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Steel versus Paper

The Conservation of a Piece of Modern Art Consisting of a Rust Print on Paper

'Empreinte de trois plaques de fer rouillées' (Print of three rusted iron plates) was produced in the 1970's by the artist Bernard Pagès according to a specific protocol: steel plates were etched with a hydrochloric acid solution then pressed onto paper to create rust prints. Although contemporary, this work of art already shows serious damage caused by the presence of iron in the paper. Various conservation treatment possibilities were explored to preserve the artefact. Replicas were made and used to evaluate the different options. The application of the calcium phytate treatment, initially developed for iron gall ink corroded manuscripts, was investigated and it turned out to be efficient in preventing degradation of the replicas. The addition of alcohol to the treatment solutions was also considered, but was eventually not tested, as it tends to decrease the treatment effectiveness. It was finally decided to use pure aqueous solutions and to float the artefact during the treatment. The treatment was undertaken twice without inducing any visual changes to the artefact. Some free iron (II) still remained in the paper after treatment but to a much lesser extent than before treatment. This suggests that the intervention was at least partially effective. As the artefact required consolidation and lining, the capabilities of several commonly used adhesives that change paper sensitivity to water were evaluated and compared. Gelatine was finally chosen for consolidation as it renders the paper the most hydrophobic compared with starch, hydroxypropylcellulose or methylcellulose. After lining, the artefact was mounted in a frame specially designed for safe display.

Stahl gegen Papier: Restaurierung eines modernen Kunstwerks von Rost auf Papier

„Empreinte de trois plaques de fer rouillées“ (Druck dreier rostiger Eisenplatten) wurde in den 1970er Jahren von dem Künstler Bernard Pagès gemäß eines fest definierten Protokolls angefertigt: Stahlplatten wurden mit Salzsäure geätzt und dann auf Papier gepreßt, um rostige Drucke zu erzeugen. Obwohl es sich um zeitgenössische Kunst handelt, zeigen die Drucke bereits schwere durch das Eisen im Papier verursachte Schäden. Verschiedene Behandlungsmethoden wurden an Repliken untersucht, um die Drucke zu erhalten. Calcium-Phytat-Behandlung, zunächst für die Behandlung eisengallusgeschädigter Manuskripte entwickelt, wurde getestet und für wirksam befunden. Zusatz von Ethanol in den Behandlungslösungen wurde ebenfalls erwogen, am Ende jedoch nicht geprüft, da dies die Effektivität der Behandlung vermindert. Die besten Resultate ergaben eine wäßrige Behandlung, bei der das Objekt auf der Oberfläche reinen Wassers schwimmend gewässert wird. Diese Behandlungsmethode wurde zwei Mal ohne sichtbare Veränderungen am Objekt durchgeführt. Danach konnten zwar noch geringe Mengen freier Eisen-II-Ionen nachgewiesen werden, jedoch in deutlich geringeren Mengen als davor. Dies deutete darauf hin, daß die Behandlung zumindest teilweise effektiv war. Da die Objekte im Anschluß konsolidiert und kaschiert werden mußten, wurden mehrere allgemein gebräuchliche Klebstoffe und deren Fähigkeit, Wasser abzustößen, getestet. Am Ende wurde Gelatine zur Konsolidierung verwendet, da es das Papier im Vergleich zu Stärke, Hydroxypropylcellulose und Methylcellulose leicht hydrophob werden läßt. Die Objekte wurden nach der Kaschierung in Spezialrahmen montiert, die eine sichere Ausstellung ermöglichen.

The experimentation with new ideas and techniques in contemporary art can lead to substantial conservation issues in the near future, especially when the artist tries to fuse two incompatible materials into one object. This is the case with the 'Print of three rusted iron plates', created by the French contemporary artist Bernard Pagès and now in the Museum of Modern Art, Saint Etienne, France. Although contemporary, this print already shows serious damage caused by the presence of iron in the paper. It was so brittle that it could no longer be handled nor be displayed. This type of artefact raises fundamental questions for paper conservators: its degradation is extremely rapid and inherent to the combination of its constituents (iron compounds and paper) that obviously cannot be separated. It also necessitates investigation into the possibilities of using available chemical treatments in order to limit the degradation mechanisms while keeping in mind that the visual appearance should remain unchanged.

