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Paper Conservation Methods: A Literature Review.

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Abstract

The main paper conservation methods are presented, classified in the following categories: preparation of the intervention, disinfestation and disinfection/sterilization, surface/dry cleaning, wet cleaning, chemical stabilization, paper repairs, consolidation and strengthening. Treatment documentation is also discussed. The targets, the historical aspects, the general principles, the materials and equipment, the acceptance and criticism pertaining to each method are briefly reviewed, and the most important research for their evaluation is presented. Several paper stabilization strategies, such as deacidification and iron gall ink stabilization, applicable to paper are elucidated. Specific consolidation and strengthening methods for paper, such as lamination and paper splitting are also discussed. The review mainly focuses on the established methods, but experimental, abandoned or insufficiently documented methods are also included. Shortcomings and limitations of several methods were found in the literature, concerning health issues, limited effectiveness, adverse side-effects on the treated artefacts and restricted applicability.

Keywords: paper conservation, disinfestation, chemical stabilization, deacidification, paper repairs, consolidation

1. Introduction

Paper is the most common substrate for recording information, and if manufactured properly, can be very resistant to ageing (Cernic Letnar and Vodopivec 1997a; Zervos 2010). Due to inherent instability, some paper grades (mainly acidic paper manufactured between the middle of the 19th and the end of the 20th centuries) deteriorate fast, thus jeopardizing their potentially valuable information content (Lee et al 2010, Zyska 1996). A large number of documents, manuscripts, books and artistic works on paper are in danger of imminent destruction, and more are becoming fragile and unusable as time advances (Buchanan 1987; Sobucki and Drewniewska-Idziak 2003; Zyska 1996). The rate of deterioration depends on internal factors, such as the raw materials, the production process, the pH etc. and is controlled by external factors such as the climatic conditions, the pollution levels and the biological activity (Area and Cheradame 2011; Arney and Chapdelaine 1981; Bigourdan and Reilly 2002; Fellers et al. 1989; Graminski et al. 1979; Gurnagul et al. 1993; Johansson and Lennholm 2000; Wilson 1995; Wilson and Parks 1979; Zervos 2010).

The target of conservation, according to the International Council of Museums, Committee for Conservation (2008), is to slow down the deterioration and extend the useful life of the artefact. There are several strategies available for paper conservation, targeting various aspects of deterioration, which are further specialized, depending on the targets and limitations of the specific implementation.

Few classifications of paper conservation activities can be found in the relevant literature, most of them in online -yet credible- resources (American Institute for Conservation - Book and Paper 2013; Glaser 1999; Nordstrand 1987; Ogden 1999; Verheyen 1991). For the purposes of this work, paper conservation activities are classified under the following categories, which may also be considered to roughly represent the generic steps of a paper conservation treatment:

1. Preparation of the intervention
    - Materials and decay characterization
    - Treatment planning and documentation
    - Isolation of the artefact, separation from other materials
2. Disinfestation and disinfection/sterilization
3. Surface/dry cleaning
    - Mechanical cleaning
    - Laser cleaning
4. Wet cleaning
    - Washing in water/organic solvents
    - Enzymes treatment
    - Bleaching
5. Chemical stabilization
    - Deacidification (aqueous, in organic solvents, gaseous, mass deacidification)
    - Other chemical stabilization methods (reduction with borohydride, transition metal deactivation, iron gall ink stabilization)

6. Paper repairs (lacunae filling, tears stabilization, filling missing material, vacuum table, leaf-casting)
7. Consolidation/strengthening (lamination, impregnation, paper splitting, mass strengthening methods
8. Treatment documentation, which runs parallel to all previous steps

In the discussion that follows, the categorization of the paper conservation methods presented above will be followed. We will present the main targets of every intervention category and the associated methods, including historical aspects, general principles, materials and equipment, acceptance and criticism. Given the extent and the complexity of the subject, the emphasis will be on the most important publications of the last 35 years. The specifics of the conservation of works of art on paper will not be discussed in this review, and the interested reader is referred to the relevant literature [for example, Ellis (2014)].

2. Preparation of the intervention

The preparation of the intervention comprises several important steps, including materials and decay characterization, documentation of the current condition of the artefact and treatment planning. If the artefact is part of a more complex system, it also includes its isolation and separation from other materials.

According to the American Institute for Conservation (1994), before any action is taken, a thorough examination of the artefact to be treated is paramount. It may entail a simple visual examination, but also the use of sophisticated scientific techniques such as one or more NDTs [NonDestructive Testing techniques, for example see Hahn (2010)]. Scientific examination, apart from the diagnosis of the decay and the material characterization, can also answer questions concerning origin, dating or attribution of cultural objects, which can help the conservator make educated decisions about their conservation. Some of the usual issues that may call for investigation are the composition of inks, dyes and pigments (Hahn 2010), the origin and the tanning system of leather (Nikolova and Velcheva 1996), the composition, origin and processing of paper. The results of the investigation are used for the selection of compatible materials and safe and suitable conservation treatments (Nikolova and Velcheva 1996; Verheyen 1991).

Unfortunately, the average paper conservator rarely has access to advanced scientific techniques when performing routine conservation. The most usual tests performed routinely apart from the visual examinations are the pH testing (non destructively, with contact electrode or pH indicator strips) (Tse 2007) and the testing of the stability of inks, dyes and pigments when immersed in water or other solvents (Verheyen 1991). More sophisticated examination techniques may be used, such as FTIR (Fourier Transform Infrared Spectroscopy), XRF (X-Ray Fluorescence spectrometry), SEM (Scanning Electron Microscopy), image analysis, AFM (Atomic Force Microscopy), fluorescence under UV illumination and many more, but these methods are not normally available to conservators (Banik and Ponahlo 1983; Barrett et al. 1996; Bicchieri et al. 2001; Buzio et al. 2004; Calvini and Gorassini 2002a, b; Calvini and Silveira 2008; Choisy et al. 1997; De Silveira et al. 1995; Havermans et al. 2003b; Sistach et al. 1998; Vives et al. 2001; Vives et al. 2004; Wallbacks et al. 1991; Zervos and Moropoulou 2006). NDT methods are preferred to those that need sampling.

The integrity of the artefact is then verified; for example, if it is a book, it is examined for missing leaves, endleaves, maps, engravings, illustrations etc.

The Guideline for practice of the American Institute for Conservation (1994) states that the conservator is obliged to keep permanent records of the examination and conservation of the treated artefacts. The documentation is achieved by photographic techniques, by descriptive text and sketches that depict all the necessary details and may take the form of laboratory notes, annotated photographs, checklist, work log etc.

If the artefact is part of a more complex system, it may need to be isolated before it is treated. Books with much deteriorated bindings and stained acidic paper may need to be disbound, so that the paper leaves are washed and deacidified. Backings may need to be removed from graphics and photographs, either scraped off dry, or better yet by wetting the material to be removed with thick paste or poultices. This must be done with great care so that the artefact is not damaged. The use of carbopol poultices for that purpose is discussed by Bluher et al. (1995). Enzymes may also be used to dissolve the old paste and facilitate the easy removal of backings. (They will be discussed later on.)

In the rare cases that the accompanying material is in good condition, does not endanger the artefact and does not obstruct the following treatment steps, the intervention may be applied to the composite object. If the conservator must remove accompanying components of the artefact, they must be meticulously documented, and if they constitute a functional part of the object or contribute to its intrinsic value, they must be treated separately, reinforced, stabilized chemically and finally embedded to it at the final stage of the treatment (for example leather covers, decorative papers etc.).

3. Disinfestation and disinfection/sterilization

Disinfestation and disinfection/sterilization aim at protecting the artefact from biological agents. In simple words and in the conservation context, disinfestation entails the extermination of rodents and insects, while disinfection/sterilization the elimination of microorganisms, mainly of fungi. In the past, both processes used toxic chemicals, while nowadays, especially for disinfestation, more benign methods are preferred (Smith 1986).

The properties, usage and effects on human health of the various chemicals, as well as the principles and practice of disinfection and sterilization in general are discussed at length by Fraise et al. (2012). Critical and comparative reviews on the various paper disinfestation/disinfection methods have been published by Craig (1986), Smith (1986), Valentin (1986), Nitterus (2000b), Sequeira et al. (2012) and Sequeira et al. (2014). Brokerhof (1989) and Nitterus (2000b) discuss methods and chemicals for coping with the problem of fungal infestations in libraries and archives. The antifungals in paper conservation are also discussed in an extensive review by Sequeira et al. (2012). It must be stressed here that climatic control is the only long term reliable strategy for dealing with the fungi issue that is safe for both humans and objects (Nitterus 2000b; Sequeira et al. 2014; Smith 1986). The possibility of disinfection should be considered in case of salvaging flooded books or archival materials, if flood water is contaminated or if the material gets moldy before freezing (Fischer 1977; Silverman et al. 2008).

In the rest of this chapter, the most important methods and chemicals for disinfestation and disinfection will be presented. For several of them, the authors of the relevant papers do not discuss health risks for personnel and users, and/or possible negative effects on the treated artefacts. Except from the use of modified atmospheres, the rest of the methods have serious drawbacks concerning health

issues, limited effectiveness, or negative effects on the treated materials. Therefore, the authors of the present review cannot recommend any of them as both effective and totally safe.

Thymol: Thymol (2-Isopropyl-5-methylphenol) has been used as a fungicide in paper conservation for over 60 years. It is easily absorbed by the human organism through breathing or skin contact and is considered to be of medium toxicity. Its use in paper conservation is described by Johnson (1988). It can be applied to paper either as an alcoholic solution by spraying, or more often as a vapour produced by sublimation in exposure chambers (fumigation). Several studies suggested that thymol is a strong bactericide and/or fungicide. Nevertheless, Craig (1986) and Gustafson et al. (1990) report that paper fumigation with thymol is ineffective for combating paper fungi infection. In addition, Daniels & Boyd (1986) showed that it is absorbed by paper and photoxidized, causing intense paper yellowing. Degradation of the paper support, watercolour binder, and iron gall ink were observed by Isbell (1997) after thymol fumigation. In addition, the high temperature and the long exposure (73°C and 72 hours) of paper in the fumigation chambers reported by Craig (1986) is equivalent to conditions sometimes used in accelerated ageing. Overall and according to the cited literature, thymol should be avoided, because it poses serious risks for the treated objects and does not offer proved antifungal protection.

Quaternary Ammonium Salts (quats): Quaternary ammonium salts comprise a large group of active compounds widely used as disinfectants. Strzelczyk & Rozanski (1986) studied the effects of disinfection with quats solutions on paper. They found that they are effective against fungi in ~1% aqueous solutions and that their surfactant properties facilitate paper cleaning. After quats treatment, thorough washing is recommended (3 water baths at 50°C). An accelerated ageing study indicated that their residue (around 400 ppm) does not affect the ageing rate of paper. A newer study verified their effectiveness and demonstrated that they don't have any important side effects on paper stability (Karbowska-Berent et al. 2011). No data on their toxicity are presented in this study, but their health effects are not to be underestimated since they are toxic and may have a negative effect on the human immune system [http://www.inchem.org/documents/pims/chemical/pimg022.htm, Sequeira et al. (2012)].

Ethylene oxide (Ballard and Baer 1986; Brokerhof 1989; Craig 1986; Fischer 1977; Hengemihle et al. 1995; Hofenk de Graaff and Roelofs 1994; Nitterus 2000b; Residori and Ronci 1986; Sequeira et al. 2012; Silverman et al. 2008; Smith 1986; Valentin 1986): Ethylene oxide is very effective and has been widely used in the past for the mass sterilization and disinfestation of books and archival material, but it is flammable, mutagenic and carcinogenic. Its use has been banned in several countries, and wherever it is allowed, it can be used under severe restrictions and multiple safety measures (Sequeira et al. 2012). Paper treated with ethylene oxide is more susceptible to microbial attack (Valentin 1986).

Formaldehyde: Formaldehyde has been used as a gaseous fumigant in mixture with water vapour. Formaldehyde has strong microbicidal properties but low power of penetration and a limited fungicidal effect when used for library material fumigation (Valentín Rodrigo and García Ortega 1999). It was used for the in-situ disinfection of the building and the material (8.1 million volumes) of the Russian Academy of Sciences Library. The repositories were flushed with formaldehyde ($40 g/m^3$) and then neutralized with ammonia gas (Nyuksha and Leonov 1997). Formaldehyde is carcinogenic and toxic to humans (Sequeira et al. 2012).

