Digestum vetus, parchment manuscript (XIV century), Biblioteca Nazionale Universitaria, Torino

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SUSTAINABLE PRESERVATION OF HISTORICAL PARCHMENTS

The protection of parchment collections in public and private libraries, archives, museums still represents a conservation problem which has not been solved. In the present paper, the state of the art in damage assessment of parchments is given and results of the European research project IDAP "Improved Damage Assessment of Parchments" (2002-2005) and Italian research project OPERA "Old Parchments Evaluating, Restoration and Analysis" (2006-2009) are described. For the first time a comprehensive procedure for grading damage in historical parchments is proposed.

Parchments, what they are?

The almost entire intellectual heritage of the Western World, from classical times to the Renaissance, has been delivered to us on parchment in form of scrolls, manuscripts, codices, book covers, etc. Earliest records of writing on skins date back to *the Fourth Egyptian Dynasty* (2700 B.C.). With the adoption by Assyrian people of Aramaic writing (about 800 B.C.), for which clay tablets were not suitable, the use of parchment spread widely. The ability to write on both sides of parchment and its very lasting structure enabled the successful replacement of papyrus scrolls. As ink and paint had no spill-out effects due to absorption of ink on the parchment surface, illuminated codices became real works of art. Eventually new techniques in paper milling allowed much cheaper and more abundant paper production. The advent of printing led to parchments being progressively replaced by paper. Parchment, unlike leather, is not tanned, except in a few cases, e.g. the Qumran Dead Sea scrolls, and thus it is very reactive with changes in relative humidity. The manufacturing methods, known from antiquity essentially follow the traditional main steps: hide salting, liming, dehairing, fleshing, drying under tension and finishing.

Structure of parchment

As parchment is manufactured from animal hide after strong alkaline removal of the epidermis and subcutaneous tissues, collagen is by far its main component. Collagen is a fibrous protein that amounts to approxi-

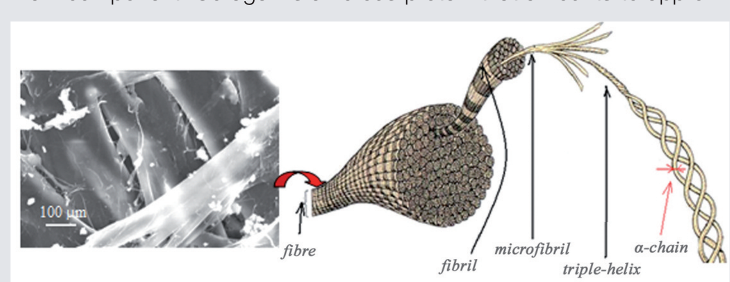


Fig. 1 - Hierachical organisation of collagen in animal hide and parchment

mately one third of proteins in animal and human organisms (94% in tendon, 75% in skin and 50% in bone tissue). The standard model for the structure of collagen is an ascending bundling hierarchy (Fig. 1). Collagen type I molecule from skin is a triple helix about 285 nm long and 1.5 nm in diameter resulting from the right-handed super-coiling around a common axis of three left-handed helices (α-chains) [1]. Lateral and longitudinal packing results in the assembly of almost cylindrical fibrils of 30 to 500 nm in diameter. Fibres with diameters ranging from 50 to 300 μm are then built up through the tight packing of fibrils by means of intermolecular interactions [2, 3]. The mesoscopic arrangement of collagen fibres in skin is a three-dimensional network of thin, wavy and loosely intertwined bundles [4, 5], whereas a parallel alignment of fibres characterises parchment as a consequence of hide processing operations such as liming and drying under tension. The key of success of parchment as biomaterial rests thus on its hierarchical structure characterised by the intimate relationship and connectivity between individual molecules and their organisation in microfibrils, fibrils and fibres.