Historical Background

'Print of three rusted iron plates' is one of the series of prints and drawings created in the 1970's by Bernard Pagès while he was

working under the influence of the avant-garde, Supports/Surfaces' group (Abadie 1999; Exhibition catalogue, 1991, 2002). As an established sculptor, he worked with raw materials and tried to escape from personal expression by using printing methods that place the object in the centre of the creation process. These methods consisted of etching a hydrochloric acid solution on steel plates in order to encourage the formation of rust. These plates were then pressed on the paper, producing prints of rust. Finally, the artwork was completed with graphite pencil and black ink lines that emphasised the borders of the central plate (Fig 1).

Description of the Artefact

From a chemical point of view, the work is in fact a thick rust layer embedded in a contemporary wove paper, commonly known as 'Vélin d'Arches' and manufactured by Canson. One edge features a watermark, 'Dessin-Ja-Arches-France'. The paper is now very brittle and fractured in many places. Two pieces are missing. The sharpness of the edges shows that the fractures are due to loss of paper flexibility. Raking light observation revealed undulations. Unprinted areas are light brown and hig-

hly hydrophobic. The pH of the paper, measured on the verso side with a microelectrode, was 3. Diffuse halos of brown compounds were visible around the corrosion deposits. Using the batho-phenanthroline test (Neevel and Reissland 2005), the presence of iron (II) was confirmed, not only on the print itself but also on the verso of the paper.

The combination of the use of acidic compounds during the printing process, together with the presence of iron (II) in the paper, account for the poor current condition of the artefact. However, it was noticed that similar prints of the same period that were bound together in a book were well preserved. Also the storage conditions of the artefact during the last 30 years, although not documented, were probably important damaging factors.

Analogies with Iron Gall Ink Corrosion

The conservation issues of Bernard Pagès' print are very similar to those of iron gall ink corroded papers. In both cases, the presence of iron and acids in the paper promotes cellulose oxidation and hydrolysis. The chemical treatments that were developed to inhibit iron gall ink corrosion were therefore investigated to determine to which extent they could be adapted to this case.

Of all the anti-oxidant and de-acidification possibilities, the calcium-phytate/calcium-bicarbonate [1] treatment (hereafter named the Calcium Phytate treatment) remains the most investigated method. It has been tested over several years (Botti et al 2005; Neevel 1995; Reissland and Goot 1999; Neevel 2001; Kolar et al 2003; Wagner et Bulska 2004; Zappala and Stefani 2005; Kolar et al 2007; Rouchon et al 2008). Its effectiveness and safety

tes the immersion of the document for a period of more than 30 minutes, there are risks of several side effects occurring. Firstly, a modification of the visual appearance may occur due to the solvent, the ink may bleed and the paper may become lighter. These risks, that are unacceptable in the case of a work of art, should be fully understood prior to any implementation.

Secondly, the immersion of very fragile artefacts in aqueous solutions induces strong mechanical stresses. The highly corroded areas are very hydrophobic whereas well-preserved areas are more hydrophilic. When water penetrates the paper unevenly, tensions have been observed between the hydrophobic and hydrophilic zones, leading to the formation of cracks and holes in the most fragile areas (Reissland and de Groot 1999).

Thirdly, the chemical reactant present in solution may precipitate due to the formation of small particles of calcium-phytate salt. This deposit may constitute a kind of 'anti-oxidant' reserve, thus improving the future preservation of the artefact. Co-precipitation of the iron-phytate complex may also be brought about. If so, it is necessary to check that these particles are invisible to the naked eye or with examination under a stereomicroscope.

