rate laws are based on arbitrary mathematical conversions that linearize the experimental data[8]. The most prominent paper property used is the folding endurance. It has been found that in most of the cases the logarithm of the number of folds (FE) depends linearly on the time of ageing:

    $$FE = FE_o - k\,t \qquad (2)$$

  - The second approach is based on the measurement of the extent of the glycosidic bond breakage as ageing advances[24, 12, 15, 25]. It can be proved that[26], regardless of the reaction order and the kinetics of cellulose degradation, equation 2 gives the percentage δ% of the glycosidic bonds that break in time t:

    $$\%\ \text{broken bonds} = \delta\% = 100 \cdot \left(\frac{1}{DP_t} - \frac{1}{DP_o} \right) \qquad (3)$$

    where DP_o and DP_t are the number average degree of polymerization in times 0 and t.

    It has been established that the initial stage of cellulose depolymerization follows equation 4[27, 24, 9-16].

    $$\frac{1}{DP_t} - \frac{1}{DP_o} = k' t \qquad (4)$$

    By combining equations 3 and 4, equation 5 is derived.

    $$\delta\% = k\,t \qquad (5)$$

    Equations 4 and 5 indicate that $1/DP_t - 1/DP_o$ and the percentage of the broken bonds are linear functions of the reaction time. The plot gradient of $1/DP_t - 1/DP_o$ or δ% versus time equals the rate constant k. By applying the treatment presented above, E and A can be determined and from them the rate constant at ambient temperature can be calculated. This approach overcomes the second difficulty mentioned before, because equations 4 and 5 have been derived by using kinetic principles and δ% represents true kinetic data, analogous to concentrations. Zou et al.[15] have provided persuasive theoretical and experimental evidence for its validity.

There are two prerequisites for either treatment to be valid[17, 18, 15]: The plot of the property (i.e. folding endurance or δ%) versus time and the Arrhenius plot must be straight lines. Nevertheless, both treatments have been criticized as being subject to large experimental errors, thus introducing major uncertainties to their results, especially when they are used for paper permanence estimations[6, 28, 29, 4]. It has been proposed that the linear equations 2, 4 and 5 apply only for ageing in ventilated ovens; in the case of prolonged ageing in sealed vessel, the process of degradation is supposed to be autocatalytic and an accelerating model applies instead of the linear[26].

Accelerated Ageing Experiments in Practice

By applying any of the two methods described above, the rate constants of the deterioration of the untreated and the treated paper can be determined at ambient temperature. The conservation treatment is considered beneficial if it lowers the rate constant. There are certain drawbacks though: apart from being controversial, these methods are laborious and time and sample consuming, especially if mechanical properties are measured. Two series of samples are needed, one for the treated and the other for the untreated samples. For any of them, it is necessary to perform accelerated ageing experiments at different temperatures (5 is the usual), so that the rate constant at ambient temperature can be determined. The ageing times at the lower temperature must be very long, in order to bring about measurable changes. In practice, the methods described above have been often used for the estimation of paper permanence or for the ranking of different papers according to their permanence, but are very rarely used for the evaluation of conservation interventions[30]. Instead, much simpler experimental setups are encountered in the relevant literature. The usual approach involves the comparison of the decrease of a property value of the treated samples to that of the untreated samples after accelerated ageing. Historically, this was the first approach for coping with the problem of ranking different papers according to their permanence[31] (TAPPI T453). It can be seen though, that this is theoretically valid only if the activation energy of the different papers is the same, so that the Arrhenius plots of the different papers are parallel[18]. Only in this case, the ranking of the papers is the same at ambient temperature and at the temperature of the accelerated ageing experiments. Nevertheless, accelerated ageing at only one temperature and the direct comparison of the decrease in the value of the paper property is the usual approach. Single temperature ageing experiments have been proposed lately for relative permanence studies[25].

The climatic conditions of the ageing experiments have also been the subject of investigation. For many years, ageing experiments were performed in dry circulating ovens at a temperature of 100-105ºC. The first ageing standard adopted this setup[31]. It has been established though, that the presence of some moisture was necessary in order to achieve results analogous to natural ageing[6, 32, 33]. Browning and Wink[17] proposed that the moisture content of paper at the conditions of the ageing experiments should be equal to that at ambient conditions, and this proposition was further supported by evidence[29]. It has also been proposed that up to 90ºC, the nature of the ageing process remains practically the same under the same RH[34-37, 29] (that is, the relative contribution of the different reactions is the same♦). Thus, a new standard was proposed and readily adopted, introducing 80ºC as temperature and 65% as relative humidity[40] (ISO 5630-3). The standard prescribed the use of circulating ovens, so that the products of paper ageing would be removed from the reaction space.

Recently, Shahani[23] proposed that ageing experiments should be performed in sealed enclosures, so that the volatile products of paper ageing remain in the reaction space. He speculated that they are acidic and autocatalyze the hydrolysis of cellulose, thus accelerating the depolymerzation reaction as ageing advances. Shahani believes that ageing is sealed vessels emulates natural ageing better than other experimental setups. The main arguments favoring his view are the following:

♦ Nevertheless, Arney et al.[38, 39] found different contribution of chain scission and atmospheric oxidation at different temperatures, the higher the temperature the more significant becoming the effect.

- A paper leaf stored in archives or libraries never ages alone, but together with large quantities of other paper, inside books or files.
- Inner book leaves exhibit lower strength and higher acidity than the outer ones. This finding has been attributed to the action of the trapped volatile acidic products of paper ageing. Similar results have been reported for paper sheets artificially aged in stacks or in sealed enclosures.
- Paper has a strong tendency to retain the acidic volatile products of its ageing.
- Paper that has been artificially aged in stacks or sealed vessels develops odor similar to that of old books.

Researchers readily accepted this setup[15], partly because of the convenience of its application, and a new standard (ASTM D6819-02e2) was developed according to this finding[25, 41]. The moisture content of paper is maintained equal to that at ambient conditions by conditioning the samples before the vessels are sealed or by using suitable saturated salt solutions[42].

Except from heat and humidity, other means have been used to accelerate the ageing of paper: light ageing and pollution with corrosive gasses typically found in modern city atmospheres (SO_2 and NO_x). These methods have not yet been standardized and a multitude of proprietary methods has been used (see table 1). Daniel[43] describes a pollution chamber for the accelerated deterioration of paper.

Experimental Setup

Important issues concerning experimental setup include experimental design, sample selection and preparation, number of samples and assignment of different treatments to different group of samples, choice of ageing method and time intervals.

The most usual experimental design encountered in the literature includes the creation of two groups of similar samples. One group undergoes the conservation treatment and than, both groups are artificially aged. Conclusions are drawn by the comparison of the residual properties (or their decrease) after ageing of the treated and untreated samples. In some studies, more than one ageing times are used and very rarely more than one temperature. When only the immediate effects of the treatment are of interest, the properties of treated and untreated samples are directly compared[44].

The paper samples usually include historic paper of the same composition and of the same historical period as the original paper that the conservation intervention is to be applied. Additionally, more historic paper of various compositions and ages is desirable in order to test the range of the applicability of the method. The conservation method is very often tested on pure cellulose paper, so that its results can be studied on a simple system consisting of the major paper component, that is, cellulose. Various grades of Whatman filter paper usually serve as model paper of pure cellulose as they are available worldwide, consist of α-cellulose (>98%), are free of additives and their properties exhibit relative repeatability[45, 10, 46-51, 37, 52-67]. Paper used as sample should be characterized as best as possible and its fibre origin and composition, sizing system and additives must be known in advance. Historic paper must be blank, since writing or printing interferes with the strength properties, as has been observed by Green et al.[49].

The number of samples varies according to the test methods included in the evaluation. For every test method, the number of samples suggested in the appropriate standard is used in most of the cases, although exceptions are not rare. The method for the assignment of the samples to the various treatments is not clearly stated in most of the relevant articles. Nevertheless, some researchers report random assignment while others apply statistical methods. The issue of the sample assignment is considered rather unimportant, since the paper consisting one sample source is considered

homogenous. It has been demonstrated though that even the same batch of a paper such as Whatman paper that has been manufactured with high standards and great care exhibits considerable inhomogeneity[67]. Thus, a method for the homogenous distribution of the samples among the different treatments is necessary.

The choice of the ageing method appears not to be critical, since, as it can be seen in table 1, a multitude of standardized and proprietary methods of ageing has been used, with ISO 5630-3 being the most widely practised. The same applies to the duration of the ageing and the time intervals used.

Conditioning of the samples is a very important matter concerning sample preparation before testing. The issue has been analyzed elsewhere[67] and will not be given here a lengthy account. We will only stress the significance of preconditioning on the credibility of the mechanical testing results[20, 67]. Mechanical testing is often done in both directions (MD and CD) but it has been suggested that only one direction might be sufficient[3].

Methods for the Evaluation of Paper Properties

In this chapter, the results of the bibliographical survey concerning the methods used for the evaluation of paper properties are reported*. Together with them, we present the methods that have been used in paper permanence studies, since the evaluation of a conservation intervention is virtually a comparative permanence study. We also present methods that have been used for the evaluation of paper deterioration, as monitoring the paper deterioration is required in such studies. The methods presented here include established methods already in use (table 2), various methods that have been sparingly used and methods that have never been used but have the potential to evolve and apply to specific problems of the evaluation (table 3).