Parchment deterioration

Deterioration of parchment is a complex process that strongly depends on the environmental conditions (humidity, temperature, light, pollutants), which have changed over the years of storage, as well as on biological agents, extreme events (fire, floods, earthquake, wars), improper handling and conservation/restoration operations. The general deterioration path of parchments develops from an intact fibre structure (collagen) of high hydrothermal and mechanical stability through different stages with decreasing stability until a terminal stage characterised by a considerable disintegrated fibre structure that may transform into a gelatinous substance by contact with water or storage in moist conditions. Much less is known about the physical-chemical and structural alterations that occur in specific conditions (i.e. drying, changes induced by hydrothermal-dehydrothermal treatments, light irradiation, etc.) and under the pollutants attack produced by complex and dynamic interactions with environment. Due to its exceptional longevity, parchment physical features were considered for restoration purposes only , e.g. the pioneering work of the chemist Icilio Guareschi *Della pergamena, con osservazioni ed esperienze sul ricupero e sul restauro di codici danneggiati negli incendi e notizie storiche*, Supplemento annuale all'Enciclopedia di Chimica, vol. XXI, Torino, 1905, which followed the fire ravage of the manuscripts of the National Library of Turin in 1904, and very rarely individual aspects (e.g. microbial damage, humidity/heat damage) were studied. No standard methods for the assessment of deterioration, nor detailed descriptions of the damage type and causes existed up to nowadays to assist both planning and execution of conservation/restoration actions. The general lack of scientific data on deterioration mechanisms and pathways has retarded analysis of historical funds, assessment, and monitoring to develop an effective preservation strategy. As a consequence, the protection of large parchment collections in public and private libraries, archives, museums and religious institutions still represents a conservation problem which has not been solved. Questions still remain: "What are the causes and what are the mechanisms of deterioration in parchment?" The two questions "What is the best way of storing parchment? and "How can we detect deterioration without invasive sampling of documents?" have now become very important.

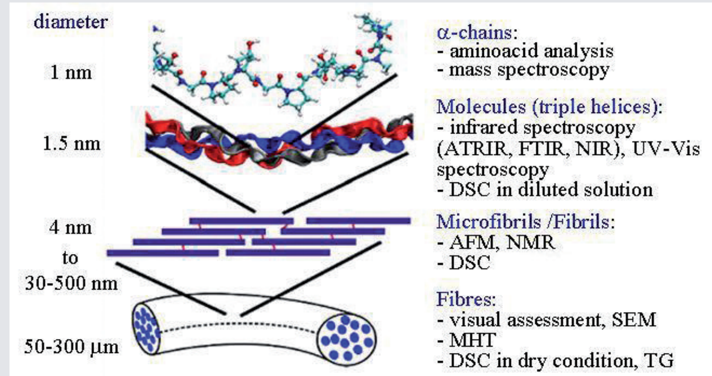


Fig. 2 - Integrated physical-chemical investigations of collagen in parchments from its molecular to macroscopic levels

State of the art of research

For these reasons, the need to establish a joint research project gave rise to a EU Commission project, *Improved Damage Assessment of Parchments* (IDAP, EVK4-CT-2001-00061, 2002-2005). The main objective of IDAP was to deliver standardised methods for damage assessment and damage diagnosis tools for both researchers and conservation professionals. For this purpose specific procedures for accelerated ageing were developed in close cooperation with end-users. An in-depth characterisation of the mechanisms of ageing and deterioration induced by the environment (temperature, relative humidity, light irradiation, chemical pollutants) was achieved by non-invasive, non-destructive or micro-destructive techniques. These target parchment properties at the molecular, nanoscopic, mesoscopic and microscopic level (Fig. 2) and their variations during deterioration. Techniques include ATR/FTIR, GC-MS, HPLC, AFM, micro-thermal analysis, NMR, XRD, TG/DTG, DSC, controlled environment DMTA, thermo microscopy and SEM [6-11]. IDAP project main deliverables were: a programme for the assessment of damage in parchments (PDAP - Parchment Damage Assessment Programme), an early warning and prevention system (EWS - Early Warning System), and a simple, digital atlas on parchment damage assessment (DUPDA Digitised User-Friendly Parchment Damage Atlas). They are now operative within the compass of the non-profit IDAP Network[1] and available on internet at www.idap-parchment.dk (accessed date etc). The results obtained using parchments subjected to accelerated ageing have been validated through the analysis of historical parchments provided by European archives and libraries [12-16]. In this context, the research project *Old Parchments Evaluating, Restoration and Analysis* (OPERA D39 CIPE 04, 2006-2009) funded by Piedmont Region through *Bando CIPE 2004, section Nanotechnology and Nanosciences* has applied and deepened the IDAP achievements by collaborating with Piedmont archives and libraries. OPERA project has also focused on spreading the assessment culture in Piedmont by bridging the gap between researchers and practitioners and improving communication and diffusion of the expertise and knowledge gathered in Europe.