The Chosen Approach

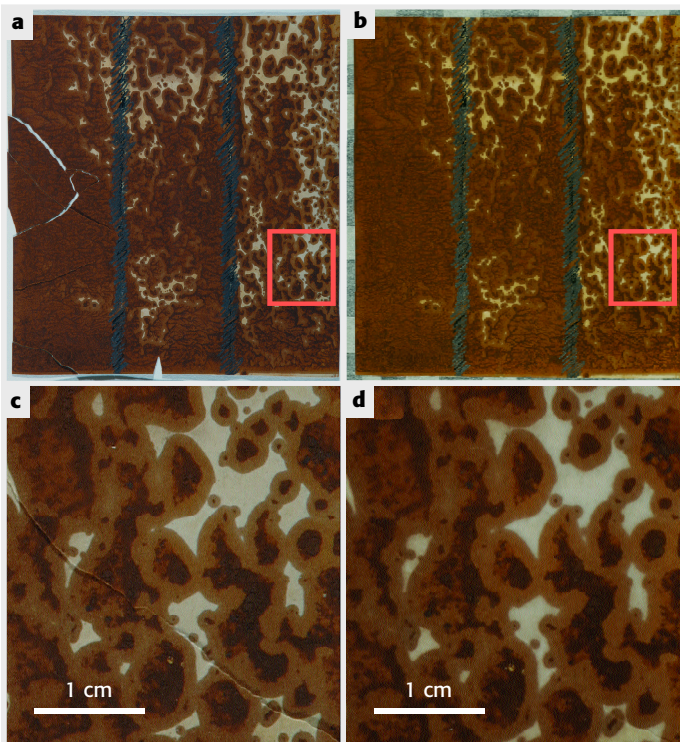
Considering the high degradation rate of the artefact and the loss of its mechanical properties within the short period of three decades, it was difficult to limit intervention to preventive conservation. It was thus decided to explore the possibilities of applying a full conservation treatment. As the artefact was very fragile and covered with a powdering rust deposit, it could certainly not withstand immersion in a series of treating solutions. Other modes of application, such as flotation, were considered. As the paper was very hydrophobic, the possibility of adding alcohol to the treating solutions in order to increase penetration of reactive components into the paper was tested. All these tests were performed on replicas and the effectiveness of the different applications was visually evaluated after artificial ageing.

As physical consolidation of the object was necessary, a suitable adhesive had to be selected. In order to protect the paper from future hydrolysis the adhesive should render the paper hydrophobic. The sizing capacities of several adhesives were evaluated in order to select the most appropriate.

Calcium Phytate Treatment: Evaluation of Replicas

Preliminary Observations

The artist was contacted and he kindly shared his memories regarding his creative process. The implementation of the Calcium Phytate treatment was carried out on replicas that were prepared according to his instructions. For making the first set of replicas, a test paper was required that was as close as possible to the original; 'Johannot' from Canson was the most suitable. A 1M hypochlorite solution and a steel plate were used. The resulting prints presented similar halos to the original (Fig 2) and the rust deposits had a similar appearance and composition to that of the artefact [3]. A Calcium Phytate treatment was then carried out on the replica using a 15 minute flotation on each



1 'Empreinte de trois plaques de fer rouillées' (Print of three rusted iron plates), inv. n 92.9.256. Size: 26,5 x 24 cm: General view before (a) and after intervention (b); detail before (c) and after intervention (d).

solution. The visual examination of the treated object under a stereomicroscope showed no white deposit on the replicas. However, Scanning Electron Microscopy revealed that reactants were deposited on the paper as small particles of calcium (probably calcium carbonate), approximately 5 microns in size. A diffuse deposit of phosphorus on the paper fibres was also indicated, suggesting a diffuse presence of phytate.

After the treatment, the bathophenanthroline test was found to be negative on the recto and verso of the sheet. However, when applied to the core of the paper, it was positive, meaning that the phytate could not reach all the free iron (II). This was probably due to the strong hydrophobic property of the paper that would have seriously limited the penetration of reactants during the treatment.

Effectiveness of the Treatment/ Impact of Alcohol Additive

In order to encourage the penetration of the active reagents into the paper, the addition of alcohol to the treatment solutions was investigated. Absorption measurements [4] were carried out on the replicas with different solutions of alcohol and water in order to determine the appropriate alcohol/water ratio. These measurements (Fig 3) showed that the minimum percentage of ethanol to maximize the solution absorption was 40 %.

As the impact of the ethanol addition on the treatment effectiveness was still to be evaluated, a second set of replicas was produced. Using the hydrophobic *Johannoti* paper, it turned out to be impossible to achieve a comparable and reproducible replica. However, the uniformity of the sample set was an important determinant factor for the comparison of the different treatments. Finally, a hydrophilic laboratory paper was found (Whatmann 2001) that gave regular and reproducible rust deposits.

The quantity of rust deposited on the paper was controlled by weight measurements before and after the printing process: it was the same for all replicas.