Established Methods

Evaluation of Mechanical Properties

Folding Endurance

It equals the common logarithm of the number of the double folds that a paper strip held under tension can endure before it breaks[68, 11, 69, 70]:

FE = log (number of double folds) (6)

The method exhibits highly scattered results, but is very sensitive towards natural or artificial ageing[23], as folding endurance is proportional to the eight power of fibre strength[71]. Researchers[72, 73] believe that folding endurance expresses the usability of paper better than the other mechanical properties. For these reasons, it is the most commonly used mechanical property for paper permanence studies and for the evaluation of paper conservation interventions.

Folding endurance is very sensitive to relative humidity changes. A small increase in relative humidity can cause a disproportional increase in folding endurance, hence the climatic conditions must be strictly controlled during its measurement[74]. Accelerated ageing studies in dry (105°C) or humid circulating ovens[17, 18, 7] have shown that folding endurance usually (but not always[75]) decreases linearly with the time of ageing.

Various instruments can be used for the measurement of folding endurance, having different advantages or disadvantages: Kohler Molin, Lhomargy, MIT and Schopper[69]. The dimensions of the paper samples vary according to the instrument used. The corresponding International standard demands ten measurements at least, in both directions of paper, because the dispersion of the results is high. A serious disadvantage of the method is that it cannot be used for brittle, moldy and generally weak historic paper. Barrow et al.[72] describe a modified instrument for the measurement of the folding endurance of weak samples.

Tensile Properties

Under this heading, the following paper properties are classified:

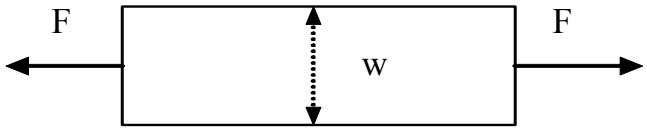
Tensile strength (TS): The tensile stress that causes the failure of the paper sample[68, 76, 77, 78]. The tensile stress is defined as:

$$\sigma \equiv \frac{F}{w} \quad (7)$$

Where:

* A more extensive selection of bibliographical references can be found in the Appendix, tables 2-4.

σ: Tensile stress, N/m, F: Tensile force, N, w: initial width of the sample, m.



Stretch at break, stretch, elongation (SAB): The elongation of the sample at the instant of failure.

Tensile Energy Absorption (TEA): The total work absorbed until the failure per square meter of paper. It can be calculated from the formula:

$$\mathrm{TEA} = \frac{\mathrm{E}}{\mathrm{w} \cdot \mathrm{l}} \qquad (8)$$

Where: E: work absorbed until sample failure, w: initial width and l: initial length of the sample.

Young's Modulus: It equals the gradient of the initial linear part of the plot of tensile strength versus time.

Zero Span Tensile Strength[79]: It is measured by the same apparatus as tensile strength, but with no separation left between the jaws that hold the sample. It serves as an index of the fibre strength.

Tensile strength is among the most favorite paper properties in paper conservation evaluation studies. However, it is not considered to determine the usability of paper, since a very brittle, thus unusable paper can exhibit a high value of tensile strength[80, 81, 73]. It is also insensitive to accelerated ageing, exhibiting little change even after long exposures. It has been demonstrated though, that tensile testing can record changes after aqueous treatments that indicate paper damage[67]. The literature survey showed that often, the tensile strength results, although collected, are not finally used for the evaluation.

Stretch at break and tensile energy absorption are both considered to connect better to paper usability than tensile strength. It is assumed that the more energy a paper sample can absorb (through deformation) before it fails, the more usable it is. Both SAB and TEA are considered more sensitive than TS and are often included in paper conservation evaluation studies.

Tearing Resistance

Tearing resistance is sensitive to a number of factors such as the length and strength of the fibers and the effectiveness of the connections between them. For papers exhibiting good cohesiveness of fibers, TR is proportional to the square power of the fiber strength[82]. TR is measured by the Elmendorf method as described by the appropriate standards[68, 83, 84]. It is used as a quality criterion for printing and writing papers. It is often included in paper conservation evaluation studies as a complementary mechanical property, with no special attributes. However, Barrow et al.[72] concluded from studying the functionality of book pages that testing tearing resistance is important in the cross direction. Roberson[20] considers tear testing to be important for paper permanence evaluation.

Bursting Strength

Bursting strength is used as a measure of the resistance of paper to rupture. It is defined as the hydrostatic pressure required to produce rupture of the specimen[68, 85]. The test is easy, fast and inexpensive and whenever it is included in the evaluation process it is not given any special attribute but evaluated together with the other mechanical properties. It has been especially used for the evaluation of the treatment of iron-gall ink corrosion with phytate[86].

Tensile post Fold

Bansa suggests that tensile strength after one defined fold (tensile post fold, tpf) is the most appropriate mechanical test for evaluation purposes[3, 80, 87]. He considers that it best suits the weak historical papers that are usually treated in a conservation workshop, the folding endurance test being to strong for them.

Evaluation of Chemical Properties

Determination of the Degree of Polymerization

The average Degree of Polymerization (DP) of cellulose is an important index of paper condition, since it is related to the average length of cellulose macromolecules. The decrease of the average length of the cellulose chains causes a decrease in the strength of paper fibers. DP measurement is the most popular chemical method found in the literature for the monitoring of paper degradation during ageing experiments.

The average DP of cellulose can be determined by direct methods based on various physicochemical properties[88, 16] (light scattering, sedimentation/diffusion, vapor pressure osmosis). However, these methods are complex, time consuming and demand complicated equipment. A simple and fast method for the determination of the DP is based on the determination of the viscosity of cellulose solutions. Capillary viscometers of various types can be used and the most commonly used solvent is cupriethylenodiamine (CED). Thus, the so-called "viscosity average degree of polymerization", DP_v, of cellulose is determined, which is approximately equal to the weight-average DP, DP_w[16].

Viscosity is connected to the DP_w by an empirical formula[89, 16], known as the Mark – Houwink – Sakurada equation:

$$[\eta] = K\, DP_w^{\alpha} \qquad \text{(Mark – Houwink – Sakurada equation)} \qquad (9)$$

The symbols K and α are used for experimentally determined constants and [η] is the intrinsic viscosity that depends only on the DP of cellulose and is independent on the solvent viscosity, the polymer concentration and the viscometer type.

The intrinsic viscosity is defined by the formula[16]:

$$[\eta] = \lim_{c \to 0} \frac{\eta_{spec}}{c} \qquad (10)$$

where $\eta_{spec} = \frac{t - \tau}{\tau} = n_{rel} - 1$ = specific viscosity and c = concentration of the solution, t = solution efflux time, τ = solvent efflux time, $n_{rel} = n_{solution}/n_{solvent} = t/\tau$. Intrinsic viscosity can be determined by consequent measurements at different polymer concentrations and extrapolation to zero concentration. Tables (ASTM standard D 1795 – 96[90]) or empiric formulae[89, 16] can also be used that allow the calculation of the intrinsic viscosity from only one measurement of the specific viscosity of a dilute solution (usually 0.5%).

A practical drawback of the method lies in the selection of the values of the constant K and α, which depend on the solvent, are influenced by the region of the viscosity values• and are taken from tables. These values have been determined after calibration with other direct methods and may differ significantly, depending on the

• From the results of Bicchieri et al.[91, 92], it seems that these authors used the following values for the constants: α = 1 and K = 0.807 for [η] < 199 and K = 0.64 for [η] ≥ 199 ([η] in ml/g). The same values appear in Grobe[89] and Klemm et al.[16], with K changing at [n]=240. Margutti et al.[64] used: invariable K = 0.666 ml/g and α = 1.

calibration method, the kind and the origin of cellulose and the researcher. The literature survey showed that from a multitude of choices, the most commonly used values for CED at 25°C are: K between 0.807 and 0.64 ml/g (for values of [η] between 100 and 2140 ml/g, Grobe[89] p. 146 table. 1.1., Klemm et al.[16], they result after calculation from the values of K in table 3.1.1. p. 172 and the M.M. of anydroglucose, 162) and a = 1. However, it is common and more practical to use the tables provided by the appropriate ASTM standard[90], where [η] is directly derived by the value of n_{rel} and the concentration, and calculate DP after multiplying [n] (in g/dl) by 190.

The average degree of polymerization can also be determined by Gel Permeation Chromatography (GPC) or Size Exclusion Chromatography (SEC), together with the molecular mass distribution of cellulose, which provides data about the randomness and the mechanism of depolymerization[93, 13, 14, 94, 95].

PH Determination

The detrimental effects of acidity on paper have been well documented: catalysis of acid hydrolysis of cellulose molecules, enhancement of the oxidative action of metal ions, light and oxidative agents. The pH of paper functions as an index of the alkalinity or acidity of paper[75], permitting among others the rough estimation of the permanence of paper and the evaluation of deacidification treatments.