[1]IDAP project and network were identified as a major infrastructure of interest for the humanities in Europe by the European Science Council in 2006.

Damage assessment techniques and strategy

Evaluation of deterioration degree of historical parchments is rather difficult due to both the complexity of the material and heterogeneity of damage related to environmental ageing, conservation history, use and handling of documents. The damage picture of a parchment (sheet/binding/codex) reflects the complexity and dynamics of its deterioration. Generally, deterioration has a progressive character caused by environmental factors such as pollutant gases, temperature and relative humidity.

Traditional visual damage assessment concerns the whole object including ink, dyes and pigments as well as how much a text is readable. The very recent holistic approach of cultural heritage study has promoted the awareness that single documents are not only witnesses of intellectual history, but of materials and techniques as well. This new concept has produced a deep attention towards aspects which were considered as merely accidental, such as materials study. Both IDAP and OPERA projects focused on parchment structure assessment and the collaboration between different fields, namely chemistry, physics, biology, conservation science and collection management.

As deterioration proceeds from the parchment surface to inner layers and from macroscopic to molecular levels, simple visual observation is unable to provide information on the impairment of hierarchical organisation of collagen and hence its stability. On the other hand, it should be noticed that changes in materials characteristics do not necessarily reflect the evolution in the damage of parchment structure. An effective analysis of deterioration requires further experimentation which may be non-invasive or invasive. Usually non-invasive experiments are observational experiments, e.g. establishing the colour of a parchment by comparison with a colour chart. Most non-invasive experiments are only descriptive, offering no explanation of the types of deterioration that take place. Invasive experiments, however, give rise to ethical considerations between on the one hand preserving full integrity of the parchment and on the other hand enabling characterisation and grading of deterioration, essential for understanding how parchment finds should be better preserved. To solve this dilemma, IDAP and OPERA proposed a systematic, multidisciplinary investigation through non-invasive, non-destructive, micro-invasive or micro-destructive techniques based on the correlation between changes/alteration of physical-chemical and structural properties of parchments at macro and microscopic levels (visual assessment) and mesoscopic, nanoscopic and molecular ones (advanced physical-chemical investigation).

Parchment intrinsic measurable properties for grading damage and qualitative markers for characterising individual deterioration pathways were identified and damage assessment protocols were drafted for each type of investigation technique. A valuable tool called *The Digitised User-Friendly Parchment Damage Atlas (DUPDA)* was thus built up to group parchments in four categories of damage by cross-correlation of data detected at the different structural levels of parchment: (i) not damaged parchments; (ii) parchments displaying minor damage; (iii) parchments displaying medium damage and (iv) parchments displaying significant damage.

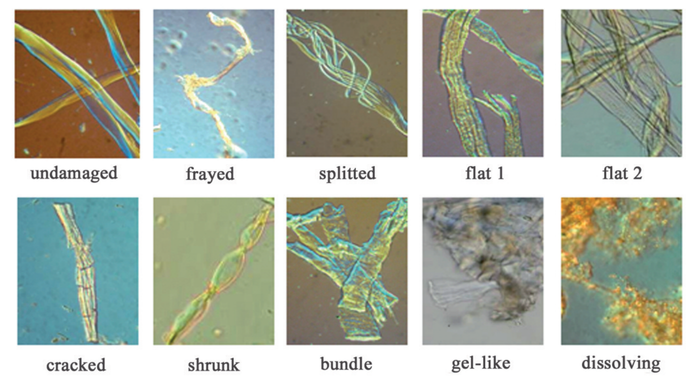


Fig. 3 - Microscopic characteristics used to describe parchment fibres in aqueous milieu and their damage