All replicas were pre-aged for 7 days at 85 °C and 65 % RH then divided into two sections; the right section to be treated and the left section remaining untreated. Each section was then divi-

ded into three sub-sections. The first was the control (V0) while the other two were artificially aged for 12 days (V1) and 20 days (V2) at 85 °C and 65 % RH respectively. After ageing, all the samples were reassembled (Fig 4). The treatment consisted of floating the sample on pure water for 5 minutes, then on a calcium phytate solution (pH between 5 and 5.5) for 15 minutes, and finally on a calcium carbonate solution for 15 minutes. Two options were tested: with the first, '100 % aqueous', the solutions were prepared in water only. In the second, '60 % aqueous', the solutions were diluted with 40 % v/v of ethanol.

This empirical procedure illustrates the effectiveness of the phytate treatment in limiting the paper degradation, even when the samples were not immersed but floated. The appearance and the mechanical properties of the treated area remained unchanged after artificial ageing, whereas drastic changes were observed on the non-treated areas that became dark and brittle. Moreover, the bathophenanthroline test, performed on all the samples, turned out to be negative on all the treated samples, even after artificial ageing.

However, a slight but significant darkening of the paper was observed after 20 days of artificial ageing on the sample treated with the alcohol addition (V2 in Fig 4), suggesting a lower effectiveness. Time being limited further investigations and being unsure of the validity of the addition of alcohol, it was decided to opt for a 100 % aqueous treatment.

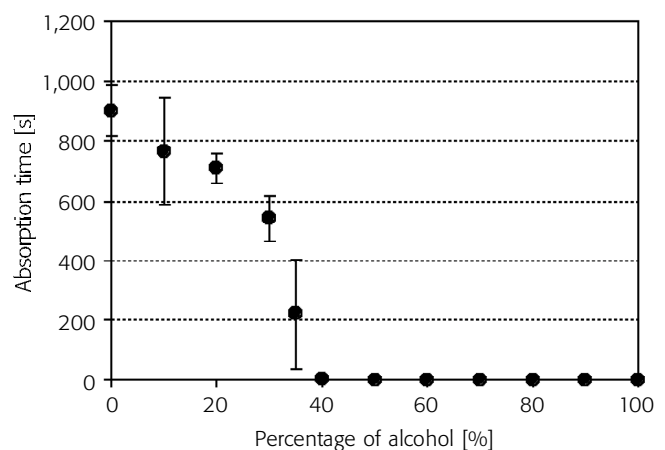
Evaluation of the Adhesives Used for the Physical Consolidation of the Artefact

General Considerations

The work of art consists of oxidised iron products and acids that have impregnated the cellulose substrate. It is expected to be sensitive to climatic changes due to mechanical and chemical factors. Firstly, as the paper loses its mechanical properties, eventual dimension variations due to humidity changes may create new fractures. Secondly, the exposure to high humidity conditions or condensation may promote hydrolysis mechanisms. The calcium carbonate de-acidification remains a gentle



2 Replicas of the artefact using a Johannot paper.



3 Absorption time of a 1 l drop of water/alcohol mixture against the percentage of water. The values correspond to the average of five measurements. Error bars correspond to standard deviations.

treatment. It helps to decrease acidic hydrolysis mechanisms, but in the case of a very acidic paper such as this, reaching an alkaline pH after treatment cannot be guaranteed. The choice of an adhesive that limits the interaction with water, and consequently the paper hydrolysis, would appear to be a wise additional precaution. Thus four adhesives that are commonly used on manuscripts were chosen for comparison.

Wheat starch (ZinShofu: Atlantis), here called 'starch', was first considered [5]. It is highly appreciated by paper conservators for its high adhesion, harmlessness and reversibility. However it is also mentioned (Hofmann et al 2004) for its ability to cause iron migration and halos during its application to iron gall ink manuscripts. As the artefact was to be treated in an aqueous solution for at least 30 minutes, it was to be expected that all the products that were susceptible to migration would be dissolved in the solution. This migration risk was considered to be negligible.

Secondly and thirdly, the methylcellulose, Tylose (Tylose: ShinEtsu Tylose), and the hydroxypropylcellulose, Klucel (Klucel G: Hercules International), were both selected as they form transparent and reversible films. Both are easily prepared and stored in a ready-to-use form [6] and offer complementary properties depending on the solvent to be used. Being soluble in polar solvents, Klucel is preferred for use on water sensitive objects. However, its adhesion remains limited in comparison to the other adhesives.

A bovine gelatine (225LH30: Rousselot) was the last adhesive to be chosen [7]. It is largely used for paper sizing and for the consolidation of friable pigments. It is also recommended as a satisfactory product for the consolidation of iron gall ink manuscripts because of its ability to fix iron ions (Kolbe 2004, Nguyen 2005).