One must bear in mind that since paper is not a solution, pH cannot be defined for it. Instead, an empirical definition of paper pH has been introduced, specifying that it equals the pH of the extract of a certain quantity of paper in a volume of water. The exact quantities of paper and water depend on the standard used (for ISO 6588[96] they are 2 g. of paper in 100 ml of water). Either cold or warm water can be used for the extraction of paper, resulting in two different methods. The cold extraction is usually preferred for the evaluation of deacidification because it exhibits a better association with the ageing of paper[20]. It has been proposed that the hydrogen concentration inside the fiber can be determined more accurately by extraction in 0.1 N sodium chloride solution[97, 13].

Determination of Alkali Reserve

The term “alkali reserve” is used for an alkaline compound introduced into paper in order to neutralize the acids that might be produced in the paper itself or absorbed by the environment in the future. Alkali reserve is introduced to paper through deacidification, whose purpose is to protect paper from acid hydrolysis. An alkaline compound is used for the neutralization of the current acidity and the excess left on paper forms the alkali reserve. Thus, the determination of alkali reserve is associated with deacidification and is used for the evaluation of deacidification interventions.

The method consists in titrating with NaOH solution the excess of HCl solution added to the suspension of 1 g. of paper in water and is described in ISO 10716[98]. Liers[99] has published a detailed method for the determination of alkalinity or acidity of paper which is also based on titration.

Determination of Copper Number, Kappa Number

Copper number (CN) is defined as the number of grams of metallic copper (as Cu_2O) resulting from the reduction of $CuSO_4$ by 100 g of paper fibres. The copper number may be regarded as an index of those impurities in paper, such as oxycellulose, hydrocellulose, lignin and sugars, which possess reducing properties[100]. High values of copper number of lignin-free papers are an indication of unstable and degraded cellulose. An increase in copper number after ageing indicates deterioration. Copper number has not been extensively used for the evaluation of paper conservation

interventions, probably because of its empirical nature; nevertheless, a number of researchers include it in the process of the evaluation. Kappa number is another index of the oxidizable content of paper (it is used in paper industry as an indication of the lignin content) and has been used sparingly instead of copper number.

Infrared Spectroscopy

Infrared spectroscopy, most usually in the form of Fourier Transform Infrared Spectroscopy (FTIR) has been used in the following relevant fields of paper study:

- For the study of paper ageing[64, 101-104] and foxing[105]. For the determination of crystallinity changes due to ageing[106, 107]. For the identification of paper degradation products (cellulose chain fragments and their oxidation products) after sample extraction[54]. For the determination of characteristic groups (carbonyls, carboxyls, amines, conjugations etc.)[108, 109, 107]. For the study of the influence of copper compounds[110] and ferric and copper ions on paper ageing[111]. For the study of the composition and the degradation of modern and historic paper[112]. Johansson et al.[113] used DRIFTS (Diffuse Reflectance Infrared Fourier Transform Spectroscopy) for the study of the effects of the atmospheric pollutants on paper. Yang et al.[114] used FTIR-PAS (FTIR Photoacoustic Spectroscopy) for the study of cotton-cellulose photooxidation.
- For the quantitative determination of lignin, cellulose and xylose[115]. For the identification of lignin[116], gelatin[117, 105, 103, 118, 119], and various paper additives[120, 121].
- For the evaluation of paper conservation interventions (deacidification[122], laser cleaning[109, 62]).

FTIR microscopy (μFTIR) is an ideal non-destructive method for the determination of paper surface composition. μFTIR can be used for point to point examination of the sample surface, thus providing information about the distribution of a compound on the paper surface[101].

Evaluation of Physicochemical Properties

Optical Properties - Colorimetry

The optical properties of paper concern the reflection and the absorption of light by it. The main optical parameters describing them are the following:

- Absorption Coefficient (k)[123, 124, 125]: An index of the light absorption of paper. At a given wavelength, it is the product of the Beer-Lambert extinction coefficient and the concentration of chromophores.
- Scattering Coefficient (s)[123, 124, 125]: An index of the light scattering of paper. It is determined by the fibre dimensions and the degree of interfibre bonding.
- Brightness (B) is a precisely defined measurement of the reflectance of visible blue light from an opaque stack of paper[124-127].
- Opacity is the ratio of diffuse reflectance measured on a single sheet of paper backed by a black material to that measured with the sheet backed by a white material[125, 128, 129].

The optical parameters k, s and B of paper are related by the Kubelka-Munk equation[123, 124, 125]:

$$\frac{k}{s} = \frac{(1 - B)^2}{2B} \qquad (11)$$

Color is filed among the optical properties of paper and can be measured according to the standards TAPPI T 524[130] and TAPPI 527[131]. Lately, the L*, a* and

b* coordinates of the three-dimensional CIEL*a*b* (1976) color space[132] have been introduced[133, 134, 59] and gradually started to replace the "traditionally" used[135-140] optical parameter "brightness" in paper conservation evaluation studies. The polar coordinates L* (Lightness), a* (position in the red-green axis) και b* (position in the yellow-blue axis) are vectors which are normal to each other.

The total color difference between two paper samples can be calculated by the formula[141]:

$$\Delta E = \left(\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}\right)^{1/2} \quad (12)$$

where ΔL*, Δa* and Δb are the differences of the three coordinates.

The determination of the optical properties is an easy, fast and non-destructive process. Specialized instruments can be used (colorimeters) that can directly measure brightness and color coordinates of the CIEL*a*b* color space. They can also measure the whiteness index and the yellowness index (Wi, Yi, ASTM standard E 313-96[142]).

The contribution of color measurements to the evaluation of paper conservation interventions is invaluable, since they make possible the objective esthetic evaluation. Color measurements have also been used to monitor paper degradation, either as a natural process or after accelerated ageing. Chemical reactions detrimental to paper, such as the oxidation of the paper components or of the products of their degradation, produce chromophores (chemical species that usually absorb in the blue region of visible light, thus appearing yellow) that reduce the brightness and increase the yellowness of the samples. At the same time, successful cleaning procedures increase brightness and reduce yellowness. Thus, the useful optical parameters that should be monitored are brightness (or lightness, or whiteness index) and the b* coordinate of the CIEL*a*b* color system[134, 143] (or yellowness index). An increase of brightness (lighter paper) and a decrease of b* (less yellow paper) are desirable, since such changes increase the contrast between text (or image) and paper, increasing thus the document legibility. The inverse changes are considered detrimental, since they decrease legibility and manifest degrading chemical reactions.

Optical Microscopy

It has been mentioned above that the paper samples that are used for the evaluation of a paper conservation intervention should be defined and characterized as best as possible. Optical microscopy is a useful tool for the evaluation of the quality and the determination of the origin of the paper fibers. It can also be used for defining the pulping method and the processing of the pulp (degree of beating, bleaching method).

The method consists in the microscopic observation of properly prepared samples of paper fibers (about 0.25 g.) after staining with various reagents that dye the fibers selectively[68, 144-151]. In the case of mixture of fibers of different origins and processing (i.e. different plants, chemical or mechanical pulps), a quantitative determination of the proportion of the different components is possible. There are several publications presenting representative microphotographs of fibres of different plants that can be useful for the determination of the fibre origin[152-156].

Nevertheless, optical microscopy is useful for the evaluation itself, since it can be used for the observation of the paper texture and its alterations due to the conservation treatment (excessive pressing, depositions etc.). It can also be used for the identification of microorganisms and for the evaluation of disinfection methods.

Scanning Electron Microscopy (SEM) and X-ray Microanalysis (EDS, Energy Dispersive Spectroscopy)

Electron microscopy (SEM) offers better resolution than the optical microscopy and in combination with EDS can be used for acquiring topochemical data of the paper surface. Grant[68] proposed the method for research purposes in the filed of paper in the early sixties. The use of SEM and EDS for the surface analysis of paper is described by De Silveira et al.[157, 158]. The field of application, the potential and the limitations of the method for the analysis of paper, together with the preparation of the sample, are described in detail by Friese et al.[121].

The combination of SEM and EDS has been used for the determination of the origin of paper damage[159], for the identification of additives and impurities[159, 101] and for the determination of the distribution pattern of the deacidification agent[159, 160]. SEM has been used for the observation of the morphological alterations of the fibers of old and brittle paper[159, 160, 101], for the identification of fungi, fungi spores and fragments[159], for the identification of insects, parasites and their eggs[159, 161] and for the investigation of the results of eraser dry cleaning on paper[159].