IDAP visual damage assessment protocol

IDAP protocol is based on a general evaluation of the appearance and condition of both sides of the whole parchment followed by a more specific evaluation of both sides of selected specific areas. At the macroscopic level, changes of features like colour, stiffness, thickness, flexibility, transmission of light may reflect damage and can be determined using simple methods. At the microscopic level, damage in fibre structure can be detected and measured by light microscopy [10, p.17-21], whereas hydrothermal stability is determined by micro hot table method (MHT), a thermal microscopy technique [17]. For fibres evaluation nine major features were identified by microscopic observation of fibres in water as typical and representative for damage characterisation (Fig. 3). In most cases one or more of these features are present to various degrees in the same fibre sample, and often on single fibres, in the event of complex deterioration. Normally, damage evolves during a flattening, splitting and/or fraying of the fibres and leads to a fragmentation ending with a gel-like substance that may melt by contact with water as the final state of deterioration. The calculation of the amount of damaged fibres as the fraction of damaged fibres of the total number of fibres observed is used to assign the parchment to one of the following damage categories: (i) none or little damage: ≤30% damaged fibres; (ii) minor damage: >30%-≤50% damaged fibres; (iii) medium damage: >50%-≤75% damaged fibres and (iv) heavy damage: >75% damaged fibres. However, deterioration cannot be detected only by visual and microscopic assessment. The lack of advanced investigations may lead to further damage of parchments caused by inappropriate treatment or storage. Advanced analysis and experiments, as well as correlation of data provided by simple visual and advanced investigations add knowledge to the deterioration characteristics, evolution patterns and causes.

Advanced physical-chemical investigation

In depth characterisation of damage was achieved by advanced physical-chemical techniques targeting its molecular (UV-Vis, ATR/FTIR, FTIR, GC, HPLC), nanoscopic (NMR, AFM, micro-thermal analysis XRD) and mesoscopic (TG/DTG, DSC, DMTA, SEM) properties and their alteration during deterioration. Changes revealed by these techniques provided quantitative indicators for damage grading and prediction the stability of parchment during storage and conservation [18, 19]. A path for analyses of increas-

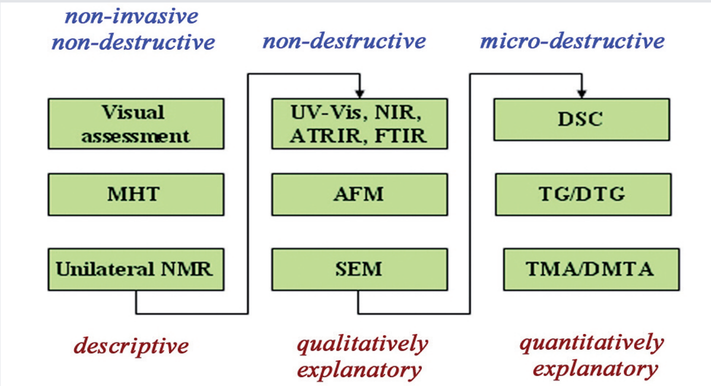


Fig. 4 - Increasing invasiveness of the techniques for the damage assessment of parchment

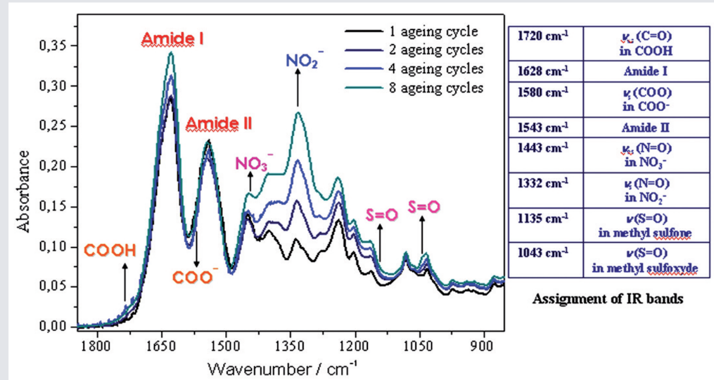


Fig. 6 - Overlay of ATRIR spectra of parchments exposed to NO_x and SO_2 (50 ppm in air, at 25 °C and 50% RH) for increasing number of ageing cycles (one cycle 2 weeks)

ing invasiveness was set up to optimise the ratio between the invasiveness of investigation and quantity and quality of information obtained (Fig. 4).