Attention was first focussed on the evaluation of the capacity of these four adhesives to affect water absorption in a laboratory model paper, the Whatman no 1. Paper sheets were impregnated

with the adhesives and their capacity to absorb vapour or liquid water was evaluated. Each sample corresponds to six sheets, each 5 cm large and 10 cm long. The adhesive concentration corresponds to that commonly used for lining purposes, ranging from 20 to 50 g/L (2 to 5 % w/v) depending upon the adhesive.

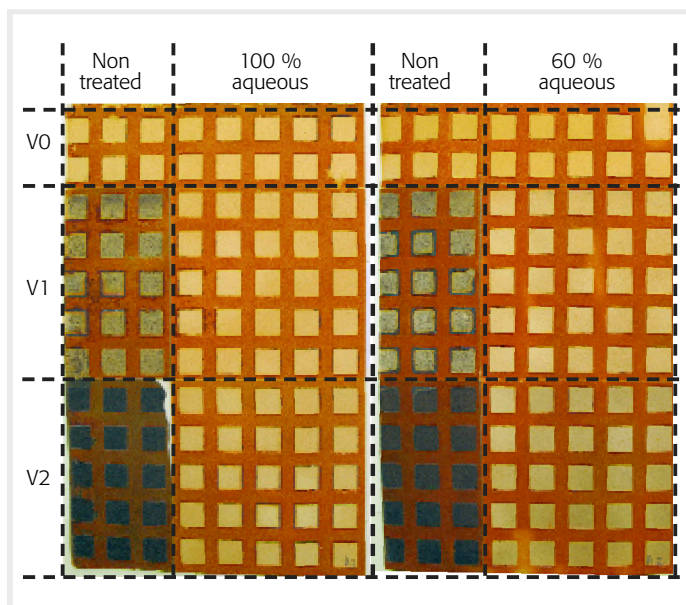
The percentage of adhesive deposited on the paper was estimated by weighing the sample before and after application [8]. Despite the fact that the adhesive application was performed manually, a linear correlation was achieved between the gelatine solution concentration and the amount of gelatine deposited on the paper (Fig 5), indicating that the sample preparation was satisfactorily reproducible. However, in the case of the other adhesives, this correlation was not found, probably because the adhesives were less liquid and more difficult to apply. Based on this experience, samples with the same content of adhesives were subsequently selected. Liquid and water vapour absorption measurements were undertaken after determining the adhesive concentrations in the paper.

In a second run, some empirical testing was performed on the replicas to check if the adhesives were suitable for lining. Some of the lined replicas were also artificially aged in order to compare the evolution of the consolidation at least visually.

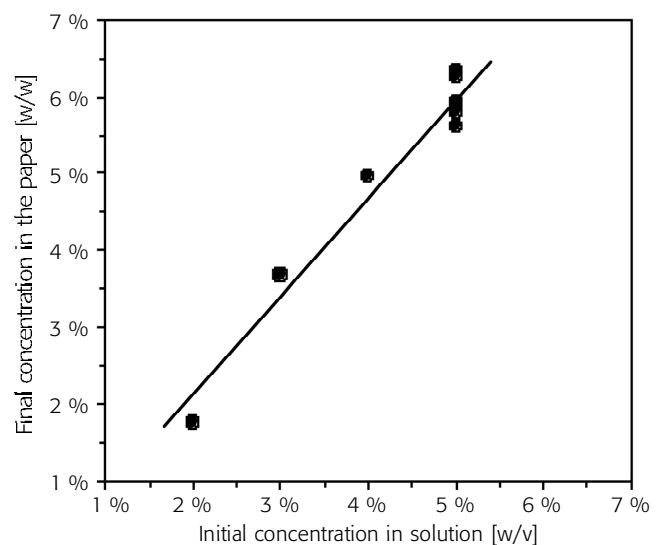
Water Vapour Absorption Measurements

The water vapour absorption capacities of the sized papers were evaluated by measuring their loss or gain of mass when exposed to varying conditions of humidity at the same temperature (23 °C). The samples were exposed successively to 85, 50, 23, 50 and 85 % RH, each humidity conditioning lasting for a minimum of 4 hours. The percentage of absorbed water was then determined relative to the dry mass of the sample [9].