Other Methods

Many other methods have been used by various scientists for the evaluation of paper conservation interventions and for the evaluation of paper deterioration (monitoring of the ageing process and determination of the current status of preservation of a paper object) or paper permanence (table 3, see appendix). The most important of them are:

- Thermal Analysis[162-165]. Thermogravimetry (TG) has been used by Cardwell and Luner[166, 167] for the studying of various pulps and the establishment of stability criteria. Basta et al.[168] used TG for the evaluation of the effects of grammage and gelatin on the durability of paper. Differential Thermal Analysis (DTA) and Differential Scanning Calorimetry (DSC) have been used for the studying of the thermal decomposition of cellulose[9] and for the evaluation of the stability of various pulps[33] and papers[20, 21, 169]. Toth et al.[170, 171] used Dynamic Thermo-Mechanical Analysis (DTMA) for the studying of paper ageing. The application of these methods for paper permanence evaluation gave moderate results and they seem to have a potential for the evaluation of paper conservation interventions, especially DTMA.
- Determination of α-cellulose, alkali solubility[172, 173]. These empirical methods are based on the same principal, and they consist in the determination of the soluble fraction in strong alkali. The lower the α-content and the higher the alkali solubility, the more degraded the cellulose is. The methods have been used for the evaluation of paper permanence and were found to correlate well with paper stability[20, 32, 33].
- Functional Group Determination: Carbonyl and Carboxyl content. High carbonyl content destabilizes cellulose and promotes chain scission[174-176]. High carboxyl and carbonyl content is an indication of oxidized cellulose.
- Ultrasonic testing has been used for the non-destructive determination of the preservation status of historical papers[117, 119] and for the monitoring of the ageing process[177]. It has been attempted to correlate ultrasonic specific modulus of paper to the gelatin content with moderate success[118].
- Fluorescence has been used for the study of foxing stains[178, 105, 179] and of the brown lines at the wet-dry interface[180]. Barrett et al.[118] used fluorescence measurements for the quantitative determination of gelatin.
- The Russell Effect. The autoxidation of organic materials and metals can be detected photographically. This phenomenon is called Russell Effect after its

discoverer. Daniels presents the method and reviews its possible uses in conservation and in the examination of materials[181-183]. Caverhill et al.[109] used Russell images for the determination of peroxide activity in their study of laser cleaning of paper as a conservation technique.

Table 3 also presents methods that apply to specific problems of paper conservation evaluation and methods that have never been used but have the potential to evolve. Some of them concern newly developed methods for more detailed microscopic observation, 3-D topography and topochemical mapping and others chemical mapping of the paper surface and additive identification. Other methods concern the identification of paper degradation products, some of them being conventional and some others modern. These auxiliary methods can help in understanding and visualizing what really happens at chemical and physical level as the result of the conservation intervention under study and what are the effects of ageing. Non-destructive methods like ultrasonic testing deserve special attention and need further development, because, since they can apply to the same object before and after the conservation treatment, they could be the ideal tools for the evaluation.

Criteria

The literature survey yielded the following list of criteria for the characterization of a paper conservation intervention as successful or unsuccessful. Most of them are not explicitly stated but are implied in most of the publications studied.

Successful intervention

- Immediate improvement of paper properties:
  - Increase of strength (usually as a result of consolidation)
  - pH increase (deacidification, up to 9 – 9.5 results chemical stabilization)
- Deceleration of paper ageing (chemical stabilization). It is evaluated by comparing the deterioration rate of the main properties of the treated paper to that of the properties of the untreated paper during accelerated ageing. Treated paper should also have better strength properties, higher DP and pH and less oxidized group than the untreated after ageing.
- Esthetic improvement:
  - A slight (and even major in some cases of objects of high esthetic or symbolic value) esthetic improvement is desirable
  - Cleaning results in the improvement of contrast between text and substrate and increases legibility

Unsuccessful intervention

- Immediate deterioration of paper properties:
  - Loss of strength (mechanical properties)
  - Decrease of DP, increase of oxidized group content (chemical properties)
- Acceleration of paper ageing
- Alteration of the interpretation of the object
- Alteration or destruction of the original elements of the structure and the materials of the object
- Esthetic degradation[80]:
  - Alteration of the visual appearance of the object: discoloration, smudging or running of inks and dyes, changes in the color of paper (excessive cleaning or yellowing due to lignin oxidation)
  - Perceptive alteration of the tactile properties of paper: changes in paper weight, thickness and stiffness; changes of the roughness of the paper surface. Such changes occur in almost every treatment, but are difficult to quantify and the minimum perceptible change differs from one person to the other.

In the hypothetical case that the intervention has neither positive nor negative results, it is considered unjustified and unsuccessful, since it unnecessarily alters the original state of the object. The esthetic aspect of the evaluation appears to pose certain difficulties, since it is partially based on subjective criteria. The contribution of colorimetry to it is invaluable. The consultation with a board of referees can reduce the subjective factor[80].

As a rule, a paper conservation intervention has both positive and negative effects. Thus, the ranking of the criteria is inevitable for the final assessment and this is where subjectivity sometimes interferes. Nevertheless, as paper conservation evolves and feels the influence from other fields of the conservation science, the ranking order seems to change in favor of minor and less interfering interventions.

Conclusions

Summarizing the results of the survey, we can see that:

- It is common practice to study the results of the conservation interventions on a standard paper such as Whatman filter paper, apart from original historical paper.
- A method for the accelerated ageing of the samples is utilized in most of the cases in order to demonstrate the future effects of the intervention. The most widely used ageing method is the one described in ISO 5630-3[40] (80°C and 65% RH). It has been proposed recently that ageing in sealed vessels simulates natural ageing better than ageing in ventilated ovens.
- The evaluation of a paper conservation intervention can be seen as a comparative permanence study of the treated and untreated paper. Thereby, the methodology of permanence study can apply to the evaluation of a conservation treatment.
- Various physicochemical properties of the samples are determined before and after ageing for reference and treated samples. Ageing is applied for one or several periods of time, the latter case allowing for a more in depth study of the change of the property.
- From the mechanical properties, the most popular is folding endurance. Tensile strength, tensile energy absorption, stretch at break and tearing resistance are used less frequently. Since it is mainly folding endurance and to a lesser extent tensile energy absorption and not tensile strength that determine the usability of paper[72, 184, 73, 80] and considering the sensitivity of folding endurance towards accelerated ageing, it seems plausible that this mechanical property should be included in the evaluation protocol.
- As far as chemical properties are concerned, the most widely used is pH, which also serves as a crude stability index. The determination of the degree of polymerization allows for a more detailed study of the deterioration process during ageing. FTIR spectroscopy can supply data on the chemical changes during ageing.
- Colorimetry contributes significantly to the evaluation process, especially to the aesthetic part of it. It also provides data that can be interpreted at a chemical level.
- There are several other methods that are either used for the characterization of the samples or that apply to different aspects of the evaluation. Some of them are in use while others are not, and a number of them have the potential to evolve.
- Specific attention should be given to the further development and the application of non-destructive methods that can measure mechanical strength, such as ultrasonic testing.

We believe that apart from answering if the conservation method in question is beneficial or not, experimental evidence must be supplied that explain how paper is affected by the treatment and that the mechanism of the alterations induced should be studied. That is why we think that except from the experimental protocol used for the evaluation, complementary methods that allow for a more in depth examination of the effects of the treatments are necessary. Wilson and Parks[6] recommendations that were presented in the introduction can help choose the most appropriate methods. Depending on the conservation method under study, one or more from the other methods presented above, can also serve that purpose.

The literature survey pointed out that there are certain gaps in research concerning the evaluation methodology of paper conservation interventions, mainly because the evaluation methodology itself is never at the focus of the investigation. Statistical data resulting from a rigorous analysis of the results of the various methods applied on the same untreated, treated, and aged standard and historical paper samples

are lacking, though they would indicate the most efficient, sensitive and repeatable methods. Although statistical data such as standard deviation, repeatability and reproducibility can be found in the appropriate standard (ISO or TAPPI) describing the method, these data refer to modern untreated paper and must be far from the figures that would result from treated and aged historical paper. A comparative statistical analysis could also reveal possible correlations among the results of different methods, giving thus the opportunity to eliminate laborious methods that correlate well to simple and fast methods[185]. Statistical tools (student t-test, ANOVA) should also be used for the purposes of the evaluation itself, in order to check if the differences found are statistically significant or not.

In a follow-up paper, the most important methods yielded by this survey will be laboratory tested and the results statistically elaborated. Based on these results, the most sensitive and repeatable methods will be chosen and a loose experimental protocol for the evaluation of paper conservation interventions will be proposed.