Ethanol: Ethanol was tested as a fungal sanitizer in paper conservation by Nitterus (2000a), Adelantado et al. (2005) and Bacílková (2006). It is a fungistatic and can inhibit fungal regrowth for 14 days (Bacílková 2006), but was proven to have no sporicidal properties (Nitterus 2000a). The application of a 70% ethanol solution is one of the most preferred options in order to arrest an active fungal growth (Adelantado et al. 2005; Sequeira et al. 2014).
Calcium propionate (Neves et al. 2009; Sequeira et al. 2012; Zappala 1997): A saturated solution (3.5 g/L) of calcium propionate in ethanol was found to significantly inhibit the fungal growth (Zotti et al. 2007).
Parabens (Gustafson et al. 1990; Neves et al. 2009; Sequeira et al. 2012; Zotti et al. 2007): Parabens are esters of p-hydroxybenzoic acid, and are mainly fungistatic and bacteriostatic.
Use of modified atmospheres: Nitrogen and carbon dioxide are gases that cause asphyxia but are not poisonous. They have been used for insect control in libraries, archives and museums (Kaplan and Schulte 1996; Rust et al. 1996; Smith 1986; Valentin and Preusser 1990; Valentin 1986). The material is placed in a chamber (in situ, together with the furniture that houses it if it is infected), the air is evacuated and nitrogen or carbon dioxide is pumped in. The method is effective for insect control, poses no threats for the health of the users and personnel and has no diverse effects on the treated materials. Nevertheless, Kobiakova and Dobrusina (2003) reported that carbon dioxide atmospheres did not harm cotton and pure cellulose paper, but that groundwood containing paper suffered a fair loss of folding endurance.
UV and γ-radiation: The use of UV radiation for disinfection is very limited due to the damage it inflicts on paper. Nevertheless, UV lamps were successfully used together with a microwave drying system to dry and disinfect wet books and documents (Hajek et al. 2011). UV radiation is used for the identification of active mould. Living fungi produce fluorescence, while dead do not. γ-radiation has been thoroughly studied for paper disinfection and disinfestation. According to Brokerhof (1989) and Justa & Stifter (1993), doses between 2 and 18 KGy are lethal for fungi, bacteria and insect larvae. Nevertheless, Tomazello & Wiendl (1995) report that doses up to 20KGy could not eliminate fungi in infected paper, although they considerably reduced its microbial load. Adamo et al. (1998) studied the effect of γ-radiation on pure cellulose paper and reported yellowing and significant depolymerization of cellulose even with low doses of 2 KGy, verifying the findings of Flores (1976) and Butterfield (1987). El-Esseily & Inaba (2004) reached the same conclusion for higher doses. In another study, Adamo et al. (2001) suggested the use of doses of 2-3KGy for the decontamination of (strong) paper, since the negative effects of radiation were considered to be negligible. In a later study though, Adamo et al. (2007) concluded that even doses up to 10 Gy do not significantly damage paper. Magaudda et al. (2000) studied the effects of γ-radiation on library infesting insects and found that the use of very low doses is effective since they induce sterility and molting to them. In a later study, Valentin Moise et al. (2012) suggest doses lower than 10 KGy for paper decontamination, and consider that a small paper degradation is acceptable, taking into account the overall preservation benefit. The good stability of the printing inks subjected to γ-radiation was verified by Rocchetti et al. (2002). Magaudda et al. (2001) and Adamo et al. (2003) reported that irradiated paper may be more prone to attack by cellulose eating insects and fungi growth. The principles of using ionisation technology for the disinfection and disinfestation of books and

documents, together with a review of several relevant experimental works are presented by Adamo et al. (2004).

Some other chemicals that have been used to counter paper biodeterioration include: the highly toxic methyl bromide, which has been used in combination with ethylene oxide, the suspected carcinogen paradichlorobenzene (insectifuge, insecticide), sodium hypochlorite, organotins, benzoic esters, sulfuryl fluoride and more (Brokerhof 1989; Craig 1986; Dersarkissian and Goodberry 1980; Gustafson et al. 1990; Hödl 1995; Johnson 1988; Kowalik 1980a, b; Sequeira et al. 2012). Laguardia et al. (2005) tested the use of plasma for the sterilization of paper. The method, apart from killing the microorganism, was found to increase the strength of paper.

Sequeira et al. (2012), concluded their review on antifungals in paper conservation by suggesting that calcium propionate, parabens and ethanol have the least effects on users health and paper stability, and that among them, parabens are the best antifungals. In another publication, Sequeira et al. (2014), express the opinion that the options currently available to counter paper biodeterioration by fungi are not totally satisfactory.

4. Surface/dry cleaning

4.1. Mechanical cleaning

Dry cleaning is used for the removal of dust, dirt, foreign materials, etc. from the paper surface for aesthetic reasons and/or to facilitate the preservation of the artefact. It can be used as a standalone cleaning technique or as the first step for a more complex intervention, for example for water washing and aqueous deacidification. Dry cleaning must precede aqueous treatments, because dirt can be transferred by water into the paper matrix and become fixed there, if it is not removed before the aqueous treatment (American Institute for Conservation - Book and Paper 2013). It is effected by use of white erasers (in block or powdered form), hard brushes, scalpels and tweezers. Specific commercial products for the surface cleaning of paper have also been developed, such as special sponges, dust absorbing materials (for example, Absorene), etc. Some of the soiling which can be generally classified as stain (for example fingerprints, blood stains, wax drops) may be important historical evidence and should not be removed. In many books, the most popular pages have more traces of usage, which should not be thoroughly cleaned so that this evidence is preserved. Apart from that, cleaning is often a disruptive process which may cause abrasion of the paper surface, force foreign materials into the paper matrix (either dust or eraser powder) and cause media damage. The dry cleaning of paper is discussed by several authors including Banks (1969), Appelbaum (1987), Nordstrand (1987), Batterham (1998) and Cumming & Colbourne (1998). Becker et al. (2011) present a semiautomatic cleaning apparatus suitable for dust removal, which uses electrostatic attraction. The apparatus can be used for fairly large objects, operates at a speed of 80 items per hour and has minimal mechanical impact, even on delicate materials.

4.2. Laser cleaning

According to Scholten et al. (2005), laser cleaning may be particularly appropriate for paper cleaning when conventional cleaning methods (mechanical, wet) cannot be applied (brittle papers, fissures and sensitive inscriptions, in the vicinity of

sensitive media). Laser cleaning would be able to provide the high spatial accuracy and the localized treatments when necessary.

Laser technology has been extensively tested for the cleaning of paper artefacts with various results, which depend on the type of laser, the light frequency, the pulse duration etc. Caverhill et al. (1999) reported paper yellowing after cleaning with Nd:YAG laser at 1.06 μm and humid thermal ageing and immediate oxidation after the treatment. Kolar et al. (2000a) used Nd:YAG laser at 1064 nm, pulse duration 6 ns and energy fluence up to 1.5 j/cm^2 and found no evidence of oxidation, but reported crosslinking of cellulose. Excimer laser at 308 nm (UV region) induced depolymerization of cellulose and loss of paper brightness, but Nd:YAG laser at 532 nm did not have any negative effect on paper (Kolar et al. 2000b). Similar results of Rudolph et al. (2004) and Kaminska et al. (2006) suggest that the usage of Nd:YAG laser at 532 nm with energy fluence under the ablation threshold must be safe for paper cleaning. Nevertheless, Balakhnina et al. (2013) reported discolouration of historic paper treated with laser at 532 nm after 5 years of natural ageing.

5. Wet cleaning

5.1. Washing in water/organic solvents

The immersion of paper in water removes the water-soluble compounds originating from paper hydrolysis and oxidation, microorganisms metabolism, atmospheric pollution, usage etc. Thus, apart from cleaning, which is the obvious result of paper washing, immersion in water stabilizes paper chemically, since a part of the removed compounds is acidic.

Clean lukewarm tap water, free from chlorine and transition metals, is preferred to distilled or deionized water. The high hardness value of water which originates from Ca and Mg salts is desirable, since it renders the water alkaline, assists paper neutralization and contributes to the alkaline reserve (Bansa 1998; Hey 1979; Lienardy and Van Damme 1990a; Tang 1981; Zervos 2007a).

Burgess (1986) used gel permeation chromatography in order to determine the molecular weight distribution of cellulose after washing with water which contained small quantities of salts (up to 40 ppm) before and after accelerated ageing. She concluded that the local water supply and the calcium sulphate solution offered better results than those of deionized water and the calcium bicarbonate solution.

In order to facilitate paper wetting, especially when paper has been infected by fungi which secrete hydrophobic products or when it is heavily sized, spraying with a mixture of ethanol or isopropanol and water (30-50% alcohol) before the water bath is recommended. Concerning the actual water bath, 3 to 4 water changes every 5-10 minutes for 40-80 minutes in total are usually sufficient (Hey 1979; Lienardy and Van Damme 1990a; Sistach 1996). Washing can be prolonged for several hours if no deterioration of the paper artefact is observed (mainly of inks and media), but interrupting it is recommended when water stops becoming yellowish. Local washing can be applied on the vacuum table.

Daniels & Kosek (2004a) studied the influence of surfactants, temperature and conditioning on the washing rate. In a next study (Daniels and Kosek 2004b), they tested seven different paper washing techniques including immersion washing, float washing, blotter washing, suction with sprayed water and a traditional Chinese washing method. The Chinese method -which essentially consisted of short immersion in hot (85°C) water- proved the most effective, most probably

because of the high temperature of the bath. Uchida et al. (2007) evaluated three aqueous washing methods by measuring the extent of the organic and inorganic acids extraction after washing. They concluded that immersion washing is the most efficient washing method, followed by suction table washing with ultrasonic mist -especially for thin and porous paper. Schalkx et al. (2011) compared the results of capillary unit, blotter or paraprint washing and recommend the first for easily wetting papers and the third for those that wet slowly. The removal of encrusted layers of mud can be made easier by using an ultrasonic oscillator in the water bath (Hummert and Pataki-Hundt 2010).

Before a wet treatment is implemented, inks and media must be tested for solubility in the solvent used (either water or organic). If they are even slightly soluble, there is a chance that they will fade, leak or smudge. Water-soluble inks or media should be fixed before aqueous treatments. Various chemicals have been proposed for that purpose, such as cyclododecane, Paraloid B72, Klucel G, PVal, Rewin ELTM, Mesitol NBSTM, Sandofix WE, Cartafix GS, Cartafix NTC and others (Bicchieri and Mucci 1996; Bluher et al. 1999; Bredereck and Siller-Grabenstein 1988; Havlínová et al. 2005; Porto and Shugar 2008). Muñoz-Viñas (2007) introduced a dual layer waterproofing technique based on cyclododecane and Paraloid B72 for fixing fugitive inks and colorants.

During wet treatments paper must be supported, so that stresses during handling that could cause strength loss or damage are kept to a minimum. To that end, non-woven polyester water permeable sheets (possible commercial names include Holytex, Remay, etc.) are used (Hey 1979). It is important that the supporting material is non-woven, so that no pattern is imprinted on the paper artefact even after pressing. At the end of the wet treatments, paper is usually dried flat under slight pressure.

Water washing, apart from its cleansing action, has also a deacidification effect because it removes the water soluble acidity of paper (Hey 1979; Lienardy and Van Damme 1990a). Several researchers report an increase in elasticity and strength (mainly folding endurance) of paper after aqueous treatments, often attributed to the rearrangement of cellulose fibres and the insertion of water molecules in the hydrogen bonds between cellulose chains (Lienardy and Van Damme 1990a; Sclawy 1981). On the other hand, a decrease in tensile strength and other strength properties has been reported by many authors after aqueous treatments (Green and Leese 1991; Lienardy and Van Damme 1990a; Sistach 1996; Wilson et al. 1981). Moropoulou & Zervos (2003) investigated the effect of aqueous treatments on the strength of paper and advise caution on their implementation. They do not consider aqueous treatments as mild interventions, and they speculate that strength loss may have been caused by mechanical damage and/or loss of bonding among cellulose fibres. In a more recent paper, Zervos & Barmpa (2011) propose a tentative mechanism that accounts for the microstructural changes, strength loss and higher stretch at break observed after aqueous treatments, based on evidence indicating a decrease in the bonded area of cellulose fibres.

Organic solvents that can be used in paper conservation include N-methyl-2-pyrrolidone as a solvent for old flour paste (Harding 1977), carbon tetrachloride and methanol (for greasy stains), hexane, toluene and ethyl acetate (for greasy stains and scotch tape residues removal) and 1,1,1-trichloroethane (for oil, grease, wax, varnishes, resins and tar) (Johnson 1988). The removal of pressure-sensitive tape from paper and the appropriate solvents are discussed by Smith et al. (1984) and Lennig (2010).

5.2. Enzymes treatment

The selectivity exhibited by enzymes is utilized in paper conservation for the removal of organic adhesive residues, since they only interact with specific substrates and have no degrading effect on paper and other media. Thus, stains of decayed, oxidized or moldy starch paste, warm glue, gelatine etc. should (at least in principle) be removed by using the proper enzyme. Enzymes can also facilitate the softening and solubilization of the adhesive, allowing for the separation of adhered fragile papers (or other materials such as cardboard, canvas, textile) without any damage to the artefact. Several authors emphasize the importance of maintaining the optimal pH and temperature for the best enzyme performance (Segal and Cooper 1977), although DeSantis (1983) concluded that the enzyme can still function adequately if only one of the prerequisite conditions is met.

One of the first references on the use of enzymes in paper conservation was made by Banks (1969), who suggested the use of collagenase for animal glue stain removal. Wendelbo & Fosse (1970) used trypsin to separate the pages of a water damaged manuscript. Segal & Cooper (1977) discuss the usage of amylase and protease for the removal of starch paste and warm glue. Hatton (1977) described the application of poultices of methylcellulose with amylase or protease. Nyuksha & Karpenko (1986) researched the use of amylase for starch paste removal. They recommended 1% concentration of the enzyme and 20-30 minutes duration of the intervention. Schwarz et al. (1999) developed a commercially available pad based on amylase for the removal of starch paste. In the same publication, they studied the results of the pad application and described its use. Amylase poulticing methods were tested by Schönbohm et al. (2004) for the detachment of silk pasted with starch from iron gall corroded documents. The poultices were prepared by incorporating methyl cellosolve and amylase into polyethylenoxide gels. The application of ultrasound was found to accelerate the action of α-amylases up to thirty times (Bartl et al. 2011). Bluher et al. (1997) attempted to remove drying oil stains from paper with lipase with little success.

5.3. Bleaching

Bleaching is classified under wet treatments, since the bleaching agent is usually applied as a solution. The main purpose of bleaching is cosmetic, that is, the removal of the overall discolouration or of disfiguring stains. It should be carried out with the utmost care – if done at all. Bleaching destroys chromophore groups by either oxidation or reduction, depending on the kind of the bleaching reagent, but at the same time affects paper (Anthony 2012). The chemistry of bleaching and the properties, the pros and cons and the preparation of the chemical bleaches used in paper conservation are discussed in several reviews (Anthony 2012; Brückle 2009a; Carter 1996c; Hey 1977; Lienardy and Van Damme 1988).