Both IDAP and OPERA projects also developed simple non-invasive or micro-invasive experiments that can be carried out *in situ*, using portable equipment (e.g. portable Raman spectrophotometer), portable NMR Profiler), or in conservation laboratories in archives and libraries (e.g. SEM, MHT). Advanced analyses provided descriptive (qualitative markers), explanatory (deterioration pathway markers) and quantitative (measurable parameters) information on deterioration occurring at various structural levels of parchment. Some results obtained using SEM, ATR/FTIR and DSC are reported below.

Scanning electron microscopy (SEM) is a micro-invasive, non-destructive high-resolution imaging technique which allows a mesoscale morphological examination and reveals the general features of the surface of samples, shape of the fibre bundles and structure of individual fibres/fibrils. These morphologic qualitative markers were used to evaluate the impact of environmental factors on parchment during accelerated ageing experiments. Based on the large collection of images obtained, two main criteria for damage assessment in historical parchments were identified: (i) persistence of the fibre network and (ii) glossy and/or granular surface occurrence. As parchments surface morphology is highly non-uniform, four deterioration markers were considered for each criteria and scores

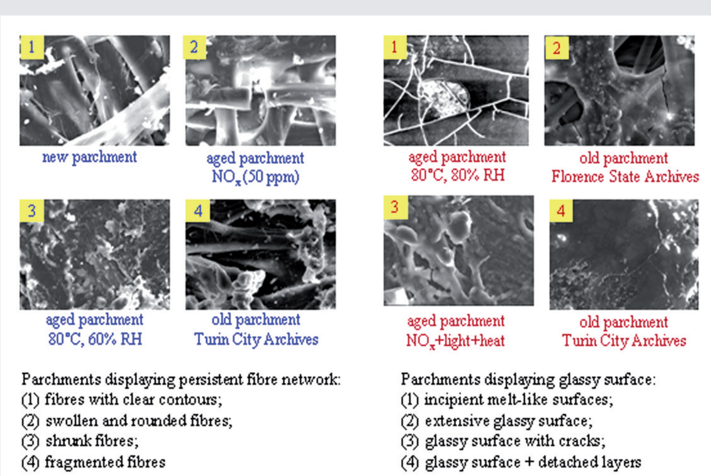


Fig. 5 - Evaluation of parchments morphological features by SEM

from one to four, corresponding to the above mentioned four levels of damage, were assigned to each marker as follows: fibres with clear contours (score 1); swollen and rounded fibres (score 2); shrunk fibres with incipient globular aspect (score 3); fragmented fibres (score 4), incipient melt-like surfaces (score 1); extensive glossy surface (score 2); glossy surface with cracks (score 3); glossy surface with detached layers (score 4) [9, 20]. It becomes thus possible to grade historical parchments deterioration using the average score calculated as the arithmetic mean of the scores assigned through the examination of their morphologic markers (Fig. 5) as well as to monitor the impact of environment [11, 21].

Attenuated total reflectance infrared spectroscopy (ATR/FTIR) is a non-destructive technique which provides the parchment chemical structure and its chemical changes caused by deterioration factors. In the mid-infrared, absorbance of radiation is related to fundamental vibrations of the chemical bonds and indicates the presence or absence of specific functional groups. ATR/FTIR analysis specifically indicates deterioration processes such as collagen triple helix conversion to disordered structures (shift of amide II band to lower values), changes in the shape of the amide I peak (1630:1660 ratio as described by Odlyha *et al.*), oxidation of the polypeptide chain (formation of new COO^- species), hydrolytic cleavage (increase in -OH stretching or bending frequencies at 3400 and 1650 cm^{-1}, respectively) [10, 14, 16]. The effects of chemical pollutants such as NO_x and SO_2 can be revealed by the presence of species like NO_2^- (1328, 1250 and 824 cm^{-1}), NO_3^- (1406 and 1038 cm^{-1}), and of methionine sulphone (1150-1000 cm^{-1}), respectively (Fig. 6) [8]. ATR/FTIR is thus an important tool for identification of deterioration pathways and monitoring the environmental impact.