Appendix

Reference	Method of Ageing - Conditions*	Methods used for the Evaluation*	Aim of the Study
Baer et al. 1972 (135)	100ºC, DO for 1, 5, 9, 16 d	FE, pH, CoC, B	Evaluation of consolidation with: PVAl, soluble nylon, Regnal
Baer et al. 1977 (19)	21ºC, 50%RH - 60ºC, 10%RH - 80ºC, DO - 95ºC, DO - 100ºC, DO for 1, 5, 9, 16 and 50 d	FE, TS	Evaluation of consolidation with: PVAl, soluble nylon, Regnal, various adhesives, PVA
Kelly et al. 1977 (136)	100ºC, DO for 36 d	B, pH, FE, TS	Evaluation of methylmagnesium carbonate as a deacidification agent
Walker 1977 (137)	100ºC, DO for 2, 4, 6, 8, 10, 12 d – 75ºC, 60%RH + 5 ppm SO_2	pH, FE, B, TR, TS	Evaluation of morpholine deacidification in gaseous phase
Williams et al. 1977 (186)	90ºC, 50%RH - 100ºC, DO for up to 645 hours	FE, pH, B	Catalytic effect of transition metals on paper ageing
Donnithorne 1979 (187)	105ºC, DO for 72 hours	pH, FE	Chlorine dioxide bleaching
Hey 1979 (138)	90ºC, 50%RH for 3 d	B, pH, AR	Water washing and deacidification
Arney et al. 1981 (8)	90ºC, 100%RH in sealed glass tubes for up to 500 hours	TS, pH, AAS	Influence of acidity on the accelerated ageing of paper
Kelly et al. 1981 (188)	88 h LA at 60ºC, 60%RH	FE	Use of I^- for the inhibition of light-sensitization of paper caused by ZnO (DEZ deacidification method). Effectiveness of ZnO conversion to $Zn(CO_3)_2$
Tang 1981 (139)	90ºC, 50%RH - 100ºC, DO for 7, 14, 21, 35 d	AAS, B, FE, pH, A, AR	Evaluation of washing and deacidification in the same operation
Williams 1981 (140)	100ºC, DO for up to 16 d	B, FE	Effect of glycerin, sorbitol and a resin (kymene 557) on the folding endurance of deacidified paper
Wilson et al. 1981 (184)	90ºC, 50%RH	FE, pH, TR, B	Effect of magnesium bicarbonate
Baker 1984 (189)	90ºC, 56%RH for 16 d	FE, pH, Munsell color	Evaluation of methylcellulose and sodium carboxymethylcellulose for use in paper conservation
Block et al. 1986 (175)	100 – 150 ºC, DO for up to 100 h	TS, TR, CoC	Effect of tetrahydridoborates on the ageing rate of cellulose
Burgess 1986 (93)	70ºC, 50%RH - 70 d	DP, GPC	The effect of water washing on the long-term stability of cellulose
Daniels et al. 1986 (45)	LA	Mass Spectroscopy	The photoyellowing of thymol
Strzelczyk et al. 1986 (47)	105ºC, DO for 6, 17, 35 d	W, pH, α-cellulose, TS, TR	Evaluation of the use of quaternary ammonium salts for the disinfection of paper
Tang 1986 (176)	90ºC, 50%RH - 100ºC, DO for 7, 14, 21, 35 d	B, FE, pH, AAS	Stabilization of paper through borohydride treatment
Bredereck et al. 1988 (190)	105ºC, DO for 3, 10 d - 90ºC, 70%RH for 3 d	TS, W	Evaluation of ink fixatives
Calvini et al. 1988 (122)	60ºC, 65% for 136 d	pH, AR, B, DP, Infrared Analysis, CaC, carbonyl content	Effects of deacidification
Barrett 1989 (117)	80ºC in sealed glass tubes for 10, 15, 20, 27, 81 d	pH, TEA, FTIR, PIXE, XRF, XRD	Comparison of production methods of contemporary handmade paper
Bredereck et al. 1990 (191)		AR, pH, AAS, titration	Evaluation of various deacidification methods and techniques
Daniel et al. 1990 (192)	SO_2 13 ppm, NO_2 4 ppm up to 12 weeks	pH, AR, W, DP, CN, FE, BS, TS	Evaluation of the protection from atmospheric pollution offered by 4 deacidification methods
Lienardy et al. 1990	105ºC, DO for 2 weeks	W, TS, FE, pH	Paper washing

* FE: Folding Endurance, TS: Tensile Strength, TpF: Tensile post Fold, DP: Degree of polymerization, BS: Bursting Strength, TR: Tearing Resistance, DO: Dry Oven, h: hours, d: days, LA: Light Ageing, AAS: Atomic Absorption Spectroscopy, B: Brightness, Y: Yellowness, A: Acidity, AR: Alkaline Reserve, W: Whiteness, DP: Degree of Polymerization, CN: Copper Number, TEA: Tensile Energy Absorption, ZSTS: Zero Span Tensile Strength, SAB: Stretch At Break, O: Opacity, M: Microscopy, CoC: Color Change (mainly ΔE* CIEL*a*b*), n: viscosity, AEF: Alkali – Extractable Fraction, CaC: Carboxyl Content, FTIR: Fourier Transform Infra-Red Spectroscopy, GPC: Gel Permeation Chromatography, GC: Gas Chromatography, XRF: X-Ray Fluorescence Spectroscopy, CIEL*a*b*: Coordinates of CIEL*a*b* color system.

(193)			
Lienardy et al. 1990 (194)	Dry and Humid Ageing	FE, TS, pH, DP, AR, B	Evaluation of 7 deacidification methods
Vodopivec et al. 1990 (44)		TS, SAB, BS, FE	Evaluation of synthetic polymers for use in paper conservation
Durovic et al. 1991 (48)	103ºC, DO for 1, 3, 6, 9, 18 d – 60ºC and cycling RH from 40% to 95% for 3, 6, 12, 18, 30 d	pH, FE, TS, W, O	Evaluation of the use of PVAC and other dispersive glues for paper consolidation
Green et al. 1991 (49)	60ºC for 28 d	M, TS, pH	Evaluation of the use of MMC for paper deacidification
Wedinger 1991 (2)	90ºC, 50%RH for 7, 14, 21, 28, 35 d	TS, TEA, SAB, ZSTS, TR, FE, pH, AR, B, Y, CN	Evaluation of the FMC mass deacidification System
Bansa 1992 (3)	80ºC, 65%RH	pH, TS, TpF, TR, FE	Recommendations for the evaluation of paper conservation interventions
Shapkina et al. 1992 (195)	UV ageing	SEM, TS, FE	Evaluation of dry leafcasting
Slavin et al. 1992 (196)		pH, GC	Environmental factors in migration-induced degradation of paper
Bicchieri et al. 1993 (50)	95ºC, DO for 7, 14, 28, 56 d – 85ºC, DO for 7, 14, 21 d – 80ºC, 65%RH for 1, 2, 3 ,4, 5 ,6, 7 d 23ºC, 50%RH for 10 years	Reversibility, pH, DP, O, TS, B, FE	Evaluation of the use of PVAl in paper conservation
Vallas 1993 (197)		pH, AR	Evaluation of the mass deacidification system used at the Bibliothèque Nationale of France
Botti et al. 1994 (198)	80ºC, 65%RH 30 and 60 d – DO 105 ºC for 3 and 9 d	B, FE, TS, TR, pH, DP, M, AR, gurley air permeability, α-cellulose	Paper packaging for the long-term preservation of photographic plates
Brandis 1994 (73)	90ºC, 50%RH for 3, 6, 9, 12, 18, 24, 30 d	pH, AR, FE, TS, SAB, TEA, alkali solubility, B, O	Evaluation of the FMC, Wei T'o and Akzo mass deacidification systems
Hanus 1994 (199)		TS, TEA, SAB, FE, pH	Changes in brittle paper during conservation treatment
Lienardy 1994 (133)	90ºC, 60%RH for 14 d – 96 h LA σε 50ºC, 50%RH	CoC, pH, AR, DP, CN	Evaluation of the 7 Mass Deacidification Systems
Strnadova et al. 1994 (51)	80ºC, 65%RH for 24 d	n, W, FE, TS, pH,	Evaluation of the use of cellulose ethers in paper conservation
Stroud 1994 (200)		pH, AR, X-ray photoelectron spectroscopy, SEM, UV-fluorescence microscopy	Evaluation of the HRHRC Diethyl Zinc mass deacidification project
Wittekind 1994 (201)	80ºC, 65%RH for 12, 24, 50, 100 d	Tear Length	Evaluation of the Battelle Mass Deacidification System
Bluher et al. 1995 (202)	22 h LA – 90ºC and cycling RH from 50% to 80% every 12 hours for 72 h	pH	Evaluation of the use of Carbopol poultices for the wetting of paper
Guerra et al. 1995 (203)		FE, TS, pH, IR, M	Evaluation of simultaneous deacidification and sizing of paper
Hanus et al. 1995 (204)	103ºC, DO for 3, 6, 12, 24 d	pH, FE, TR, TS, SAB, TEA	Evaluation of the influence of boxing materials on the properties of different paper items stored inside
Havermans et al. 1995 (205)	90ºC, 50%RH for 12 d – SO_2 (10 ppm) and NO_x (20 ppm) for 4 d at 23ºC, 50%RH	pH, CN, AEF, TR, FE, DP, AR, SEM, EDX	Evaluation of the mass deacidification of archival materials using diethyl zinc
Havermans 1995 (206)	90ºC, 50%RH for 12 d – SO_2 (10 ppm) and NO_x (20 ppm) for 4 or 12 d at 90ºC, 50%RH	pH, CN, AEF, ZSTS, DP, FTIR	Effects of air pollutants on the accelerated aging of cellulose-based materials
Neevel 1995 (86)	90ºC and cycling RH from 35% to 80% every 3 hours for 3, 6, 12, 18 d – 90ºC, 50%RH for 3, 6, 12, 18 d	BS, pH	Evaluation of calcium phytate as a conservation agent for the treatment of ink corrosion caused by irongall inks
Suryawanshi et al. 1995 (207)	105ºC, DO for 48 h	TS, TR, BS, FE, α-cellulose, A, CN	Evaluation of hand-made Nepalese paper for lining paintings
Bicchieri et al. 1996 (91)	80ºC, 65%RH for 7, 14, 21, 28 d	pH, B, CaC, n	Influence of ferric and cupric ions on the degradation of cellulose
Bicchieri et al. 1996 (92)	80ºC, 65%RH for 7, 14, 21, 28 d	pH, B, CaC, n, CoC	Evaluation of hydroxypropyl cellulose and polyvinyl alcohol as fixatives for pigments and dyes on paper