There are various bleaching methods, but the ones based on oxidative reagents are gradually being abandoned as routine treatments in paper conservation practice since they induce oxidation and may cause extended paper degradation, which entails the depolymerization (decrease of DP) of cellulose, the increase of carbonyl and carboxyl content, and strength loss. According to Hey (1977), bleaching may be one of the most harmful paper restoration treatments, and at the same time the least researched. In the introduction of Hey's authoritative article, which may be more than 35 years old but some of the views expressed in it are still current, she states the principles that must govern bleaching: "… *bleaching should only be carried out when stains detract from the visual appearance of the*

object or when either text or design is obliterated. Even then bleaching is only carried out to the extent that the physical appearance is improved and not necessarily to the point of total removal". And later on, she suggests that if paper is washed and deacidified, bleaching may not be necessary. In the same article, Hey presents the most important bleaching methods and provides information about their underlying reaction mechanisms, their practical applications and their advantages and disadvantages. She recommends deacidification before and after bleaching (the method with chlorine dioxide excluded), because some bleaching agents are very aggressive and others emit gases in acidic environments. She advises thorough washing and the use of acetic acid as anti-chlor after bleaching with chlorine containing reagents.

The need for thorough washing in order to remove the residues of the bleach and the use of anti-chlor for chlorine containing reagents are also discussed by Daniels (1976). The role of the OH radical and its kinetics in bleaching is discussed by Strofer-Hua (1991). Several publications compare the effects of various bleaching methods on the physical and chemical properties of paper (Burgess 1988; Burgess and Hanlan 1979; Henniges and Potthast 2009; Hofmann et al. 1991). It seems that the parameters of the procedure, that is the washing and deacidification before and after bleaching, the pH and the duration of the immersion are more important than the choice of the method itself (Hofmann et al. 1990). As a general conclusion, the best results concerning the chemical and colour stability are achieved with sodium borohydride and the worst with potassium permanganate (Anthony 2012; Hofmann et al. 1991).

Two issues of Restaurator dedicated to paper bleaching (issue 30:4, 2009 and 33:3-4, 2012) marked the reappearance of bleaching in the relevant literature after nearly 20 years of absence. The subjects discussed in the first, include industrial pulp bleaching (Suess 2009) and its similarities and differences from conservation bleaching (Brückle 2009a), the impact of bleaching on cellulose and paper (Henniges and Potthast 2009) and the decision-making parameters involved (Brückle 2009b). The second double issue deals among others with research on pretreatments that stabilize iron containing paper bleached with hydrogen peroxide (Niehus et al. 2012), light bleaching with various light sources (Schopfer 2012; Verborg 2012) and the evaluation of historical bleaching with chlorine containing reagents (Smith 2012). These new studies stress the risks of bleaching, verify the findings of the previous ones about the negative impact of oxidative bleaching on cellulose and paper and favour the use of borohydride.

Bleaching agents/methods in use, recommended in the literature

Sodium Borohydride (SB) *(or tetrahydroborate)* $NaBH_4$ (Anthony 2012; Heitner 1996; Henniges and Potthast 2009; Hey 1977; Hofmann et al. 1991; Lienardy and Van Damme 1988; Malešič et al. 2008): Borohydrides are reducing agents and apart from their mild bleaching effect, they stabilize cellulose by reducing the carbonyl groups produced by oxidation (Anthony 2012; Lehtaru and Ilomets 1997; Raber et al. 1981; Tang 1986). Instructions on the implementation of the method for paper bleaching are given by Hey (1977) and Lienardy & Van Damme (1988), who also acknowledge the stabilization effect of borohydrides. They recommend the use of 1 g of SB for 100 g of paper in aqueous or alcoholic solutions, and treatment duration from several minutes to 24 hours. According to them, the most important drawback of the method is the emission of hydrogen gas, and Lienardy & Van Damme (1988) recommend the approach of Tang (1986) in order to cope with it (see below). Tert-butylaminoborane has similar but weaker bleaching and reducing properties (Henniges and Potthast 2009).

Borohydrides are discussed again as chemical stabilization agents further below.

Hydrogen Peroxide H_2O_2 (Anthony 2012; Henniges and Potthast 2009; Hey 1977; Hofmann et al. 1990; Hofmann et al. 1991; Hummert et al. 2012; Lienardy and Van Damme 1988; Malešič et al. 2008; Niehus et al. 2012; Strofer-Hua 1991; Vodopivec and Letnar 1990): A mild bleach, it must be used in alkaline solution, because in acidic environment it releases oxygen bubbles which harm paper mechanically. The same effect is observed when used on paper containing metal ions (especially iron or copper), and its use is not recommended in such cases. Niehus et al. (2012) studied several pretreatments that stabilize iron containing paper when bleached with hydrogen peroxide. Although it leaves no residue, washing the paper after treatment is recommended.

Light Bleaching (Henniges and Potthast 2009; Hofmann et al. 1991; Lienardy and Van Damme 1988; Pavelka 1990; Schaeffer et al. 1997; Schopfer 2012; Verborg 2012): Both natural and artificial light can be used. There are several variations, but the most common entails the immersion of paper in dilute solutions of calcium hydroxide or magnesium bicarbonate. The UV component must be cut off by filters (Plexiglas, Mylar, Lexan). Recommended treatment duration: 3-5 hours for natural light, 8-16 hours for artificial light. Light bleaching is not recommended for lignin containing paper. The conservator must keep in mind the so called post-irradiation effect, which essentially means that apart from the inevitable degradation caused directly from the light exposure, the degrading mechanisms continue to be active for months after the treatment, even in dark storage (Atalla et al. 2000; Wilson and Parks 1983).

Bleaching agents/methods, not recommended in the literature or having several issues

Potassium Permanganate $KMnO_4$ (Baynes-Cope 1977; Henniges and Potthast 2009; Hey 1977; Hofmann et al. 1990; Hofmann et al. 1991; Lienardy and Van Damme 1988; Strebel 2012; Vodopivec and Letnar 1990): Potassium permanganate is a strong oxidizer and a very efficient bleach, used in the past to bleach all kind of stains, including foxing and ink stains. It causes extensive cellulose degradation. Because of the violet colour of the solution, the optical control of the bleaching is impossible. A dark brown precipitate due to manganese dioxide is formed inside the paper matrix, which is dissolved and removed at a second step which entails the use of sodium hydrosulphite, oxalic acid, citric acid or potassium metabisulphite solution. The traces of manganese that remain catalyze the further degradation of paper. Its use is not recommended by several researchers (Hey 1977; Lienardy and Van Damme 1988).

Sodium Hypochlorite, NaOCl (Anthony 2012; Hey 1977; Lienardy and Van Damme 1988; Smith 2012; Strofer-Hua 1991): Sodium hypochlorite is another strong oxidizer and very efficient bleach for the removal of foxing. It also causes extensive cellulose degradation.

Calcium Hypochlorite, $Ca(OCl)_2$ (Anthony 2012; Henniges and Potthast 2009; Hey 1977; Hofmann et al. 1990; Hofmann et al. 1991; Lienardy and Van Damme 1988; Malešič et al. 2008; Strofer-Hua 1991): Calcium hypochlorite is a strong oxidizing reagent, very efficient bleach and milder than sodium hypoclorite, causing less degradation to cellulose. Hypochlorites are considered the only bleaches that can remove fungi stains, but render paper unnaturally white.

Chloramine-T, (N-chloro-p-toluenesulphonamide), $C_7H_7ClNNaO_2S$ or *Chloramine-B (N-chlorobenzenesulfonamido),* $C_6H_5ClNNaO_2S$ (Anthony 2012; Daniels 1976; Hey 1977; Lienardy and Van Damme 1988; Smith 2012): An

oxidizer, it was considered a mild bleaching reagent with good results (it does not produce so white paper as hypochlorites). It was shown that it forms complexes with inorganic salts (such as alum) and that it is almost impossible to remove from paper (Daniels 1976; Hey 1977; Lienardy and Van Damme 1988).
Chlorine Dioxide ClO_2 (Anthony 2012; Brückle 2012; Donnithorne 1979; Hey 1977; Lienardy and Van Damme 1990b; Meynell 1979; Smith 2012): An oxidizing reagent, very efficient bleach especially for lignin containing paper and foxing stains (Hey 1977). It poses health hazards because it is highly toxic and explosive (Anthony 2012). It is produced on the spot by mixing sodium chlorite and formaldehyde (Donnithorne 1979; Hey 1977).

Other bleaching reagents whose usage in paper conservation has been recently introduced or is not well documented include: *Sodium percarbonate* ($2Na_2CO_3 \cdot 3H_2O_2$) (Baldin et al. 2008), *Ozone* O_3 (Lienardy and Van Damme 1988), *Chlorous acid* $HClO_2$, a slow and mild bleach (Hey 1977) and *Sodium Perborate* $Na_2H_4B_2O_8$, another slow and mild bleach with no permanent results (Lienardy and Van Damme 1988; Poot 1964). More easily accessible information (albeit somewhat dated) on bleaching, including practical aspects, can be found online at http://www.conservation-wiki.com/wiki/BP_Chapter_19_-_Bleaching. We found particularly interesting the last chapter entitled "Special Considerations".

6. Chemical Stabilization

6.1. Deacidification

Deacidification is the main chemical stabilization strategy for paper. It is considered as the most important conservation intervention concerning the long-term preservation of paper. The negative effect of acidity on the longevity of paper was early recognized, but it was due to the work of Barrow (Barrow and Sproull 1959; Roberson 1981) that this understanding was spread and solidified among the archival and library community.

The principle behind deacidification is quite simple: since acid hydrolysis is by far the most important degradation mechanism in the case of paper (Barański et al. 2005; Barrow and Sproull 1959; Baty et al. 2010; Carter 1996b; Fellers et al. 1989; Gurnagul et al. 1993; Roberson 1981; Whitmore and Bogaard 1994; Wilson and Parks 1979; Zervos 2010; Zou et al. 1994), the neutralization of the acid content of paper appears as the obvious solution. The target of deacidification is not only the neutralization of the acids, but also the deposition of an alkaline substance that will neutralize the acidity that may develop in the future (alkaline or alkali reserve). The adequacy and stability of the alkaline reserve is an important criterion of a successful deacidification intervention (Ahn et al. 2012a; Begin et al. 1999; Lienardy 1994; Zervos and Moropoulou 2006). Deacidification with various mildly alkaline agents has been demonstrated to significantly reduce the degradation rate of paper and is a widely practiced stabilization strategy (Bansa 1998; Barrow and Sproull 1959; Baty et al. 2010; Brandis 1994; Bredereck et al. 1990; Bukovsky 1999; Calvini et al. 1988; Carter 1996b; Cheradame et al. 2003; Daniel et al. 1990; Daniels 1996; Dupont et al. 2002; Green and Leese 1991; Guerra et al. 1995; Hanus 1994; Havermans et al. 1995; Hey 1979; Kelly and Fowler 1978; Kelly et al. 1977; Kolar and Novak 1996; Lienardy 1991, 1994; Lienardy and Van Damme 1990b; Liers and Schwerdt 1995; McGee 1991; Middleton et al. 1996; Mihram 1986a; Mihram 1986b; Moropoulou et al. 2001; Morrow 1988; Rousset et al. 2004; Shahani and

Hengemihle 1995; Smith 1977; Smith 1988; Stauderman et al. 1996; Stroud 1994; Tang 1981; Vallas 1993; Walker 1977; Wilson et al. 1981; Wittekind 1994; Zervos 2007b, 2010). The final pH of paper must be in the neutral or mildly alkaline region (7-9.5). Higher pH values facilitate other degradation routes such as alkaline degradation and autoxidation, especially for oxidized cellulose present in old paper (Golova and Nosova 1973; Havermans and Dufour 1997; Kolar 1997; Kolar and Novak 1996; Kolar et al. 2001; Whitmore and Bogaard 1994). It should be noted that deacidification cannot restore the lost mechanical strength of aged paper.

The idea of deacidification appears at the end of the nineteenth century. Around 1890, barium hydroxide in methanol was used for deacidification in the Victoria and Albert Museum (Smith 1988). Methods based on aqueous solutions followed, and then on organic solvents and in gaseous phase. Several methods were abandoned for various reasons and others evolved to complicated mass deacidification systems of industrial proportions. Mihram (1986a; 1986b) has published two annotated reviews on deacidification methods with summaries, structured in thematic sections. Extensive literature reviews on deacidification methods have also been authored by Linerdy & Van Damme (1990b), Lienardy (1991, 1994), Cedzová et al. (2006) and Baty et al. (2010). Practical advice on the choice of the most efficient deacidification system and the related equipment in large conservation workshops is given by Bredereck et al. (1990).

The ideal deacidification method should (Blüher and Vogelsanger 2001; Brandis 1994; Wittekind 1994):

- remove all the soluble acidic content of paper
- fully neutralize any remaining acidity
- remove as much as possible of the products of neutralization
- deposit a chemical substance (alkaline reserve) capable of rendering the paper alkaline -with a pH between 8 and 9.5- and keeping it so indefinitely (Ahn et al. 2012a; Bukovský 2005). Alkaline reserve should exceed 0.5% in magnesium carbonate, or 1% according to newer findings (Ahn et al. 2012a). The most accurate method for the determination of alkaline reserve is titration (Ahn et al. 2012a; Bukovský 2005), but other non-destructive methods have also been suggested (Vives et al. 2004).
- ensure a even deposition of the alkaline reserve in the mass of paper – even in the mass of a whole book, for mass deacidification
- have no negative effects on the materials of the deacidified objects (paper, leather, ink, dyes etc.) and must not accelerate their ageing
- not use toxic and environmentally hazardous chemicals
- not implement extreme conditions that may be detrimental to the materials of the artefacts (such as high temperature, intense drying, mechanical stresses etc.)
- not visibly alter the artefacts and leave residues and remaining odours

The ideal mass deacidification method should be applicable to all kinds of books and archival materials without preselection and preparation.