A method which offers an integral response relevant to the whole sample mass and provides both explanatory and quantitative results using micro-samples (1-4 mg) is differential scanning calorimetry (DSC). Moreover, DSC results can be successfully combined with the results from non-invasive techniques, i.e. unilateral NMR (nuclear magnetic resonance), minimally invasive techniques, i.e. micro hot table (MHT) as well as infrared (FTIR) and ultraviolet-visible (UV-Vis) spectroscopy, and partially invasive techniques, i.e. ESEM (environmental scanning electron microscopy) and AFM (atomic force microscopy) [10, 16, 20-22]. DSC measurements in both dry and excess water conditions give insight into collagen changes

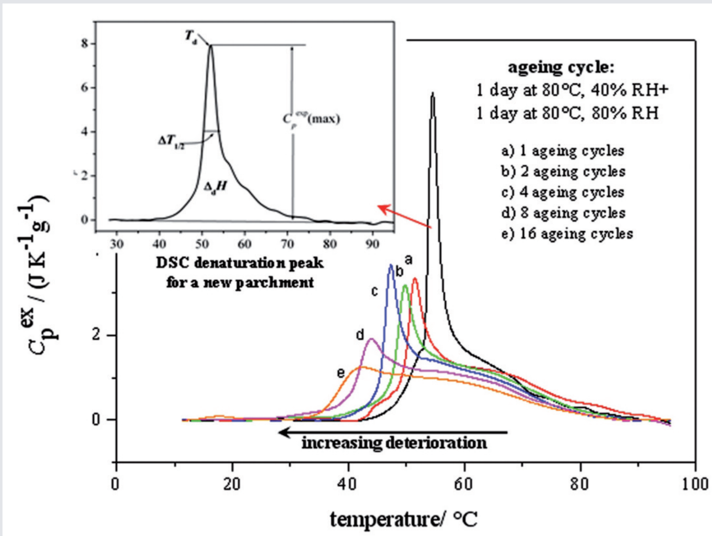


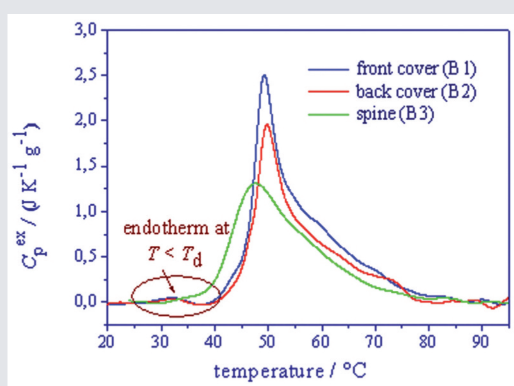
Fig. 7 - DSC in excess water curves for thermal denaturation of parchments subjected to accelerated ageing

induced by ageing and deterioration through the determination of the energy needed to collapse and break-down its structure. Collagen denaturation is thermodynamically significant: (i) at mesoscopic level, when assembly of fibrils is weakened and finally lost, (ii) at molecular level, when triple helix uncoiling and peptide chain cleavage occur. The analysis of DSC denaturation peak provides quantitative parameters such as T_d (denaturation temperature), $\Delta_d H$ (associated enthalpy change), $\Delta T_{1/2}$ (peak half-width) and C_p^{ex}(max) (peak height) (Fig. 7). Deterioration level of parchment is evaluated from the departure of denaturation parameters from the reference values (i.e. those determined for new undamaged parchments). Generally, the advance of deterioration during accelerated ageing experiments is characterised by progressive shift of T_d to lower temperatures, DSC peak shape broadening and lowering, giving rise to an increase of $\Delta T_{1/2}$ and decrease of both C_p^{ex}(max) and $\Delta_d H$ (Fig. 8). It should be noted that lower $\Delta_d H$ values of old parchments do not always correspond to lower T_d values due to the complex dynamics of ageing and deterioration. In fact, an earlier crosslink formation may stabilise parchment structure prior to deterioration through polypeptide chain cleavage.