Dupont 1996 (53)	80ºC, 65%RH for 24 d		Degradation of cellulose at the wet/dry interface. The effect of some conservation treatments on brown lines
Hofenk de Graaff et al. 1996 (208)	SO_2 (10 ppm) and NO_2 (20 ppm) at a flow of 800l/h for 3 months	pH, AR, XRF	The effect of alkaline boxes and file folders on the accelerated ageing of paper by air pollution
Kolar et al. 1996 (209)	80ºC, 65%RH for 28 d	n, pH, B	Effect of various deacidification solutions (calcium hydroxide, magnesium bicarbonate) on the stability of cellulose pulps
Liers et al. 1996 (210)		TS, pH, acid and alkali content	Evaluation of paper splitting
Sistach 1996 (160)		TS, SEM, EDS, M, pH	Results of deacidification
Stauderman et al. 1996 (211)	90ºC, 50% RH for 7 d	AAS, SEM, M, pH, CIEL*a*b*	Evaluation of the use of Bookkeeper deacidification spray for the treatment of individual objects
Suryawanshi et al. 1996 (212)	105ºC, DO for 24, 48, 72 h	B, FE	Evaluation of adhesives and supporting materials for the process of lamination of old documents
Bansa et al. 1997 (80)	90ºC, 50%RH for 518 h	TpF, pH, CoC, thickness, stiffness	Effect of different strengthening methods on different kinds of paper
Bicchieri et al. 1997 (134)		CIEL*a*b*, DP	Evaluation of the use of borane tert-butylamine complex for the bleaching of paper
Bukovsky 1997 (213)	Indirect natural light ageing for 1, 16, 55, 79, 134, 185 d – natural ageing (no light access) for 51/2 years	AR, B, carbonyl content	Effect of deacidification with methyl magnesium carbonate on the yellowing of newspaper
Lehtaru et al. 1997 (214)	75ºC, 40%RH for 28 d	B, CaC, carbonyl content	Use of chelating agent EDTA with sodium thiosulphate and sodium borohydride in bleaching treatment
Letnar et al. 1997 (215)	80ºC, 65%RH for 6, 12, 24 d	pH, DP, CN, TS, SAB, BS, TR, FE, stiffness, W, Y, B, porosity	Influence of paper raw materials and technological conditions of paper manufacture on paper aging
Letnar et al. 1997 (216)	80ºC, 65%RH for 6, 12, 24 d	pH, DP, CN, TS, SAB, BS, TR, FE, stiffness, W, Y, B, porosity	Evaluation of permanence and durability of the laminated material on paper
Schaeffer et al. 1997 (55)	70ºC, 50%RH for 6 weeks	CIEL*a*b*, pH, TS, SAB	Evaluation of aqueous light bleaching of modern rag paper
Zappala 1997 (217)	Wet and dry ageing	AAS, DP, B, pH	Conservation of acid paper
Adamo et al. 1998 (56)	80ºC, 65%RH for 12, 24 d	CIEL*a*b*, TS, TR, FE, BS, DP, pH	Effect of gamma rays (biological recovery treatment) on pure cellulose paper
Bansa 1998 (87)	105ºC, DO for 3, 6, 12 d – 80ºC, 65%RH for 12, 24, 48 d	CIEL*a*b*, TpF, FE, pH, DP	Comparison of aqueous deacidification methods based on calcium and magnesium
Begin et al. 1998 (218)	80ºC, 65%RH for 5, 20, 50 d	ZSTS, TR, SAB, TEA, FE, B, pH, AR, DP	Impact of lignin on paper permanence
Guerra et al. 1998 (219)		TS, TEA, SAB, FE, elastic modulus	Effect of alkoxypolyethyleneglycols (deposited on paper as a side effect of deacidification) on the mechanical properties of paper
El-Saied et al. 1998 (220)	103ºC, DO for 3, 6, 12 d	α-, β- γ- celluloses, CaC, DP, pH, alkali solubility, B, W, O, FE, TR, BS, TS	Problems and permanency of alum-rosin sized paper sheets from wood pulp
Bansa et al. 1999 (81)		FE, TS, TR, TpF, CIEL*a*b*, thickness, stiffness	Evaluation of fibers of different origin for the strengthening of paper
Begin et al. 1999 (221)	80ºC, 65%RH for 5, 20, 50 d – 80ºC, 65%RH for 1, 5, 12 d	DP	Effect of air pollutants on paper stability
Bicchieri et al. 1999 (58)	80ºC, 65%RH for 7, 14, 21, 28 d	UV-VIS determination of carbonyls	Quantitative measure of borane tert-butylamine effectiveness in carbonyl reduction of aged paper
Bukovsky 1999 (222)	103ºC, DO for 12 d	FE, pH, CaC, carbonyl content, B	Evaluation of deacidification as a means for rescuing historic newspapers
Caverhill et al. 1999 (109)	48 and 120 h LA (UV) - 90ºC, 50%RH for 7, 30 d	Russell effect, CaC, FTIR, CoC	The effect of aging on paper irradiated by laser as a conservation technique
Havermans 1999 (223)	70ºC, 55%RH for 24 d	pH, CN, W, FE, TR	Aging behavior of encapsulated paper

Nada et al. 1999 (224)	80ºC, 30%RH for 3 d – 80ºC, 60%RH for 3 d – 80ºC, 94%RH for 3 d	TS, TR	Evaluation of the use of emulsified polymers for the consolidation of deteriorated paper
Schwarz et al. 1999 (60)	80ºC, 65%RH for 7 d - 90ºC and cycling RH from 35% to 80% every 3 hours for 3, 6, 12 d	DP	Development of a ready-for-use pad to locally remove starch with enzymes
Sistach et al. 1999 (59)	70ºC, 35%RH for 7, 27 d	CIEL*a*b*	Ageing of laboratory irongall inks
Uyeda et al. 1999 (225)	80ºC, 65%RH for 12 weeks	TS, FE, B, pH	Effect of cooking agents on Japanese paper
Bicchieri et al. 2000 (61)	80ºC, 65%RH for 14 and 28 d	CIEL*a*b*, pH, carbonyl content	Evaluation of seven borane complexes for the stabilization of oxidized paper
Bukovsky 2000 (226)	Natural sunlight for 15, 32, 48, 77, 115, 156 d	FE, B, pH, carbonyl content, CaC	Influence of light on aging of newsprint paper
Bukovsky 2000 (227)	Natural sunlight for up to 3 years	carbonyl content	Natural ageing of paper after exposure to daylight
Bulow et al. 2000 (228)	90ºC, 69%RH for 5, 9, 16, 30 d - 80ºC, 65%RH for 7, 15, 28, 50 d - 70ºC, 61%RH for 12, 32, 61, 110 d in stacks of 50 leaves	CIEL*a*b*, B, Y, moisture content, ZSTS, pH, DP	Migration of volatile compounds through stacked sheets of paper during accelerated ageing.
Carter et al. 2000 (229)	90ºC, 69%RH in stacks of 30 leaves for 7 d	CIEL*a*b*, ZSTS, pH	Migration of volatile compounds through stacked sheets of paper during accelerated ageing
De Feber et al. 2000 (230)	90ºC, 50%RH for 3, 6, 12 d	TS	Effect of the composition of the iron-gall ink on the rate of iron-gall ink corrosion
El-Saied et al. 2000 (231)	100ºC, DO for 3, 6, 12 d	α-cellulose, carbonyl content, DP, B, W, O, FE, TR, BS, TS	Correlation between permanence of paper made from straw pulps and ageing variables
Kolar et al. 2000 (62)	90ºC, 65%RH for 6, 28 d	FTIR (DRIFT), B, DP	Effect of Nd:YAG laser radiation at 1064 nm on paper
Nada et al. 2000 (232)	130 - 150ºC for 1 hour	B, TS, TR, BS	Physicomechanical properties of paper treated with polymers
Adamo et al. 2001 (63)	80ºC, 65%RH for 12 d	TS, TR, FE, SAB, B, CIEL*a*b*, pH, Kappa Number, FTIR	Gamma radiation treatment of paper in different environmental conditions
Bukovsky et al. 2001 (233)	35 d natural daylight	FE, B, carbonyl content, AR, pH	The influence of deacidification with Mg compounds on the light induced oxidation of newsprint
Dufour et al. 2001 (234)	LA, 70ºC for up to and 800 hours	FE, Y, B, FTIR, SEM	Photo-oxidation of mass-deacidified papers
Flieder et al. 2001 (235)	80ºC, 65%RH – LA at 25ºC, 50%RH - SO_2 (10 ppm) and NO_2 (20 ppm) at 30ºC, 50%RH for 2, 3, 6 weeks	TS, pH, W, CN	Properties of new and historic papyrus
Magaudda et al. 2001 (236)	80ºC, 65%RH for 12 and 24 d		Evaluation of gamma radiation for the disinfection/disinfestation of paper
Moropoulou et al. 2001 (143)	105ºC, DO for 3 and 6 d	FE, pH, CIEL*a*b*	Quality control and optimization of large scale conservation treatments
Zappala et al. 2001 (66)	80ºC, 65%RH for 25 d	DP, pH, CIEL*a*b*	Effect of trehalose treatment on paper stability
Calvini et al. 2002b (111)	90ºC, 60%RH in sealed glass tubes	FTIR, DP, pH	The degrading action of iron and copper on paper
Dupont et al. 2002 (237)	50 ppm of NO_2 at 23ºC, 50%RH for 5 d	SEM, EDS, pH, AR, CIEL*a*b*, B	Testing CSC Book Saver, a commercial deacidification spray
Inaba et al. 2002 (238)	80ºC, 65%RH for 16 weeks	pH, TEA, FE, CoC, DP	Effect of cooking agents on the permanence of Japanese paper
Letnar 2002 (239)	80ºC, 65%RH for 24 d	pH, CN, DP	The influence of unbleached pulp content on the permanence and durability of paper
Letnar et al. 2002 (240)	80ºC, 65%RH for 24 d	Density, grammage, kappa number, pH, DP, CN, TR, FE, bending stiffness, internal bond resistance, different surface properties, FTIR, B, Y, M	The effect of accelerated ageing on graphic paperboards degradation
Malesic et al. 2002 (241)	80ºC, 65%RH	pH, DP, carbonyl content	Factors affecting ageing of alkaline paper (effects of overdeacidification)
Rocchetti et al. 2002 (242)	80ºC, 65%RH	CIEL*a*b*	Evaluation of gamma radiation for the disinfection/disinfestation of paper (effect on printing inks)
Adamo et al. 2003	80ºC, 65%RH for 12 d		Evaluation of gamma radiation for the