Deacidification in conservation workshops is mainly achieved by immersion of the paper artefact in the deacidification bath, which can be either aqueous or based on organic solvents. As with washing, paper objects must be supported during wet treatments with non-woven polyester web, in order to avoid physical damage during handling (Hey 1979). As will be discussed later, spraying with the deacidification agent is another usual technique. There are also gaseous methods, mass deacidification methods and miscellaneous methods. In the next chapters,

the most important deacidification methods will be presented, with emphasis given on the recommended methods. The interested reader is strongly advised to also study the very thorough review on deacidification by Baty et al. (2010), which is readily accessible online.

6.1.1. Aqueous deacidification

As mentioned above, partial deacidification is accomplished by washing in water for 1 to 2 hours, because of the removal of the water soluble acidity of paper (Hey 1979; Lienardy and Van Damme 1990a). For that reason, but also because the swelling of paper accomplished by washing facilitates better penetration of the deacidification agent into the paper matrix, the washing stage is considered necessary before the actual deacidification (Lienardy and Van Damme 1990b). Thus, more alkaline reserve is deposited when washing precedes aqueous deacidification (Lienardy and Van Damme 1990a; Lienardy and Van Damme 1990b). At the actual stage of deacidification, the deacidification agent neutralizes the remaining water insoluble acidic content of paper, and dissolves and facilitates the removal of some of the products of deacidification. Part of the deacidification agent is deposited in the paper matrix and forms the alkaline reserve, which remains in the paper. Hey (1979) and Lienardy & Van Damme (1990b) discuss practical aspects of aqueous deacidification. Kelly & Fowler (1978) studied the penetration of the deacidification agent in the paper matrix.

Aqueous deacidification is preferred for the treatment of loose sheets of paper, and bound books must be disbound and separated into loose leaves before the application of an aqueous treatment (Baty et al. 2010).

Aqueous deacidification agents in use, recommended in the literature

The following aqueous deacidification agents are in use, and are recommended in the literature (Hey 1979; Lienardy and Van Damme 1990b):

Calcium Hydroxide, $Ca(OH)_2$: According to the literature, deacidification with calcium hydroxide is the easiest to apply, the cheapest and the most common method (Hey 1979). According to Lienardy & Van Damme, (1990b) and Hey (1979), it also has the best results. The high pH of the deacidification bath (~ 12) is considered as the most serious drawback of the method, since it may cause yellowing to lignin-containing paper (Hey 1979) and change the colour of iron gall ink from black to brown (Lienardy and Van Damme 1990b; Reissland 1999). The method has been extensively studied by several scientists (Bredereck et al. 1990; Calvini et al. 1988; Hey 1979; Jancovicova et al. 2012; Lienardy and Van Damme 1990b; Pavelka 1990; Reissland 1999; Sundholm and Tahvanainen 2003a, b, 2004; Zappala 1997; Zervos 2007a). Sistach (1996) reports a loss of tensile strength due to deacidification with calcium hydroxide. Shahani et al. (1995) reported that calcium hydroxide fixes the Cu ions that have been adsorbed by cellulose, making their removal by later treatments more difficult. The washing and deacidification of paper with calcium hydroxide in the same operation has been studied by Tang (1981). Kolar & Novak (1996) carried out ageing experiments on paper samples treated with calcium hydroxide and concluded that this method has better DP retention after accelerated ageing than deacidification with $Mg(HCO_3)_2$. Sundholm and Tahvanainen (2003a, b, 2004) proposed and extensively tested a method for the simultaneous deacidification and strengthening of paper with a mixture of calcium hydroxide and methyl cellulose.

According to Hey (1979), the preparation of the deacidification bath is quite simple: 2 grams of $Ca(OH)_2$ are added per litre of water ($Ca(OH)_2$ solubility: 1.6 g/l), the mixture is shaken, and the suspension is kept undisturbed until all the

undissolved solid precipitates. The clear solution is decanted and can be used as is, but it is usually diluted with the same volume of water. The resulting solution needs no titration and is referred to as semisaturated solution (~ 0.8g/l or 0.018 N), because it contains half the calcium hydroxide of the saturated solution. Diluting the solution considerably slows down the precipitation of $CaCO_3$ due to the absorption of CO_2 from the air. A half hour bath is recommended. The calcium hydroxide that remains in the paper matrix is gradually converted to calcium carbonate, by reacting with the carbon dioxide of the air (eq. 1):

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \quad (1)$$

The produced calcium carbonate comprises the alkali reserve, which can neutralize acids according to the following reaction (eq. 2):

$$CaCO_3 + 2CH_3COOH \rightarrow (CH_3CO)_2Ca + CO_2 + H_2O \quad (2)$$

Magnesium Bicarbonate $Mg(HCO_3)_2$: Magnesium bicarbonate is a common and recommended aqueous deacidification agent. It produces very good results, evaluated by mechanical properties after moist ageing, but after dry ageing, the deacidified papers may deteriorate faster than the untreated ones (Calvini et al. 1988; Lienardy and Van Damme 1990b; Middleton 1977). Two drawbacks of the method are cited in the literature, the first concerning paper yellowing (more intense than calcium hydroxide), especially for lignin-containing papers, and the other, the deposition of magnesium bicarbonate crystals on the surface of paper, producing the so called "gritting effect" (Hey 1979). It has been reported that these deposits are converted to foxing stains after humid ageing (Hey 1979; Kolar and Novak 1996). Several other publications deal with this method (Bansa 1998; Bredereck et al. 1990; Calvini et al. 1988; Hanus 1994; Jancovicova et al. 2012; Kelly and Fowler 1978; Kolar and Novak 1996; Pavelka 1990; Wilson et al. 1981; Zappala 1997). Shahani & Hengemihle (1986) report a reduction of the catalytic action of Fe and Cu ions on the autoxidation of cellulose after deacidification with magnesium bicarbonate. Enhanced ageing rate was determined by Daniel et al. (1990) for chemical pulp papers which were deacidified with magnesium bicarbonate and exposed to environment polluted with SO_2 and NO_2. Contrariwise, filter and newsprint paper were protected from the effects of pollution after deacidification with magnesium bicarbonate. Aqueous magnesium bicarbonate is the deacidification agent in the mass deacidification method of Bückeburg, presented further below.

The simplest and safest preparation of the deacidification solution is described by Hey (1979): 1.5 – 8.8 g of magnesium hydroxide are added per litre of water, and then carbon dioxide is bubbled through until the solution becomes clear. No titration is needed; the final concentration of the solution is determined by the initial concentration of the magnesium hydroxide. In order to achieve the recommended concentration of 0.04 M, 2.33 g $Mg(OH)_2$ per litre of water are required. Other researchers propose the use of basic magnesium carbonate or magnesium carbonate, but according to Hey (1979), these methods are complex, need titration of the final solution and may contaminate it with iron ions.

The magnesium bicarbonate that remains in the paper matrix is converted to magnesium carbonate or according to other researchers (Calvini et al. 1988) to magnesium oxide, which comprise the alkali reserve (eq. 3 and 4). Both magnesium carbonate and magnesium oxide can react with acids and neutralize them.

$$Mg(HCO_3)_2 \rightarrow MgCO_3 + H_2O + CO_2 \quad (3) \text{ or}$$

$Mg(HCO_3)_2 \rightarrow MgO + H_2O + 2CO_2$ (4)

Calcium Bicarbonate $Ca(HCO_3)_2$: Calcium bicarbonate is another common and recommended deacidification agent with good results. Lienardy & Van Damme (1990b) do not recommend it because it causes paper yellowing and produces the lowest paper pH compared to the previous methods (up to 8.1). On the other hand, Bansa (1998) concluded that calcium bicarbonate is better than magnesium bicarbonate considering mechanical strength and colour. Zappala (1997) states that the pH of the saturated solution of calcium bicarbonate (and that of magnesium bicarbonate) is not reproducible. The deacidification solution can be prepared in a similar manner to the magnesium bicarbonate solution presented above. Calcium bicarbonate is converted to calcium carbonate which acts as discussed previously.

Mixture of Magnesium Bicarbonate $Mg(HCO_3)_2$ *and Calcium Bicarbonate* $Ca(HCO_3)_2$ (5/1): It has been used by Sistach (1996) with very good results. Bredereck et al. (1990) describe alternative methods for the preparation of the solution of the two salts.

Borax ($Na_2[B_4O_5(OH)_4]\cdot 8H_2O$): The use of borax is not recommended for lignin-containing papers (Lienardy and Van Damme 1990b). It may cause changes in the colour of paper, inks, and dyes (Baty et al. 2010; Botti et al. 2006; Daniel et al. 1990). Lienardy & Van Damme (1990b) do not reject the method, although other researchers were more skeptic about it (Daniel et al. 1990).

Zappala (1997) tested calcium propionate and recommends it as an effective deacidification agent with antioxidant and fungistat properties. Rao & Kumar (1986) studied the use of 1% aqueous solution of sodium dehydroacetate (SDHA) as deacidification agent. A treatment combining deacidification with a mixture of magnesium and calcium bicarbonates, stabilization with the antioxidant KI and strengthening with a cationic starch Empresol N has been tested with good results by Jancovicova et al. (2012).

From the deacidification agents presented above, Bansa (1998) recommends the use of calcium bicarbonate, Hey (1979) and Kolar & Novak (1996) the use of calcium hydroxide and Sistach (1996) the use of 5:1 mixture of magnesium bicarbonate and calcium bicarbonate.

Aqueous deacidification agents now obsolete or not recommended in the literature

The following aqueous deacidification agents are now obsolete or they are not recommended in the literature (Baty et al. 2010; Hey 1979; Kelly 1972; Lienardy and Van Damme 1990b):

Barium Hydroxide $Ba(OH)_2$: Barium hydroxide is very toxic, and causes intense paper yellowing.

Barrow two-bath method $Ca(OH)_2 + Ca(CO)_3$: Complicated, it doesn't offer more than a single calcium hydroxide bath.

Sodium Hydroxide $Na(OH)_2$: A strong base, it causes severe alkaline degradation to cellulose and extensive shrinkage to paper (Calvini et al. 1988; Hey 1979).

Sodium Bicarbonate $NaHCO_3$: It causes alkaline degradation to cellulose (Calvini et al. 1988; Hey 1979).

Calcium Carbonate, Magnesium Carbonate $CaCO_3$, $MgCO_3$: Since both compounds are practically insoluble in water, the deposited alkali reserve is inadequate (Baty et al. 2010; Hey 1979; Reissland 1999).

Calcium Chloride and Ammonium Carbonate $CaCl_2/(NH_4)_2CO_3$: Lienardy & Van Damme (1990b) rejected this method and did not test it, because technical information was not available. They also expressed concern because of the

chlorine ions involved. On the basis of the literature findings, the method cannot be evaluated, and more research is needed to determine its effectiveness.

6.1.2. Deacidification in organic solvents

Aqueous deacidification is effective, simple and poses no health risks for users and conservators. Nevertheless, it presents two serious drawbacks: it cannot be applied to artefacts with water-sensitive inks and dyes, and is unsuitable for mass treatments. These shortcomings were overcome with the introduction of deacidification methods based on organic solvents. To quote Smith (1971): "*organic solvents are used because they wet paper more rapidly than water, have less swelling or distorting effect on paper, and are easier to dry from paper than water*". The evolution of these methods led to the mass treatment methods presented further below. In this chapter, methods that do not require sophisticated equipment and are suitable for workshop application are presented.

Non-aqueous deacidification agents in use, recommended in the literature

The following deacidification agents are in use, and are recommended in the literature (Baty et al. 2010; Lienardy and Van Damme 1990b):

Magnesium Carbonate $Mg(CO_3)_2$ *in methanol or ethanol*: Seki et al. (2005) and Seki et al. (2010) tested the simultaneous deacidification with magnesium carbonate and strengthening with cellulose ethers in methanol and ethanol. The dispersions were applied by spraying. It was estimated that ethanol takes approximately 3 times longer than methanol to dry, but since it is non toxic, it may be preferable.

Barium Hydroxide $Ba(OH)_2$ *in methanol*: Despite the high toxicity of both ingredients, the method has been reported to be in use by several workshops. Lienardy & Van Damme (1990b) measured an increase in folding endurance and pH, which was retained after accelerated ageing, but also a decrease in paper brightness. It also caused the fading of inks, with the notable exception of iron gall ink, which was not affected. A concentration of 1% and a 20 minutes bath are recommended. The method has been evaluated by Baynes-Cope (1969), Daniel et al. (1990), Lienardy & Van Damme (1990b) and Green & Leese (1991).

Methoxy Magnesium Methyl Carbonate (MMMC), Methyl Magnesium Carbonate (MMC) in a mixture of methanol and a perfluorocarbon: The method was developed by Kelly, Tang and Krasnow in 1977 (Kelly Jr et al. 1977) in order to replace the method with magnesium methoxide. The empirical formula of MMC is $CH_3OMgOCOOCH_3{\cdot}XCO_2$ (Kelly Jr et al. 1977; Porck 1996), but its exact structure has not been determined and may be more complex[1].

According to Lienardy & Van Damme (1990b), the method results in satisfactory retention of mechanical strength after wet and dry accelerated ageing, very good pH values and adequate alkali reserve. It minimally affects various types of inks, but causes yellowing to lignin-containing papers. In the same work, a method for the synthesis of the compound is described.

[1] There seems to exist some confusion in the literature concerning the name of this compound. According to Smith (1988) both terms "Methoxy Magnesium Methyl Carbonate" - MMMC and "Methyl Magnesium Carbonate" - MMC refer to the same compound, but the first better describes its composition. Nevertheless, Wei T'o has branded several products with the following chemicals (Reissland, 1999): Wei T'o No 2: Methoxy-magnesium-methyl-carbonate, Wei T'o No 3/4: Ethoxy-magnesium-ethyl-carbonate, Wei T'o No 10: Magnesium-methyl-carbonate, Wei T'o No 11/12: Magnesium-ethyl-carbonate.