DSC damage evaluation is thus based on three parameters: T_d, $\Delta_d H$ and peak shape index expressed as peak half-width and peak maximum height ratio $Is = \Delta T_{1/2}/C_p^{ex}$ (max). Scores from 1 to 4 were assigned to each parameter based on the findings for parchments exposed to accelerated ageing [10]. For T_d scores are: 1 for 50 °C<T_d<55 °C; 2 for 45 °C<T_d< 50 °C and T_d>55 °C; 3 for 40 °C<T_d< 45 °C and 4 for T_d<40 °C. For $\Delta_d H$ scores are based on its % variation from the reference: 1, less than 10%; 2, 10-20%; 3, 20-35% and 4, >35%. For *Is* scores are: 1 for *Is*<1; 2 for 1<*Is*< 5; 3 for 5<*Is*<15 and 4 for *Is*>15. The averaged score calculated by giving weight factors to the three DSC parameters (S = 0.2 T_d score + 0.5 $\Delta_d H$ score + 0.3 *Is* score) allow us to classify a historical parchment in one of the four damage categories earlier defined (Fig. 8). Moreover, by analysing DSC denaturation curves of accelerated aged parchments it was demonstrated that RH combined with a relatively high temperature (Fig. 7) results in a progressive broadening and lowering in peak intensity and a shift to lower temperatures [6, 10] (Fig. 7). On the other hand, the presence of acid species, produced by exposure to atmospheric pollutants (NO_x and SO_2) promotes hydrolysis of the stable fraction which gives an additional shoulder peak [10, 16]. The occurrence of small and broad endotherms in the range 20 to 40 °C (Fig. 8) was related to collagen gelatinisation and the less sharp onset of the DSC peak was assigned to the transition of a disordered fraction of collagen [6, 11, 16].

Evaluation of damage for a series of archival bookbindings from the Historical Archives of Turin

Several archival bookbindings from XIV and XV centuries belonging to the Historical Archives of Turin were investigated by light microscopy, scanning electron microscopy (SEM), differential scanning calorimetry (DSC), infrared spectroscopy (FTIR) and unilateral nuclear magnetic resonance (NMR). No restoration or conservation treatments are known to have been applied to any of the selected bindings. Investigation was made on samples from the external covers, directly exposed to the environmental factors, and internal flaps which are less exposed to environment. Two or three sample areas were chosen for the assessment of each bookbinding (e.g. outer covers, internal flaps and spine). Damage scores calculated using SEM and DSC data are summarised in Fig. 9. Ranking of parchments in the four classes of damage is based on the average SEM and DSC scores: no damage for 1<*S*<1.5; minor damage for 1.5<*S*<2.5; moderate damage for 2.5<*S*<3.5 and major damage for *S*>3.5. Flaps were classified as slightly to moderately damaged, whereas covers and spines were moderately to heavily damaged. It must be noted that SEM observation solely concerns sample surface and its fibres (microscopic level) and fibrils (mesoscopic level) features, whereas DSC provides bulk characterisation of samples at both mesoscopic and molecular levels. In addition, pathway markers provided by the UV-Vis, unilateral NMR, DSC peak shape analyses and data correlation pointed to deduce a strong



Sample	T_d / °C	$\Delta_d H$ / (J·g^{-1})	I_S
new	52.9 ± 0.7	54.8 ± 1.4	0.8 ± 0.1
B1	49.4	28.7	2.5
B2	49.1	26.6	3.7
B3	48.3	24.5	8.2

Sample	Damage score	Damage level
new	0	undamaged
B1	3.2	medium
B2	3.5	medium-to-heavy
B3	4.0	heavy