(243)			disinfection/disinfestation of paper (effect on printed paper)
Adamo et al. 2003 (244)	80ºC, 65%RH for 12 and 24 d		Evaluation of gamma radiation for the disinfection/disinfestation of paper (susceptibility of cellulose to attack by microfungi after treatment)
Basta 2003 (245)	100ºC for 36, 72, 108, 144 hours	TS, TR, B, IR spectra	The role of chitosan in improving the ageing resistance of rosin sized paper
Basta et al. 2003 (168)	100ºC, 93% RH for 3 and 10 d	FTIR, thermogravimetric analysis (TGA), TS, TR, BS, FE, B	Effects of grammage and gelatine additive on the durability of paper
Kobiakova et al. 2003 (246)	105ºC in sealed tubes for 72 hours	B, TS, FE, pH,	The behaviour of paper treated in a carbon dioxide modified atmosphere
Letnar et al. 2003 (247)	80ºC, 65%RH for 24 d	FE, TR, pH, DP, CN, B, Y, O	The permanence and durability of graphic art paper
Moropoulou et al. 2003 (67)		TS, TEA, SAB, FE, FTIR, pH, water vapor sorption	The immediate impact of aqueous treatments on the strength of paper
Sundholm et al. 2003 (30)	40, 60 and 90ºC, 50% RH for up to 36 days	DP, ZSTS, TS, B, AR	Evaluation of aqueous solutions of calcium hydroxide/methyl cellulose for paper conservation
Basta 2004 (248)	100ºC for 144 hours	TS, TR, Y, Thermogravimetric Analysis, FTIR	Performance of improved polyvinyl alcohol as an ageing resistance agent
Capolongo et al. 2004 (249)			Freeze-drying of water-damaged paper material
Letnar et al. 2004 (250)	80ºC, 65%RH for 24 d	Formation index, pH, Kappa number, DP, CN, TR, FE, B, Y, O	Parameters that affect leafcasting and optimization of the technique
Daniels et al. 2004 (251)		Uptake of water, reflectance	Study on the washing of paper
Kolar et al. 2004 (252)	70ºC, 50%RH for 3 d	DP, BS, B	Evaluation of the effects of treatment on iron gall ink corroded documents
Kolbe 2004 (253)	70ºC, 50%RH for 3 d - 90ºC, 35 to 80%RH (cycling) for 3 hourly cycles	BS	Gelatine as an inhibiting agent for iron-gall ink corrosion
Rousset et al. 2004 (254)	80ºC for 1, 4, 8, 12, 14, 15 d	A, pH	Research in mass deacidification agents
Sundholm et al. 2004 (255)	40, 60 and 90ºC, 50% RH for 2, 4, 12, 36 and 48 days	DP, ZSTS, TS, B, AR	Evaluation of aqueous solutions of calcium hydroxide/methyl cellulose for paper conservation

Table 1: The most important references of the last 30 years concerning the evaluation of conservation treatments and other relevant topics.

Method	Measured Property - Scope	Standard*	References♦
Accelerated Aging		ISO 5630 – 1, 2, 3, 4 TAPPI 453, 544, 260 ASTM D 776 ASTM D6819-02e2 CSN 50 0375 UNI 10256	75, 256, 72, 17, 18, 257, 135, 19, 20, 7, 136, 137, 186, 38, 39, 187, 138, 258, 6, 32, 8, 259, 188, 22, 21, 74, 139, 140, 184, 33, 189, 175, 10, 260, 47, 176, 34, 190, 122, 117, 11, 261, 262, 192, 193, 194, 28, 48, 49, 2, 35, 3, 50, 263, 264, 198, 73, 12, 133, 51, 37, 201, 13, 15, 202, 36, 204, 205, 206, 14, 265, 86, 23, 266, 207, 91, 92, 53, 209, 211, 212, 80, 267, 214, 215, 216, 55, 268, 56, 87, 218, 220, 58, 222, 109, 223, 106, 224, 60, 59, 225, 269, 61, 226, 227, 228, 229, 230, 270, 231, 94, 62, 232, 29, 63, 234, 235, 185, 236, 143, 66, 4, 271, 25, 111, 112, 237, 272, 238, 95, 239, 240, 241, 242, 243, 244, 245, 168, 246, 247, 30, 273, 248, 274, 253, 250, 252, 254, 255
Folding Endurance	Folding Endurance (Strength)	ISO 5626 TAPPI 423,511 AFNOR Q03-001 APPITA 423 ASTM 2176 BS 4419 CPPA D. 17P CSN 50 0345 DIN VZ, PCIV/12 GOST 13525.2	75, 72, 68, 17, 18, 135, 20, 19, 5, 7, 136, 137, 186, 187, 285, 188, 22, 21, 74, 139, 140, 184, 189, 260, 176, 262, 192, 193, 194, 71, 44, 48, 2, 3, 195, 50, 264, 198, 73, 199, 51, 203, 204, 205, 23, 266, 207, 80, 15, 275, 276, 215, 216, 56, 87, 218, 219, 220, 81, 222, 223, 225, 226, 231, 63, 233, 234, 143, 238, 240, 168, 246, 247, 67, 250
Tensile Properties	Tensile Strength (TS), Stretch at Break (SAB), Tensile Energy Absorption (TEA)	ISO 1924-1, -2 TAPPI 404, 494 AFNOR 003-001 APPITA P425 ASTM D 828 BS 4415 CPPA D-6 DIN 63112 GOST 13525.1 SCAN P 16 UNI 6438	75, 68, 17, 18, 257, 19, 5, 136, 137, 258, 8, 184, 175, 47, 190, 117, 192, 193, 194, 44, 48, 49, 2, 3, 195, 50, 264, 198, 73, 199, 51, 13, 203, 204, 265, 266, 207, 210, 160, 80, 276, 215, 216, 55, 268, 56, 218, 219, 220, 81, 106, 224, 225, 229, 230, 231, 232, 63, 235, 4, 238, 95, 245, 168, 67, 248, 255
Zero span tensile strength	Fibre Strength	TAPPI 231	258, 32, 117, 2, 264, 13, 205, 276, 268, 218, 106, 228, 229, 95, 30, 255
Tearing Resistance	Tearing Resistance (Strength)	ISO 1974 TAPPI 414 APPITA P 400 CPPA D. 9 ASTM D 689 SCAN-P 11:73 UNI 6444	17, 20, 5, 137, 258, 184, 175, 47, 82, 2, 3, 198, 204, 205, 23, 207, 276, 215, 216, 56, 218, 220, 81, 224, 223, 231, 232, 63, 4, 240, 245, 168, 247, 248, 250
Bursting Strength	Bursting Strength	TAPPI 403	68, 192, 44, 86, 207, 215, 216, 56, 220, 231, 232, 168, 252, 253
Tensile Post Fold	Tensile strength after one fold		3, 80, 87, 81
Intrinsic Viscosity of Cellulose	Degree of Polymerization, Extent of depolymerization of cellulose	ISO 5351/1 TAPPI 206,230 Afnor NFT 12-005 ASTM 1795 – 96 CPPA G.24 SCAN-C 15-16	88, 5, 93, 10, 122, 89, 261, 192, 194, 50, 264, 198, 133, 51, 37, 13, 205, 206, 265, 52, 15, 91, 92, 209, 134, 215, 216, 217, 56, 87, 218, 220, 16, 221, 60, 228, 270, 231, 62, 179, 64, 65, 185, 66, 4, 271, 111, 112, 238, 239, 240, 241, 277, 247, 30, 273, 274, 250, 252, 255
pH of aqueous extracts	Acidity - Alkalinity	ISO 6588 TAPPI 435,509 AFNOR NFQ 03-005 APPITA-P422 APPITA-P421 ASTM D-542 BS 2924 CPPA G. 25P DIN 53124 GOST 12523 NEN 2151 SCAN P14	75, 135, 20, 136, 137, 186, 187, 258, 8, 21, 139, 184, 189, 260, 47, 176, 122, 117, 262, 191, 192, 193, 194, 97, 48, 49, 2, 3, 196, 50, 197, 264, 198, 73, 199, 133, 51, 200, 13, 202, 203, 204, 205, 206, 86, 266, 91, 92, 208, 209, 210, 160, 211, 15, 275, 80, 276, 215, 216, 55, 268, 217, 56, 87, 218, 220, 222, 223, 225, 61, 227, 228, 229, 270, 63, 179, 233, 235, 185, 143, 66, 111, 239, 240, 237, 238, 241, 278, 277, 247, 67, 279, 250, 254

* ISO: International Organization for Standardization, ASTM: American Society for Testing and Materials, TAPPI: Technical Association of the Pulp and Paper Industry, CPPA: Canadian Pulp and Paper Association, DIN: Deutsche Institut für Normung, BS: British Standards, APPITA: Technical Association of the Australian and New Zealand Pulp and Paper Industry, SCAN: Scandinavian Pulp, Paper and Board Tesing Committee, AFNOR : Association Fransaise De Normalisation, CSN: CzechoSlovak Standard, UNI: Italian Standard Organization, GOST: Russian Federation State Standard.