The method can be applied by immersion or by spraying, and various commercially available products have been discussed in the literature (Phizz, Wei T'o, PTS No2 by Archival Aids). A 0.025 M solution and a 10 min. immersion are recommended. The commercially available product Wei T'o has been evaluated by Bredereck et al. (1990) and Hanus (1994). Bukovsky (1997, 1999, 2000) and Bukovsky & Kuka (2001) used 5% MMC and 4% MMMC in methanol for newspaper deacidification and measured a substantial decrease of the hydrolysis, oxidation and photooxidation rate of the treated paper samples after accelerated ageing. Nevertheless, they also reported a decrease of mechanical strength and severe yellowing immediately after deacidification. Green & Leese (1991) tested an 1.3% w/v MMC solution in methanol and the commercial product Phizz (2% w/v in a mixture of methanol and trichlorofluoroethane) and recommended Phizz for workshop non-aqueous deacidification. Daniel et al. (1990) evaluated the effectiveness of MMC for the protection of paper against atmospheric pollution. They observed an increased adsorption of acidic pollutants by the deacidified paper, which was expected, but also destabilization against pollution of certain papers due to deacidification.

Magnesium oxide particles suspended in perfluoroheptane: The Bookkeeper solution is available in spray form, for use in workshops on individual artefacts (see below).

Non-aqueous deacidification agents not recommended in the literature

The following deacidification agents are not recommended in the literature (Lienardy and Van Damme 1990b):

Magnesium Acetate, Calcium Acetate in methanol, ethanol (Zappala 1997): their hydrolysis produces acetic acid.

Magnesium Methoxide: The first non-aqueous deacidification method that could be practically applied by spraying to large amounts of paper material, developed by R.D. Smith in 1970 (Smith 1971, 1977). Unstable at high humidities, it is converted to white oxide gel. It was originally used in the commercially available product Wei T'o, but was replaced by MMMC because it clogged the spraying nozzle (Porck 1996).

Non-aqueous deacidification applied by spraying, compared to aqueous immersion deacidification, has the disadvantage that the products of neutralization are not removed from the treated paper. More options on spray deacidification for workshop applications can be found in the mass deacidification chapter below.

6.1.3. Gaseous deacidification

Gaseous deacidification is an ingenious idea that solves many technical issues pertaining especially to mass deacifidation. The most important yet unfruitful gaseous mass method presented so far is the DEZ method described further below. A variety of other gaseous methods have been proposed but were not adopted for various reasons (Lienardy 1991; Porck 1996):

Gaseous Ammonia: Lethal above concentrations of ca. 500 ppm and volatile, remains in the paper for very short period of time, thus offering very limited protection (Roberson 1981; Smith 1988).

Various Amines, Morpholine, Cyclohexylamine carbonate (Baty et al. 2010; Roberson 1981; Smith 1988; Walker 1977): Volatile, offer limited protection, carcinogenic.

6.1.4. Miscellaneous methods

Interleaving (Baty et al. 2010; Langwell 1973; Middleton et al. 1996; Vinther Hansen 2005): One of the easiest, inexpensive, not infrastructure demanding and risk free deacidification methods consists of placing the acidic sheet of paper in close contact with an alkaline sheet of paper containing calcium carbonate. According to Middleton et al. (1996), by maintaining high RH (97%) and applying pressure (350 KPa) to keep the sheets together, deacidification can be completed in less than a day. Langwell (1973) suggested the use of paper sheets impregnated with cyclohexylamine carbonate, but the method poses health risks since cyclohexylamine carbonate is carcinogenic. Interleaving can be used for the deacidification of bound books, but the extra bulk may damage their spine.

6.1.5. Mass deacidification systems

The main target of the mass deacidification methods is to rescue a significant part of the material of libraries and archives, mostly that produced between 1850 and 1970, from the destruction caused by acid hydrolysis (Hubbe 2005; Sclawy and Williams 1981; Smith 1987). These methods utilize advanced technical infrastructure of industrial proportions and can deacidify large quantities of books and paper material (in the order of 100,000 volumes per year). "*To be considered as a mass process, the maximum price for treating a 500g-book was set at EUR 25, and that for a double page of archival material at EUR 0.5*" (Anonymous 2006). It should be noted here that paper considered for mass deacidification must be sufficiently strong to be handled, since mass deacidification alone cannot restore lost strength. There exist a few mass methods that combine deacidification and strengthening, which will be discussed here and in the strengthening section further below.

Several research articles and reviews deal with the philosophy, the principles, the evolution, the evaluation and the viability of the mass deacidification methods (Anders 2013; Banik 2005; Baty et al. 2010; Baty and Sinnott 2005; Blüher and Vogelsanger 2001; Brandis 1994; Carter 1996b; Cheradame et al. 2003; Cunha 1977; Cunha 1987; Lienardy 1991, 1994; Smith 1971; Smith 1987, 1988; Thompson 1988; Turko 1990; Williams 1971; Yasue 1997). Lienardy (1994) tested the Wei T'o, Archival Aids, FMC, Bookkeeper, DEZ, BPA and Vienna methods; Brandis (1994) evaluated FMC, Akzo and Wei T'o; Carter (1996b) presents a summary of the literature on the evaluations of the DEZ, Wei T'o, FMC, Bookkeeper, Viennese, Book Preservation Associates, Sable and the Batelle processes; Dufour & Havermans (2001) studied the photosensitivity of mass deacidified paper by the Battelle, Bookkeeper, DEZ, FMC and Sable methods and concluded that deacidification increases the photo-oxidative degradation of the treated samples; Ramin et al. (2009) tested Libertec, Papersave Swiss, Bookkeeper and CSC Booksaver. There are also two excellent online mass deacidification bibliographies, one of them annotated by its author, which include evaluation of the quality of the publications (Adams 2011; Zimmmerman 1991). The mass deacidification systems can be classified in three major categories: liquid solutions, liquid suspensions and gas phase methods, the latter expected to have minimal interactions with sensitive media (Baty et al. 2010).

No mass deacidification method is suitable for all types of material, so in most cases preselection is necessary. Some of them may cause serious damage to some materials, such as immediate reduction of the strength properties of paper, smudging and fading of inks and media, distortion of books, white deposits and

persistent remaining odours, while others do not deposit adequate alkaline reserve (Anders 2013; Brandis 1994; Lienardy 1994; Porck 1996). Several aspects of mass deacidification need further research in order to minimize or eliminate those side-effects. Better delivery methods should be developed that achieve more homogenous distribution of the alkali reserve into the paper matrix (Banik et al. 2006; Wagner et al. 2008), especially for deacidification systems that rely on dispersions or gas-phase deposition of solids. An idea worth considering is to subject the treated papers to a high humidity post-treatment, in order to increase the penetration and improve the thickness-wise distribution of water soluble deacidification agents.

Mass deacidification has a relatively long history of more than 40 years. During this evolutionary course, several methods have appeared, evolved and reached practical implementation while others fell out of use. The focus of the following presentation will be on the successful methods, the ones that are currently practically implemented. The other methods will be also discussed, albeit in less detail.

Mass deacidification systems currently in use

Eight true mass deacidification processes have been identified currently in use worldwide (Anders 2013; Anonymous 2006; Baty et al. 2010), in more than fifteen variations, implemented by at least nine different companies and several public organizations in Europe, Japan, South Africa, the United States of America and Canada, almost all of them based on alkaline magnesium compounds (Anders 2013; Anonymous 2006). Most of them are not free of issues, which may include the formation of powdery depositions and the bleeding of inks and colours in some occasions, as side-effects of the treatments. Compiled technical details on the processes can be found in Anders (2013) and in the Proceedings of the International Conference "Save Paper", 2006, Swiss National Library (http://www.nb.admin.ch/nb_professionnel/erhalten/00699/01490/index.html?lang=en#sprungmarke3_2).

Bookkeeper (Anders 2013; Anonymous 2006; Baty et al. 2010; Blüher and Vogelsanger 2001; Hon 1989; Jablonský et al. 2013; Lienardy 1994; Porck 1996, 2006; Turko 1990; Zumbühl and Wuelfert 2001): Implemented by Preservation Technology L.P., U.S.A. since 1994, with 8 installations in U.S., Netherlands, Canada, Poland, Japan, South Africa and Spain, it is a commercially available service (www.ptlp.com). The Bookkeeper solution is also available as a spray, for use in workshops on individual artefacts. The process has undergone important changes since it first appeared, and does not implement predrying and vacuum exposure anymore. It is suitable for bound books, unbound material and even oversized documents. It uses insoluble magnesium oxide particles as deacidification agent, suspended in perfluoroheptane. The procedure includes the fanning of the book and the infiltration of the dispersed deacidification agent. The treatment lasts around 30 minutes plus 1.5 hours for raw materials retrieval per batch, which may consist of 8 to 12 books or the content of 2 archival boxes.

Bückeburg method [Bückeburger Konservierungsverfahren für moderns Archivgut Bückeburg, Conservation Process for Modern Archival Material] (Anders 2013; Anonymous 2006; Baty et al. 2010; Blüher and Vogelsanger 2001; Porck 1996; Wagner et al. 2008): Offered as a commercially available service by Hans Neschen AG, Archivcenter, Bückeburg (Germany) since 1998, with installations in Germany, Poland and Russia, it combines deacidification and strengthening in a continuous process. It entails the immersion of loose paper sheets in aqueous solution of magnesium bicarbonate and methylcellulose

(strengthening agent). The fixation of sensitive media is achieved by polyionic fixatives (cationic Rewin EL and anionic Mesitol MBS). The method can only be applied to single sheets, thus books should be disbound before treatment.

CSC Booksaver (Anders 2013; Anonymous 2006; Baty et al. 2010; Blüher and Vogelsanger 2001; Dupont et al. 2002; Henniges et al. 2004; Wagner et al. 2008): Offered as a commercially available service since 2001 by two companies, Conservacion de Sustratos Cellulosicos (CSC) in Spain and Preservation Academy, Leipzig in Germany, it has operating installations in Spain, Germany and Russia. It is also available as a spray for individual workshop application (Dupont et al. 2002; Henniges et al. 2004). The deacidification chemicals are propoxy-magnesium carbonate in 1-propanol (70%) and heptafluoropropane (HFC 227, which is odourless, non-toxic and non-flamable and environmentally friendly). One of the advantages of the method is the stability of the deacidification agent in the presence of moisture, especially the moisture of paper, a fact which allows the application of the method without pre-drying (Baty et al. 2010).

Libertec – SOBU or *Forced air* (Anders 2013; Banik 2005; Baty et al. 2010; Blüher and Vogelsanger 2001; Porck 1996; Ramin et al. 2009): Implemented by Libertec Bibliothekendienst GmbH and SOBU Sonder-maschinenbau und Buchentsäuerung, both in Nürnberg, Germany, the process is available commercially as a service since 1996. It is considered one of the simplest deacifidation systems, since the deacidification agents, calcium carbonate and magnesium oxide, are delivered by a stream of dry air circulating around the artefact. Ramin et al. (2009) evaluated 5 mass deacidification methods (Libertec, Papersave Swiss, Bookkeeper, CSC Booksaver) and concluded that the process is quite effective in achieving high pH and depositing sufficient alkaline reserve. On the other hand, the deacidification agent tends to remain on the surface of the paper and especially in the book fold, resulting in irregular deposition (Anders 2013; Banik 2005).

Papersave/Papersave Swiss, previously referred to as *the Battelle method* (Anders 2013; Andres et al. 2008; Blüher and Vogelsanger 2001; Havermans 1996; Lichtblau and Anders 2006; Liers and Schwerdt 1995; Porck 1996; Schwerdt 1989; Wittekind 1994): The process is implemented and offered as a commercial service by ZFB Zentrum für Bucherhaltung, Leipzig, in Germany, and Nitrochemie Wimmis AG, Wimmis, in Switzerland since 1996 in its initial form and 1996 and 2000 after modifications, and is suitable for books, archival material and single sheets. The Deutsche Bibliothek and the Swiss National Library have working installations of the Papersave process. The deacidification agent is magnesium titanium alkoxide $[Ti(OR)_4 \cdot Mg(OR)_2]_2$ dissolved in hexamethyldisiloxane (HMDO, $(CH_3)_3SiOSi(CH_3)_3$). It allows for the treatment of 500-2,000 books per batch (120 t/year). The process includes a drying stage at less than 50°C under vacuum, which brings the paper moisture down to less than 1%, followed by impregnation with the deacidification solution and finally the drying of the solvent. These steps last three days, but must be followed by a three-week conditioning stage and off-gassing, since alcohol produced by the process is exuded. The deacidification chemicals are transformed to magnesium carbonate, which acts as alkaline reserve. The treatment can raise the pH of paper up to 9 and deposit up to 2% of magnesium carbonate as alkaline reserve. According to Ahn et al. (2012a), the Papersave process is the most efficient in depositing adequate quantities of alkaline reserve.

ZFB:2 (Zentrum für Bucherhaltung) Procedure (Anders 2013): Offered as a commercially available service since 2012 by ZFB in Leipzig, Germany, it uses a suspension of calcium carbonate and magnesium oxide dispersed in heptane. The treatment is considered as gentle (Anders 2013), as predrying and reconditioning are not necessary. The annual capacity of the existing plant is 100 t of paper, and can treat both books and archival material. A search through Google Scholar did not yield any results concerning the scientific evaluation of the process.