Fig. 8 - DSC damage grading for three samples from both covers and spine of an old binding from the State Archives of Turin

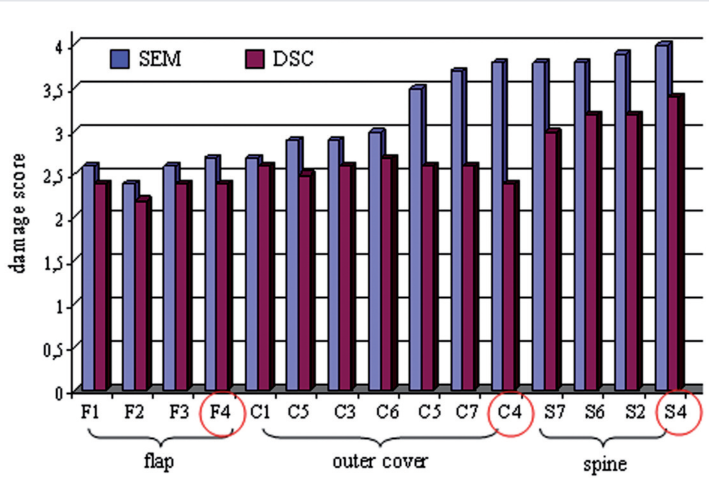


Fig. 9 - Comparative damage ranking of a group of archival bindings by DSC and SEM. Sample 4 comprehensively displays different damage level of inner flap (F4), outer cover (C4) and spine (S4)

loss and randomisation of the molecular helical structure for outer covers, a higher crosslink formation for flaps and a significant oxidation and gelatinisation evidence for spine/edges [16]. These findings allowed us to understand how the accessibility of a parchment may determine various ageing pathways under the same storage conditions.

Remarks

Combined visual and advanced assessment of parchment structure provide a picture of deterioration from macrosopic to molecular level as well as grading procedures for the evaluation of deterioration degree. The techniques and protocols developed in IDAP and adjusted and optimised in OPERA project are now used in research projects like Domesday Book (National Archives, UK), Codex Sinaiticus (UK, Germany, Russia, Egypt, USA), EtnoPel (National Research & Development Institute for Textile and Leather-ICPI Division, Romania), "Systematic description of the deterioration of leather and parchment fibers at microscopic level in order to improve the diagnosis of deterioration damages" (School of Conservation, Denmark). They have been adopted for improving the diagnosis of deterioration damages in many archives, libraries and museums throughout Italy and Europe[2]. It comes out that the transfer of knowledge gathered within IDAP and OPERA projects to conservation practitioners help to understand how a parchment can be better preserved and design effective methods of conservation/restoration to prolong its lifetime. Their proper employment to the assessment and monitoring of the most valuable documents, restored parchments and those running the risk of irreversible alterations could significantly improve strategies for the sustainable preservation of parchment funds.

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[2]Royal Library, Copenhagen (DK); National Archives, Stockholm (S); National Archives, Kew (UK); Bucovina Museum Complex, Suceava (RO); National Library, Prague (CZ); Muséum National d'Histoire Naturelle, Paris (F); British Library, London (UK); Arnamagnæan Institute, Copenhagen (DK); National Library, Oslo (N); State Archives of Turin (I); Historical Archives of the City of Turin (I); Historical Archives of the University of Turin (I).

RIASSUNTO

La conservazione sostenibile delle pergamene storiche

La protezione delle collezioni di pergamene nelle biblioteche, negli archivi pubblici e privati e nei musei rappresenta un problema di conservazione ancora irrisolto. Nel presente lavoro viene illustrato lo stato dell'arte nella determinazione del danno nelle pergamene e sono descritti i risultati del progetto di ricerca europeo IDAP "Improved Damage Assessment of parchments" (2002-2005) e del progetto di ricerca italiano OPERA "Old Parchments Evaluating, Restoration and Analysis" (2006-2009). Per la prima volta viene proposta una procedura generale per la classificazione del danno nelle pergamene di interesse storico.