♦ Bibliographic references concern: a. method description and application for the characterization of paper, b. instances of application of the method for the evaluation of paper conservation interventions, c. instances of application of the method for the evaluation of paper permanence and d. evaluation of the method.

Method	Measured Property - Scope	Standard*	References♦
Alkali Reserve	Alkali Reserve	ISO 10716 TAPPI 428 ASTM D 548	138, 139, 122, 191, 192, 194, 2, 263, 197, 198, 73, 133, 200, 205, 266, 208, 213, 218, 99, 279, 30, 255
Copper Number, i cu	Oxidizable content	TAPPI 430 ASTM D919 CPPA G.22 SCAN C-22	20, 258, 32, 33, 192, 2, 133, 205, 206, 207, 214, 215, 216, 223, 235, 239, 240, 247, 250
Kappa number	Oxidizable content	ISO 302 TAPPI 236 APPITA P 201 CPPA G.18 SCAN C1	215, 63, 240, 250
Fourier Transform Infrared Spectroscopy (FTIR), FTIR Microscopy (µFTIR), IR	Identification of additives and impurities, Chemical changes of cellulose		110, 115, 108, 122, 120, 117, 119, 114, 121, 203, 118, 54, 116, 105, 101, 109, 106, 113, 62, 63, 107, 179, 234, 102, 64, 280, 103, 104, 111, 112, 240, 245, 67, 248, 281
Gel Permeation Chromatography (GPC), Size Exclusion Chromatography (SEC)	Molecular mass distribution of cellulose, Average degree of polymerization		93, 13, 14, 94, 95
Brightness	Brightness	ISO 2470 GOST 7690 TAPPI 217, 452, 525 CPPA E. 1, SCAN P3:75 SCAN C11:75 SCAN G1:75	135, 7, 136, 137, 186, 258, 138, 139, 140, 184, 176, 122, 123, 262, 194, 2, 198, 73, 124, 91, 92, 125, 209, 212, 213, 214, 215, 216, 217, 218, 220, 222, 225, 226, 228, 231, 62, 232, 63, 233, 234, 240, 237, 245, 278, 246, 247, 30, 250, 251, 252, 255
Colorimetry (CIEL*a*b*)	Color Changes	TAPPI 524 ASTM D 2244 - 93	133, 211, 134, 55, 268, 56, 87, 71, 59, 61, 228, 229, 63, 179, 185, 143, 66, 237, 111, 132, 242
Optical Microscopy (OM)	Paper surface observation, Fibre origin and composition	ISO 9184-1 -2, -3, -4, -5, -6, -7 TAPPI 259, 263, 401 ASTM D1030 CPPA B.7 SCAN G-3 SCAN G-4	68 (p. 375-397), 152, 153, 154, 155, 156, 49, 198, 203, 160, 211, 276, 282, 179, 240, 283
Scanning Electron Microscopy - X-Ray Microanalysis (SEM – EDS)	Fibre surface observation, Elemental composition		68, 152, 159, 195, 284, 200, 157, 158, 121, 205, 160, 101, 211, 268, 282, 106, 161, 234, 237, 283

Table 2: Established methods, already in use for paper conservation evaluation

Method	Measured Property - Scope	Standard	References
Ultrasonic Testing	Velocity of ultrasonic waves - Specific modulus		285, 117, 119, 177, 118
Differential Scanning Calorimetry (DSC) - Differential Thermal Analysis (DTA) - Thermogravimetry (TG)	(Thermal) Stability		286, 287, 20, 166, 167, 169, 9, 21, 33, 162, 163, 164, 165, 102, 168, 248
Dynamic Mechanical Analysis (DMA)	Thermomechanical transitions, Brittleness		70, 71, 162,163, 164, 165
Confocal Microscopy	3-D Topography of paper surface and fibres		288, 289
Atomic Force Microscopy	Fibre surface observation and characterization		290
Photon Tunneling Microscopy	3-D Topography of paper surface		291
Carbonyl Content (mainly hydrazine method)	Carbonyl Content		292, 122, 37, 52, 213, 214, 58, 222, 226, 227, 61, 231, 179, 233, 65, 241, 278
Carboxyl Content	Carboxyl content	TAPPI 237 – ASTM 1926 - 89	122, 37, 52, 91, 92, 214, 220, 222, 109, 226
α-, β- and γ-celluloses in paper and pulp	α-, β- and γ-celluloses in paper and pulp	TAPPI 203, 429 - ASTM D-588 - CPPA G.29	293, 20, 32, 33, 47, 264, 198, 13, 207, 220, 231
Atomic Absorption Spectroscopy (AAS)	Identification of inorganic compounds		139, 184, 176, 191, 211, 217
Secondary Ion Mass Spectrometry (SIMS)	Chemical mapping of paper surface (organic and inorganic compounds), Surface observation		294
Raman spectroscopy	Chemical analysis		295
Near-Infrared Spectroscopy (NIR)	Chemical Analysis		296, 107
Electron Spectroscopy for Chemical Analysis (ESCA) - X-Ray Photoelectron Spectroscopy (XPS)	Elemental composition and chemical structure of paper surface		108, 200, 297, 298
Photon induced X-ray Emission (PIXE)	Elemental composition of paper surface		117, 299, 300
Electron Energy Loss Spectroscopy (EELS)	Chemical composition, Elemental electronic structure, Identification of additives		301
X Ray Diffraction (XRD)	Degree of crystallinity, detection of additives		302, 303, 304, 305, 117, 306, 274
Russell Effect	Autooxidation trend		181, 182, 183, 109
Fluorescence	Intensity of fluorescence		178, 118, 105, 179, 180
High- Performance Liquid Chromatography (HPLC)	Determination of paper ageing products		307
Gas chromatography - Mass Spectroscopy (GC-MS)	Identification of ageing products of paper		45, 196, 54, 270, 283
Thin Layer Chromatography, (TLC), Paper Chromatography	Identification of paper ageing products		256, 308, 54
Moisture Content – Water Absorption – Water Vapor Absorption	Moisture content, Indirect determination of cellulose crystallinity, Porosity changes	ISO 5637 - TAPPI 412,432 - APPITA P 401 ASTM D 644 - BS 3433 - CPPA G-3 - SCAN P4, T804 - ASTM D 824 - CPPA F4	309, 310, 311, 312, 304, 313, 258, 16, 106, 228, 67, 251

Table 3: Various methods that have been sparingly used or apply to specific problems of paper conservation evaluation; methods that have never been used but have the potential to evolve.

Method	Standard	References
Conditioning, Preconditioning	ISO 187 - TAPPI 402 - ASTM D685-87	
Grammage Determination	ISO 536 - TAPPI 410	
Sampling	ISO 186 - TAPPI 400 - ASTM D 585	
Thickness	TAPPI 411 - APPITA P426 - BS 3983 - CPPA D 4 - DIN 53105 - SCAN P7	
Iodine spot test (Detection of starch)	TAPPI 419	314 (p. 412), 68 (p. 366), 207, 160, 276
Alizarin-S spot test (Detection of Al^{3+})		276
Phloroglucinol spot test (Detection of lignin)	TAPPI 401	276
Raspail test, etc. (Detection of rosin)	TAPPI 408	314 (p. 405), 68 (p. 365), 207, 276

Table 4: Auxiliary methods concerning sample preparation and detection of additives and lignin

Abstract

In this paper, we present the results of a literature survey concerning the methodology and criteria used for the evaluation of paper conservation interventions. Important issues that are reviewed include:

- Accelerated ageing: theoretical principles, most common methods, standards and conditions (temperature and relative humidity).
- Experimental setup: sample selection and preparation, planning of the experiments.
- Methods for the evaluation of paper properties: established methods already in use, various methods that have been sparingly used and methods that have never been used but have the potential to evolve and apply to specific problems of the evaluation.
- Criteria of effectiveness of the intervention.

A selection of the most important relevant publications of the last 30 years and the methods yielded by the survey are presented in table format.

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