Archival Aids or *Sablé system* (Anders 2013; Arnoult 1987; Baty et al. 2010; Blüher and Vogelsanger 2001; Bredereck et al. 1990; Carter 1996b; Lienardy 1994; Vallas 1993): The process has been modified in the past and is very similar to the Wei T'o, but uses ethoxymagnesium carbonate as deacidification agent. The Bibliotheque Nationale maintains a mass deacidification installation in Sable sur Sarthe Center/France with a capacity of 60,000 volumes per year (Anders 2013). The deacidification solution (methyl and ethyl magnesium ethoxy carbonates, less than 5% methyl alcohol, less than 15% ethyl alcohol and siloxane solvents, http://www.archivalaids.com/rsm/6/products/deacidification/ptda-deacidification-treatments-for-paper) can also be purchased and used for spraying or immersion of single items in a workshop environment by commercial vendors.

The Austrian National Library Process or *Vienna method* (Anders 2013; Baty et al. 2010; Blüher and Vogelsanger 2001; Lienardy 1994; Wächter 1987a): In operation since 1987, the method is used for treating bound newspapers with a capacity of 3,000 volumes per year. Unbound volumes are immersed in aqueous solution of calcium hydroxide and methylcellulose, and since 1999 borax was also added. The material is then shock-freezed at -30°C for one hour and then freeze-dried. Lienardy (1994) has rejected the method because it introduced inadequate alkaline reserve. The treatment combines deacidification and strengthening.

Other mass deacidification systems

The following processes are also discussed in the relevant literature of mass deacidification systems (Anders 2013; Anonymous 2006; Baty et al. 2010). Some of them have fallen out of use (such as the DEZ process) or are receding. A thorough internet search using their commercial brand names and implementation centres found in past literature could not always yield useful information concerning their current status as commercial services. For some of them, their commercial vendors advertise solutions that can be applied by dipping, spraying or brushing, but have no further information about current mass treatment installations.

Wei T'o System (Anders 2013; Baty et al. 2010; Blüher and Vogelsanger 2001; Brandis 1994; Carter 1996b; Cunha 1987; Green and Leese 1991; Hanus 1994; Hon 1989; Kelly 1972; Kelly et al. 1977; Lienardy 1991, 1994; Morrow 1988; Porck 1996; Scott 1987; Smith 1977; Smith 1987, 1988; Thompson 1988; Turko 1990): The method was invented by Richard D. Smith and has been modified many times since its invention. The most recent version used methoxymagnesium methyl carbonate as deacidification agent and methanol and perfluorocarbons as solvent system. The alkaline reserve, magnesium carbonate, is formed from the reaction of the deacidification agent with water. A mass treatment installation of the Wei T'o system with a capacity of 40,000 books per year was operated by the National Library and National Archives of Canada until 2002. Today, a Wei T'o spray is available for use in workshops on individual artefacts. The web site of the firm seems that was last updated in 1998. Nevertheless, Wei T'o Soft Spray Paper Guard No.111 is advertised and can be bought today from other online vendors

(for example, Amazon). Wei T'o was named after an ancient Chinese god regarded as protector of books.

DEZ or *Akzo process* (Anders 2013; Baty et al. 2010; Brandis 1994; Cunha 1987; Lienardy 1994; Porck 1996; Smith 1987; Sparks 1987; Stroud 1994; Thompson 1988; Turko 1990): Akzo Chemicals commercialized the DEZ process invented by J.C. Williams and G.B. Kelly of the Library of Congress (LOC). The deacidification agent is diethyl zinc in gaseous form. The method was one of the three evaluated for use by the LOC in 1992 (Brandis 1994), the other two being FMC and Wei T'o. The evaluation showed that none of the three methods could effectively treat all library materials and that all three of them adversely affected the condition of the books, and as a result the LOC chose none of them. The method was thoroughly tested with promising results (Havermans et al. 1995; Kelly and Williams 1981; Lienardy 1994), but in spite of that, most probably due to the risks associated with the use of DEZ (Cunha 1987; Thompson 1988), Akzo decided to stop it in 1994.

FMC or *Lithco process* (Anders 2013; Baty et al. 2010; Brandis 1994; Lienardy 1994; Porck 1996; Wedinger 1991): The deacidification agent was magnesium butoxytriglycolate in freon 113, which was later replaced with heptane. The process involved dielectric heating (radio frequency drying), impregnation with the solution for 5-10 minutes, recovery of the solvent and finally the drying of the books for 3 hours by dielectric heating. The FMC process was also stopped.

Book Preservation Associates (BPA) and DAE process (Anders 2013; Baty et al. 2010; Lienardy 1994; Turko 1990): Books and library material are exposed first to ammonia under vacuum and then to ethylene oxide. That way, primary, secondary and tertiary amines are produced, which form the alkaline reserve. According to Lienardy (1994) the method should be rejected because the alkaline reserve was not stable. Health risks also should not be underestimated, since ethylene oxide and ethanolamines are toxic and carcinogenic. The Japanese company Nippon Filing modified the process and offered it for commercial use in 1998, and despite the fact that it uses the same reagents claims that the ethanolamines produced are more stable (Baty et al. 2010; Okayama et al. 1996).

A French research team (Cheradame 2006; Cheradame et al. 2003; Ipert et al. 2005; Rousset et al. 2004) studied the use of aminosilanes and alkoxysilanes in ethanol for mass deacidification. Their results suggest that alkoxisilanes do not only deacidify paper but also strengthen it. The use of supercritical carbon dioxide as solvent for calcium carbonate was suggested by Selli et al. (2000) for the development of a non toxic mass deacidification system, which with the addition of catechol and ethyl alcohol as a co-solvent can also increase the strength of the treated paper.

6.1.6. Emerging nanoparticle technologies

The use of magnesium and calcium hydroxide nanoparticles dispersed in alcohols for the deacidification of paper is presently very actively researched with promising results (Carretti et al. 2013; Poggi et al. 2011; Poggi et al. 2010; Poggi et al. 2014; Poggi et al. 2013; Stefanis and Panayiotou 2007, 2008, 2010; Wójciak 2015). The European Research Centre for Book and Paper Conservation-Restoration is currently working on the development of a mass deacidification system based on "*supercritical carbon dioxide as solvent or volatile organic solvents in combination with multifunctional hydrophobically modified nanoparticles and functional silanes*" (http://www.buchstadt.at/Research-Activities.299.0.html) (Baty et al. 2010).

6.2. Other chemical stabilization methods

6.2.1. Reduction with borohydrides

Alkaline environments facilitate cellulose autoxidation, and oxidized cellulose is prone to degradation even in mild acidic or alkaline environments by various routes (including β-elimination), which may lead to various low molecular products and cause cleavage of the glycosidic bonds neighbouring the introduced carbonyls (Bicchieri and Pepa 1996; Kolar 1997; Lewin and Epstein 1962; Richards 1963; Whitmore and Bogaard 1995). The above facts are of particular interest in paper conservation context because of the mild alkaline environment produced by deacidification to historic papers, which are always oxidized to a certain degree (Ahn et al. 2012b).

As discussed above, borohydrides are reducing agents. They can be used for mild bleaching, but they may have a more important function in paper conservation as they can stabilize oxidized cellulose by reducing the carbonyl groups produced by oxidation (Anthony 2012; Bicchieri et al. 1999; Bicchieri and Brusa 1997; Bicchieri et al. 2000; Henniges and Potthast 2009; Hey 1977; Hofmann et al. 1991; Lehtaru and Ilomets 1997; Lienardy and Van Damme 1988; Raber et al. 1981; Tang 1986). Several compounds are proposed in the relevant literature, including sodium borohydride (Block and Kim 1986; Nevell 1963), tetramethylammonium borohydride (Block and Kim 1986; Raber et al. 1981), tert-butylaminoborane (Henniges and Potthast 2009) and others (Bicchieri et al. 2000). Tang (1986) suggests the following procedure in order to increase the efficiency of the method and limit the side-effects, including the gaseous hydrogen production: first, deacidification with magnesium bicarbonate (0.007%), then reduction with SB (0.1%) and finally immersion in dilute calcium hydroxide solution. The effectiveness of the method in stabilizing cellulose and paper has been demonstrated by several studies (Dupont 1996; Henniges and Potthast 2009; Lehtaru and Ilomets 1997; Tang 1986; Zappala 1997).

6.2.2. Transition metal deactivation

Transition metals, especially Fe and Cu, can play a very important role in cellulose oxidation if their concentrations are relatively high, which is the case with iron stains, foxing or with the presence of certain inks and colorants (iron gall ink, verdigris) (Bicchieri and Pepa 1996; Calvini and Gorassini 2002a; Shahani and Hengemihle 1986; Williams et al. 1977). They catalyze the production of free radicals from hydroperoxides, which are mainly produced by cellulose autoxidation (Bicchieri and Pepa 1996; Kolar 1997). They can be deactivated by complexing agents/antioxidants, which either change their reductive/oxidative properties or make them inaccessible by steric hindrance (Area and Cheradame 2011).

Burgess (1991) proposes various methods for the removal of iron: acid solubilization, chelation with various chelating agents such as EDTA, or reduction of Fe^{+3} (reddish insoluble) to Fe^{+2} (colourless soluble) in combination with chelation with a reducing agent such as dithionite and EDTA. She also presents diagnostic methods for the identification of iron. EDTA and sodium dithionite were also successfully used for the removal of residual iron from platinum prints (Gent and Rees 1994). Lehtaru & Ilomets (1997) used EDTA in combination with the reducing agents sodium thiosulphate and sodium borohydride for the removal of copper and iron. An antioxidant effect in the presence of iron was observed by

Strlic et al. (2001) for the compounds DTPA, phytate and desferal. EDTA together with sodium dithionite was recommended by Suryawanshi and Bisaria (2005) for the removal metallic stains. Malešič et al. (2008) used various bleaching agents for the removal of foxing stains and found that in case of high concentrations of iron, treatment with EDTA in a sodium dithionite solution was the most efficient. For low transitional metal content, they recommend washing and deacidification, as well as treatment with sodium borohydride. Irwin (2011) used sodium metabisulfite and sodium dithionite for the removal of rust stains from paper, and concluded that sodium dithionite is the most effective of the two chemicals tested. He also discusses the health risks and side effects associated with the use of these compounds. Acid solubilisation was used by Hummert et al. (2012) for the removal of foxing stains caused by iron. Meyer and Neumann (2009) tested short and long chained gelatine and recombinant proteins for the inhibition of copper pigment (verdigris) corrosion, with the latter being the most effective.

6.2.3. Iron gall ink stabilization

Iron gall ink corrosion poses a serious threat to the world written heritage, especially of Europe and its former colonies, including North and South America and Australia, since iron gall ink was the main ink used there from the 9th until the 20th century (Metz 1997; Orlandini et al. 2008). A significant part of manuscripts and drawings made with iron gall ink is in a critical stage or unusable because of iron gall ink corrosion (De Bruin 1997).

Iron gall ink was produced by mixing iron (II) sulphate with gall extract, whose active component is tannic acid, gum arabic and various other ingredients such as colorants, wine, vinegar, etc. A pale coloured, water soluble ferrous tannate complex was produced. The final black colour of the ink developed on paper after oxidation of the ferrous tannate to insoluble black ferric tannate (De Feber et al. 2000; Neevel 1995; Sistach et al. 1999). Several publications focus on the study, identification and detection of iron gall ink (Budnar et al. 2006; Budnar et al. 2001; d'Agata et al. 2007; Havermans et al. 2003a; Havermans et al. 2003b; Lee et al. 2006; Lee et al. 2008).

Although many compositions of iron gall ink are quite stable, several others are corrosive and destroy the paper substrate (Banik 1997; De Feber et al. 2000; Hähner 2006; Neevel 1995; Sistach et al. 1999). Two mechanisms that run in parallel are discussed in the literature: acid hydrolysis due to the low pH produced by the hydrolysis of iron(III) ion, and oxidation induced by iron(II) ions that are not bound by the tannin (Banik 1997; Neevel 1995; Potthast et al. 2008; Wunderlich 1994). For the detection of the reactive iron(II) ions, the bathophenanthroline indicator paper was introduced (Neevel 2009; Neevel and Reissland 2005).

Several strategies were implemented in the past to combat iron gall ink corrosion. At the end of the 19th and the beginning of the 20th century, the methods used included impregnation with zapon (cellulose nitrate), infills to iron gall corroded parchment with gelatine and formol, ammonia vapors neutralization and collodion (cellulose nitrate) consolidation (Posse 1970). Later on (1940-1960), lamination with transparent papers and probably starch paste was introduced, together with stabilization using chiffon-silk (Schönbohm et al. 2004) and lamination using acetate or PVC-films (Reissland 1997; Wouters et al. 1990). These methods have proven very destructive and almost impossible to reverse.

Several other methods are found in the literature, which have been used the last 30 years, are still being used or are at experimental stage. They include (Neevel and Reissland 1997; Reissland 1999; Van Gulik 1997; Van Gulik and Kersten-Pampiglione 1994):

- Deacidification with various compounds
- Boiling in water (Tse et al. 2005)
- Paper splitting (Brückle and Dambrogio 2000) (see below)
- Electrolysis
- Radical scavengers
- Ammonium caseinate treatment
- Oxidation inhibitors
- No treatment or consolidation only (Low 1994; Titus et al. 2009)

There are several concerns in applying aqueous treatments to iron gall corroded paper, including ink solubilization and spreading, iron diffusion to non-inked areas, loss of fragments of corroded paper, and of course long term effects, especially the issue of "waking up" the ink (Huhsmann and Hähner 2008; Neevel and Reissland 1997; Van Gulik and Kersten-Pampiglione 1994). Many colleagues advised against aqueous treatments, while others suggested that with caution and when the ink appears stable, aqueous deacidification is a possibility that may improve the condition of the artefact (Van Gulik and Kersten-Pampiglione 1994).

From the above mentioned methods, the use of oxidation inhibitors in aqueous solutions has been thoroughly investigated and has become the established and recommended method for the treatment of iron gall ink corrosion. Several variations of the method can be found in the literature, but they all include a deacidification step, which neutralizes the acidity of the ink and slows down acid hydrolysis, and the use of a phytate compound as the oxidation inhibitor, which complexes the free iron(II) ions that act as an electrophilic catalyst. While deacidification alone can stabilize the ink, the combined treatment was able to further increase the expected lifetime of paper (Neevel 1995). The use of aqueous sodium phytate solution combined with deacidification with aqueous magnesium bicarbonate was first proposed by Neevel (1995), and was further researched and developed by several scientists (Botti et al. 2005; Havlínová et al. 2007; Henniges et al. 2008; Kolar et al. 2007; Kolar et al. 2005; Pedersoli Junior and Reisland 2003; Potthast et al. 2008; Reissland 1999; Zappalà and Stefani 2005), who verified the effectiveness of the treatment.

Havermans et al. (2003b), applied multispectral imaging, a non destructive technique, in order to assess the amount of damage of the paper substrate caused by iron-gal ink corrosion. Pedersoli Junior and Reisland (2003) discuss a methodology for the quantitative estimation of the risks associated with the possible conservation actions of iron gall damaged papers, and consider 4 possible scenarios: no action, preventive conservation, combined calcium phytate/calcium bicarbonate treatment and paper splitting. A new alternative method for the evaluation of the effects of treatments of iron gall corroded documents is proposed by Kolar & Strlic (2004). Kolbe (2004) recommends the use of gelatine for resizing iron gall ink manuscripts, since according to his research it slows down ink corrosion, an opinion shared by Titus et al. (2009). This claim was not verified by later research (Potthast et al. 2008). Gum arabic has also been found to slow down cellulose degradation due to iron gall ink corrosion (Remazeilles et al. 2004). According to Kolar et al. (2005), the pH of the phytate solution is critical, since at pH 6.2 the stabilization effect was at least double than that at pH 5.0. Malesic et al. (2005) studied the effects of various quaternary ammonium and

phosphonium halides on iron gall ink corrosion of paper in alkaline pH. Various other compounds have been studied for the stabilization not only of iron gall ink but for copper pigments (verdigris), such as 1-ethyl-3-methylimidazolium bromide, 1-butyl-2,3-dimethyl-imidazolium bromide and tetrabutylammonium bromide, in combination with magnesium ethoxide in ethanol (Kolar et al. 2008). The method of interleaving with paper impregnated with alkaline buffer and antioxidant ($CaCO_3$/NaBr) was studied by Vinther Hansen (2005). Interleaving with paper impregnated with various halides that work as radical scavengers (NaCl, NaBr, $CaBr_2$) was also studied by Rouchon et al. (2013), who point out that the addition of $CaCO_3$ does not improve the results. Kolar et al. (2007) proposes the use of magnesium instead of calcium phytate. Hahn et al. (2008) concluded that after the combined aqueous calcium phytate/calcium bicarbonate treatment, the chemical composition of the ink changes considerably, because minor and trace components are removed.

Huhsmann & Hähner's (2008) publication is required reading for any paper conservator involved in iron gall ink conservation, where the procedure for the treatment with calcium phytate/calcium bicarbonate is clearly and comprehensibly described, including step by step instructions, materials needed and related bibliography. Eleven distinct steps are described concerning preliminary testing, wetting and washing, introduction of the phytate and alkaline buffer, strengthening with gelatine solution, and drying under weight. The paper under treatment is sandwiched between two viscose mats and supported by a double screen during all wet treatments (Huhsmann and Hähner 2007), so that mechanical stresses and bleeding of the ink are minimized. The actual treatment comprises 2 steps, first the phytate treatment and then deacidification with calcium bicarbonate.

The use of magnesium and calcium hydroxide nanoparticles dispersed in alcohols for the simultaneous deacidification and iron gall ink stabilization was tested by Poggi et al. (2011; 2010) and Stefanis & Panayiotou (2010) with promising results. A key element reported by the first group of authors is the pH of paper after the treatment, which must be stabilized around 7.

7. Paper repairs

Restoration of mechanical damage and paper repairs can be accomplished with Japanese paper or paper pulp, depending on the nature and extent of the damage and the existing equipment (Johnson 1988). Japanese papers are thin, strong handmade papers, ideal for paper mending, and are discussed below in more detail.

7.1. Methods

With Japanese papers. The appropriate colour, thickness and type of the Japanese paper should be considered in advance. The dyeing of Japanese paper with various dyes in order to match the original paper is discussed by various authors (Grantham and Webber 2002; Norton 2002; Wills 2002). Tears are mended with a narrow strip of thin transparent Japanese paper (for example, Tengujo 7 – 11 g/m^2). Japanese paper should never be cut with scissors. In order to prepare strips, a ruler is used to press the desired width of paper, and the loose part of the Japanese paper is pulled until it is separated. For lacunae filling, the following procedure is recommended: the original is laid on the light-table, then a transparent polyester sheet (melinex) is laid on it, and on top of it the Japanese

paper is placed. The direction of the grain of the original paper and the Japanese paper must be paralleled. A refillable pen must be used, filled with a mixture of alcohol and water. The pen is used to trace the contour of the lacuna on the Japanese paper. The contour becomes wet and soft and the patch can be removed by pulling, and glued on the original with paste, methylcellulose (usually at a 4% consistency) or a mixture of them. The connection with the original is achieved with the fibrils protruding from the patch, with minimal overlapping. Sometimes, old or specially made western paper is used for infills. This can be cut with scissors, but should be bevelled at the edges in order to minimize bulking of the overlaps.

Mending on the light-table can be done on dry or wet paper, if wetting is possible. The dry procedure is faster, especially if a heated spatula is used for drying the adhesive, but the wet procedure has better results because differential paper shrinking is avoided. Commercially available mending tapes (of archival quality) with thermally or water activated adhesives (Pataki 2009) are also acceptable alternatives, especially when speed is required or moisture-sensitive objects are treated. The old method of silking, that is, using silk for mending and reinforcing paper is no longer regarded as acceptable (Reissland 1997; Waters 1981). Manual paper mending is discussed in detail by Jones (1978), McAusland (1978), McMullen (1978), Nordstrand (1987) and Johnson (1988).

With paper pulp. Paper pulp is added to the lacunae with a leafcasting machine (see below) for mass production or with a vacuum table for minor repairs. Keyes (1976) discusses a method for using pulp without sophisticated equipment.

7.2. Materials and adhesives

Papers. In paper conservation, various oriental papers, especially Japanese papers, are almost exclusively used. Japanese papers are made from long, strong fibres extracted from the inner bark of various indigenous Japanese plants (bast fibre, such as kozo, mitsumata and gampi), and their properties (mechanical strength, weight, pH, colour, fibre length, dimensional stability and resistance to ageing) are ideal for paper mending.

The history, the making, the properties and the types of Japanese papers are discussed by Hunter (1974), Webber & Thompson (1991) and Yang (1997). An alternative to Japanese papers, the handmade Chinese papers Xuan, which are also made from bast fibre, is proposed by Mullock (1995). Koestler et al. (1992) used EDX for the characterization (metal ions and trace elements content) of Japanese papers. The properties of traditional Japanese, Nepalese and Indian papers were studied by Suryawanshi & Agrawal (1996) and Suryawanshi et al. (1995). Collings & Milner (1978) published microphotographs of various oriental fibres, and presented the techniques used for the identification of the fibres used in oriental pulps. An important and extensive study for the revival of the western papermaking traditions, aiming at producing paper for conservation similar to the historic European paper, has been published by Barrett (1989).

Adhesives. The adhesives used in paper conservation will be discussed in more detail in the next chapter. For paper repairs, adhesives that can be used include methylcellulose, carboxymethylcellulose, starch paste or mixtures of them. Methylcellulose may have a lower bonding strength, but it is preferred because of its better resistance to both biodegradation and chemical degradation (Evetts et al. 1989; Seki et al. 2010; Strnadova and Durovic 1994).

Paper pulp. The pulp used for repairs can be prepared from Japanese paper, or from pure linen or cotton textile (unbleached, with no additives). Many

conservation workshops use bleached chemical pulp, which is commercially available for this purpose. Cernic Letnar and Vodopivec (1997a) recommend the addition of up to 30-40% of unbleached fibre to the leafcasting pulp, and advise caution regarding the use of dyed pulps because they may contain transition metal ions, such as Fe and Cu. The types of pulps, their preparation and their application in paper conservation are discussed by several authors (Bansa and Ishi 1999; Blunn and Petherbridge 1976; Cernic Letnar and Vodopivec 2004; Perkinson and Futernick 1977; Petherbridge 1987).

7.3. Equipment

Vacuum (suction) table (Cumming and Colbourne 1998; Futernick 1983; Vitale 1988; Weidner 1974). Nowadays, a commercially available piece of equipment, it can be used for local wet treatments and for filling losses of paper. Other uses, such as flattening, overall washing or spot stain removal, bleaching, Japanese paper backing, fixing, consolidation, drying etc. are discussed by Weidner (1974) and Vitale (1988). The vacuum table is an oblong box with large horizontal surface and small depth, which communicates with a vacuum pump. The top surface consists of a very fine plastic or stainless metal screen. The paper artefact is placed on the screen, supported by a permeable non woven polyester fabric, the suction is turned on and the pulp is poured on the damaged area. Maggen (1993) describes the construction and use of a small unit for treating philatelic materials.
Leafcasting machine (Alkalay 1987; Bansa 1990; Bansa and Ishi 1997; Bansa and Schoenung 1989; Blunn and Petherbridge 1976; Cernic Letnar and Vodopivec 2004; Johnson 1988; Leclerc et al. 1987; McIntyre 1987; Mowery 1991; Petherbridge 1987; Wouters et al. 1995). Developed in the Eastern Europe at the end of the 1950s, leafcasting is used for filling losses of paper with paper pulp. Leafcasting is not a mass method, but for artefacts that can withstand water, it can be much faster than the manual methods and produces better results. Advanced devices use computers for the calculation of the quantity and the colour of the pulp, so that the produced patches have the right thickness and colour (Bansa 1990; Mowery 1991). A leafcasting machine consists of an oblong relatively deep basin that can be emptied either by gravity or by use of a pump to an underlying tank. The bottom of the basin is actually a plastic or stainless metal screen. The paper artefact is placed on the screen, supported by a permeable non woven polyester fabric, and held in position by a grid placed on top of the artefact. Dilute pulp suspension is added to the basin and the pump is turned on. Paper fibres are screened and deposited in the areas of paper losses, while water passes through the screen and is collected in the tank. Nylon sheets are placed around the artefact in order to limit the deposition of the fibres to the desired areas only. For a more detailed description of the function and design of the leafcasting machine, see Blunn & Petherbridge (1976) and Johnson (1988).
Light-table: A light-table is essentially a table with a translucent upper surface, which is lit from underneath by an array of -usually- fluorescent light tubes (UV component must be cut off with filters). Paper mending with Japanese paper is done on the light-table.

8. Consolidation/strengthening

8.1. Methods

Lamination. (Cernic Letnar and Vodopivec 1997b; Wilson and Parks 1983). Weak, mouldy and brittle paper can be reinforced by pasting on one or both sides thin Japanese tissue (such as Tengujo 7 – 11 g/m^2) with methylcellulose and/or paste (Suryawanshi et al. 1996).

In a variant of lamination, Japanese tissue impregnated with a thermoplastic polymer can be hot-pressed or adhered by a heated spatula on both sides of the document (Clare and Marsh 1979). Filmoplast R, which is Japanese paper impregnated with acrylic polymer, is an example of such heat-set tissue (Bansa and Ishi 1997; Cernic Letnar and Vodopivec 1997b). Lamination has been extensively used in the past and at a mass scale, with semiautomatic machines (Hummel Jr and Barrow 1956; Nixon 1949). Regrettably, the polymers used (mainly cellulose acetate) were not stable, resulting in the destruction of the originals (Aubier et al. 1996; Waters 1981). Lamination with heat-set tissue is recommended when water soluble adhesives cannot be used due to inks and media sensitivity. Suryawanshi et al. (1996) have published an evaluation study of the materials (adhesives and papers) used for the lamination of old documents. Santos et al. (2015) synthesized sheets of bacterial cellulose and studied their use as reinforcing material for the lamination of degraded paper.

Sizing/impregnation (Anders 2013; Carrapella et al. 1990; Evetts et al. 1989; Hanus 1994; Hummert et al. 2013; Jancovicova et al. 2012; Kolbe 2004; Seki et al. 2010; Seki et al. 2005). Paper can be strengthened by impregnating it with various materials, which at the same time render it hydrophobic (sizing). Resizing is necessary after aqueous treatments, because the original size of paper may be water-soluble and thus removed by the treatment. Various glues can be used, such as methylcellulose, carboxymethylcellulose and gelatine dissolved in water or various organic solvents (such as methanol or ethanol). They can be applied by immersion, by brushing with a soft brush or even by spraying (Hummert et al. 2013). Sizing can be combined with deacidification, if a deacidification agent is added in the sizing solution (Jancovicova et al. 2012; Sundholm and Tahvanainen 2003a, b, 2004). Guerra et al. (1995) recommend a 30-45 minutes treatment with methylcellulose (0.5-0.75%) and calcium hydroxide (semi-saturated, 0.08% w/w) solution. A non-aqueous combination of strengthening with cellulose ethers (methylcellulose, carboxymethylcellulose, hydroxypropylcellulose and ethyl cellulose) and deacidification with magnesium carbonate has also been developed (Seki et al. 2010; Seki et al. 2005).

Paper Splitting (Anders 2013; Bansa and Ishi 1997; Brückle and Dambrogio 2000; Galinsky and Haberditzl 2004; Gast 1993; Liers et al. 1996; Wachter et al. 1996). Mainly developed and utilized in Germany, paper splitting is used for strengthening brittle, weak and mouldy paper. It entails the splitting of a paper sheet thickness-wise, the insertion of a healthy paper core and the reassembling of the paper sheet. Splitting is achieved by temporarily adhering two strong paper sheets (supports) on either side of the original with gelatine and pulling them apart with a fast and firm motion, so that each of the two surfaces of the original remains adhered on one of the two supports. Humidification of paper is important to reduce z-dimension tensile strength and facilitate splitting in two plies. A healthy paper core is inserted between the two surfaces of the original and glued in place with starch paste, methylcellulose or acrylic polymer emulsions. The

support papers are removed by use of a proteolytic enzyme or hot water. The adhesive and the paper core contain significant quantity of calcium and magnesium carbonate, which act as alkaline reserve. There are ethical reservations against paper splitting because it poses serious risks for the paper artefacts, alters the thickness, the weight and the bending behaviour of paper and its reversibility (or more correctly, retreatability) is doubtful (Gast 1993).

Leafcasting machine (Bansa and Ishi 1997). One or two thin paper sheets are cast and embedded while wet onto the paper artefact.

Williams (1981) attempted to restore the folding endurance of deacidified old paper by adding sorbitol, glycerin and a wet strength resin. Unfortunately, the impressive initial increase of folding endurance was lost after several days of artificial ageing. Li et al. (2014) used electrospinning to produce polyvinylidene fluoride membranes directly on paper surfaces with a significant strengthening effect.

8.2. Mass strengthening methods

In the chapter 6.1.5 on mass deacidification, two methods combining mass deacidification and mass strengthening were presented, namely the Bückeburg and the Vienna methods. The use of alkoxisilanes was also briefly discussed, which have been postulated to be applicable for both paper mass strengthening and deacidification.

Apart from these methods, the following methods for mass strengthening are discussed in the literature:

Mechanized Paper Splitting (Anders 2013; Bansa and Ishi 1997; Brückle and Dambrogio 2000; Galinsky and Haberditzl 2004; Gast 1993; Liers et al. 1996; Wächter 1987b; Wachter et al. 1996; Wächter et al. 1998). Manual paper splitting evolved to a fully mechanized continuous process, which minimizes risks due to wrong handling. In the German Library in Leipzig, the paper splitting machine has been in operation since 1994.

*Graft-copolymerization proce*ss (Anders 2013; Burstall et al. 1986; Butler et al. 1989; Carter 1996a; Clements 1987; Davis et al. 1981; Margutti et al. 2001; Princi et al. 2007; Shenton 2006). The process was developed by the British Library in the 1980s as a pilot project, but was stopped in the 1990s (Anders 2013; Shenton 2006). Strengthening was achieved by the polymerization of monomers inserted into paper fibres. A molecule of the monomer was initially bonded chemically to the cellulose macromolecule. The addition of more monomers followed, resulting in the production of polymeric chains chemically grafted on cellulose. The method could be applied to a limited number of books at a time (5-10), resulting in substantial increase in paper strength without causing any unwanted side-effects to inks and bindings. Monomer polymerization was achieved by exposing the books after their impregnation to γ-radiation for 13-16 hours. Satisfactory results were produced by using a 5:1 mixture of ethyl acrylate (CH_3=C–$COOCH_2CH_3$) and methyl methacrylate (CH_3=C(CH_3)–$COOCH_3$). The retention of significant amounts of residual monomers, which are toxic, is considered to be an important drawback of the method (Anders 2013). Reported disadvantages of the method are also the minor depolymerisation of cellulose due to γ-irradiation and the 10-20% weight increase (Baty et al. 2010).

Gas-phase strengthening with parylene (Anders 2013; Bansa and Ishi 1997; Baty et al. 2010; Carrapella et al. 1990; Carter 1996a; Dobroussina et al. 1996; Grattan and Bilz 1991; Humphrey 1990; Shiah et al. 2006). The term “parylene” is used to describe the polycrystalline polymers of xylylene. Strengthening is achieved by

controlled gas phase deposition of the polymer on paper. The method increases substantially the strength of paper and does not have any immediate adverse side effects. It has been claimed that considering the chemical stability and resistance to ageing of parylene, it may be the ideal consolidation material (Humphrey 1990).

By studying the stability of parylene with the application of thermal accelerated ageing, Grattan and Bilz (1991) concluded that specifically for parylene-C, the use of antioxidants is not required, since the useful life of the polymer was estimated to be around 130,000 years. Dobroussina et al. (1996) studied the biostability of parylene coated paper. Their results indicate that the resistance of coated paper against microorganisms increased up to 74%, and that paper was infected only at imperfections and cracks of the coating. Bansa and Ishi (1997), in a comparative study of various paper strengthening methods (splitting, leafcasting, lamination with a heatset acrylic and parylene coating), concluded that they all have acceptable results and are recommended for specific applications. Concerning the use of parylene, it gave excellent results with paper from chemical pulp, but it enhanced the decay of groundwood paper during accelerated ageing. Paper strengthening with parylene is irreversible and expensive (Anders 2013).

8.3. Adhesives and consolidants

Adhesives and consolidants include a multitude of polymers, which are mainly used as adhesives for paper repairs, lamination and paper splitting, as fixatives for the protection of fugitive inks during aqueous treatments, as coatings, and as strengthening agents for resizing and consolidation. Their desirable properties greatly depend on the specific application, but they must share the following common attributes: sufficient bonding strength, chemical inactivity towards the substrate, resistance against ageing, colour stability, and reversibility. Here, reversibility means that they should be easily removed, even after many years after their original application, without the need to resort to extreme conditions or use toxic solvents and complicated procedures. The book “Materials for Conservation: Organic Consolidants, Adhesives and Coatings”, by Horie (2010), is a basic resource on all the aspects of polymer usage in conservation. The fundamentals of adhesion are discussed by Allen (1984).

8.3.1. Natural polymers

The main natural polymers used as adhesives and consolidants in paper conservation include various forms of starch and gelatine. Warm glue was a form of impure gelatine produced by boiling in water various animal parts such as hides, hooves or bones (Horie 2010). It was used for paper sizing (Carrapella et al. 1990; Casoli et al. 2014), imparting according to various researchers improved resistance to ageing, apart from improved strength (Barrett 2011; Barrett et al. 1996; Barrett 1989; Hunter 1974; Kolbe 2004; Waterhouse and Barrett 1991). It was also used by itself or in combination with starch in bookbinding. Chitosan was tested for the improvement of the durability of rosin-alum sized historic paper by Basta (2003) with good results. Another natural polysaccharide, funori, is produced from an algae and has been used for many centuries by East Asia paper restorers (Horie 2010; Masson and Ritter 2004). Natural polymers are still in use in paper conservation, but are being replaced in many applications by synthetic polymers due to their low biostability and versatility.

8.3.2. Cellulose ethers

They are the main choice in most paper conservation treatments, such as paper mending, strengthening and resizing, in aqueous solutions ranging from 0.5 to 4%. They are produced from pure wood or cotton cellulose, which is transformed into alkali cellulose and then reacts with various alkylating agents. Horie (2010) discusses their properties in detail. The main cellulose ethers used in paper conservation with their usual commercial names are the following (Feller and Wilt 1990; Horie 2010; Strnadova and Durovic 1994; Suryawanshi et al. 1996; Vodopivec and Letnar 1990):

- Methylcellulose (MC), commercial name: Glutofix 100, glutin, MC 400, MC 40
- Methylhydroxyethylcellulose (MHEC), commercial name: Tylose MH 300
- Carboxymethylcellulose (CMC), as a Na^+ salt, commercial name: Tylose C300
- Hydroxypropylcellulose (HPC), commercial name: Klucel M, G

Strnadova and Durovic (1994) studied the use of cellulose ethers for paper strengthening. They concluded that, with the exception of carboxymethylcellulose which was not recommended, the three other ethers exhibited good biostability, were quite stable against accelerated ageing, and very effective as consolidating agents, since they substantially raised the strength of the paper they were applied to. They recommended the use of methylcellulose, which was slightly superior to the others. Carboxymethylcellulose was not recommended, because after accelerated ageing, the whiteness of the impregnated samples and its strengthening effect were considerably decreased. Seki et al. (2010) also recommended methylcellulose. Several other researchers have published studies on the use of cellulose ethers in paper conservation (Baker 1984; Bonet et al. 2007; Feller and Wilt 1990; Letnar et al. 2006; Seki et al. 2005; Suryawanshi et al. 1996; Vodopivec and Letnar 1990; Zervos 2007a). Bicchieri and Mucci (1996) studied the use of Klucel G in alcoholic solutions as a fixative for dyes and pigments.

8.3.3. Other synthetic polymers

Various synthetic polymers have been used in paper conservation, either as adhesives or consolidants, usually in aqueous dispersions. The most important of them are the following (Baer and Indictor 1977; Baer et al. 1972; Clare and Marsh 1979; Durovic et al. 1991; Nada et al. 1999; Verdu et al. 1984; Vodopivec and Letnar 1990):

- aqueous dispersions of polyvinyl acetate (PVAC) or copolymers of vinyl acetate and alkylesters of acrylic or methacrylic acid
- aqueous dispersions of polymers and copolymers of alkylesters of acrylic or methacrylic acid (Texicryl 13-002, Primal AC34)
- polyvinyl alcohol (PVal)
- paraloid (or acryloid) B 72 (polyacrylic resin)
- polyethylene (PE)
- regnal
- parylene (see chapter 8.2)
- soluble nylon

The behavior of these materials depends on many factors, such as their exact chemical composition, the presence of plasticizers and other additives, their initial

pH, etc. Acrylic polymers are considered more stable than PVAC. Aubier et al. (1996) present the problems caused by unstable polymers (mainly cellulose diacetate) and propose possible conservation treatments.

Baer et al. (1972) and Baer and Indictor (1977) studied the use of various synthetic polymers, such as PVal, soluble nylon, Regnal and PVAC for paper strengthening. The use of synthetic polymers (texicryl, an acrylic ester co-polymer, polyvinyl acetate and carboxymethylcellulose) in field work for the lamination of Islamic illuminated manuscripts is presented by Clare and Marsh (1979). Vodopivec and Letnar (1990) concluded that paraloid and PVal are not suitable for generalized use in paper conservation, and recommend the use of paraloid as fixative for water soluble inks and of methylcellulose as adhesive and consolidant. Durovic et al. (1991) studied the resistance of various commercial products (PVAC and acrylic dispersions) against ageing and recommend the use of a methylmethacrylate and butylacrylate copolymer (Sokrat 6492). In this study, various theoretical aspects of polymer properties and degradation are discussed.

PVal was recommended by Bicchieri et al. (1993) as an efficient resizing agent. Bicchieri and Mucci (1996) recommended the use of PVal and Klucel G as fixatives for inks and pigments during aqueous conservation interventions. They suggested that they protect paper during accelerated ageing.

Cernic Letnar and Vodopivec (1997b) studied the use of polyethylene (PE) in combination with Japanese paper and of Filmoplast-R (a commercially available lamination material, consisting of Japanese paper coated with an acrylic polymer) for the lamination of paper. Accelerated ageing results indicated that Filmoplast-R is a suitable lamination material, while polyethylene is unstable and prone to oxidation.

The use of dispersions of various concentrations in water of PVAC/PV versatate copolymers for the strengthening of paper was studied by Nada et al. (1999). After thermal accelerated ageing, the mechanical strength of the treated samples remained considerably higher than those of the untreated ones, especially after washing, heat pressing after the polymer application and the addition of plasticizer (3% di-octyl phthalate). Nada et al. (2000) tested polyethylene glycol and polyvinyl alcohol for the strengthening of paper and concluded that treatment of paper with a mixture of them improved both mechanical strength and brightness stability.

Roche and Dessennes (2002) tested gelatine, sturgeon glue, polyvinyl alcohol, methylhydroxyethylcellulose and paraloid B72 for the consolidation of flaking gouache on Japanese paper. Basta (2004) recommends the use of 0.5-0.75% PVal and 2% borax for the strengthening and chemical stabilization of decayed papers. The local strengthening of mould damaged manuscripts with a 2 % Klucel G in ethanol as adhesive and re-sizing agent is discussed by Martin (2011).

9. Conclusions

Paper conservation interventions have been classified here under the following categories, which also represent the generic steps of a paper conservation treatment:

1. Preparation of the intervention
2. Disinfestation and disinfection/sterilization
3. Surface/dry cleaning
4. Wet cleaning
5. Chemical stabilization

6. Paper repairs
7. Consolidation/strengthening
8. Treatment documentation

The main methods associated with each step are discussed and the most important publications concerning them are presented in the body of this review paper. The review showed that there exists a multitude of methods for treating almost all kinds of paper damage at a laboratory scale. On the other hand, mass methods exist mainly for deacidification and strengthening.

The effectiveness of the existing methods varies. For example, most laboratory deacidification methods, according to the publications that have evaluated them, seem to be very effective. Contrariwise, almost all mass deacidification processes seem to have several issues, such as immediate reduction of the strength properties of paper, smudging and fading of inks and colorants, distortion of books, white deposits and persistent remaining odours, while others do not deposit adequate alkaline reserve. Nevertheless, mass conservation processes are evolving, and there exist several commercially available processes nowadays for an organization to choose from. Concerning disinfection, there seem to be open issues, especially in regard to treating fungi infected paper, and health dangers associated with the use of many disinfectants.

The research devoted to paper conservation is impressive. Many new promising methods have emerged, especially in the field of deacidification and oxidation inhibition. The relatively recent establishment and full documentation of a method for treating iron gall ink corrosion is an important breakthrough. The use of nanoparticles technology is very actively researched, and many new chemicals are tested in order to expand the choices in the field of antioxidants. Nevertheless, various methods, especially those with a complex chemistry background (such as those presented in chapters 6.2.1 and 6.2.2), may look a little intimidating to the average conservator and may have not been sufficiently well documented to be widely implemented.

The penetration of the methods presented above into everyday conservation practice is largely unknown. We will address this issue in a follow-up paper, where the results of an international survey on paper conservation methods will be presented[2].

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