Even though only slight differences were observed from one adhesive to another, it still seemed necessary to make more measurements in order to check their significance. Thus four sets of measurements were performed. For the first two, the humidifi-



4 Visual aspect of treated replicas: V0: no ageing after treatment; V1: 12 days of ageing after treatment; V2: 20 days of ageing after treatment.



5 The gelatine content in the paper against the initial concentration in solution. The amount of gelatine impregnated in the paper is linearly correlated to the initial concentration in solution.

cation chambers were prepared manually using saturated salt solutions in the bottom of closed vessels [10]. The relative humidity reached at equilibrium depended on the chemical nature of the salt. The last two sets of measurements were conducted semi-automatically using a prototype apparatus [11] capable of following in situ the mass variation of paper sheets exposed to controlled humidity conditions.

All measurements gave comparable values but the hierarchy between the adhesives was not exactly reproducible, indicating that the water uptake (if any) was too small to be accurately measured by this methodology.

The average values of these four sets of measurements were considered and compared with the measurements performed on the unsized areas of paper (Fig 6). The error bars that are represented in Fig 6 correspond to standard deviation only and are thus underestimated. Keeping this in mind, it can only be concluded that the water vapour absorption capacities are very close for all the paper samples, indicating that the size does not drastically influence the water content of the paper when exposed to humidity.

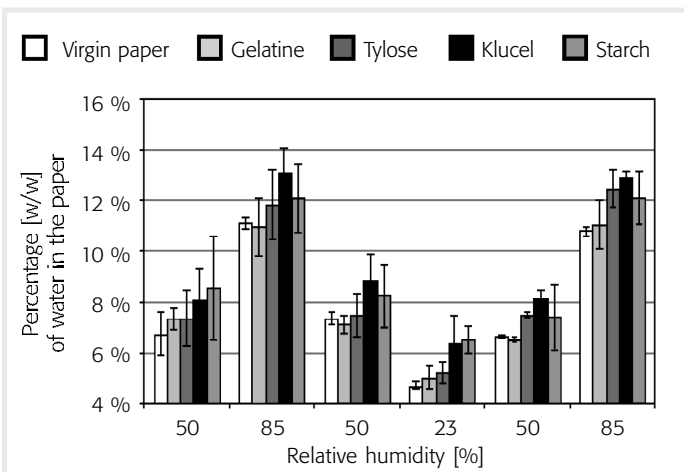
Liquid Water Absorption Measurements

As relative humidity and temperature fluctuations may cause water condensation on paper even well below 100 % RH, it was thought relevant to evaluate the liquid water absorption capacity of the paper after the application of an adhesive. This was performed with two different methods. The first consisted of measuring the time that was necessary for a 1 ml drop of water to penetrate the paper. This was carried out on a large set of samples impregnated with various amounts of adhesives. It showed (Fig 7) that the Tylose and starch impregnated papers remained very absorbent on both sides, regardless of the adhesive content of the paper. Papers impregnated with > 6 % Klucel did not easily absorb water but this effect was limited to the application side only. This side appeared glossy, probably because the adhesive did not penetrate into the paper but formed a hydrophobic

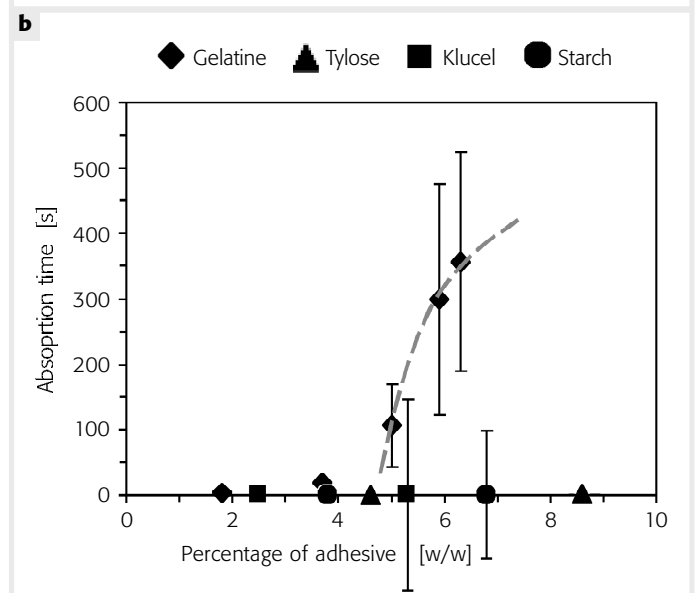
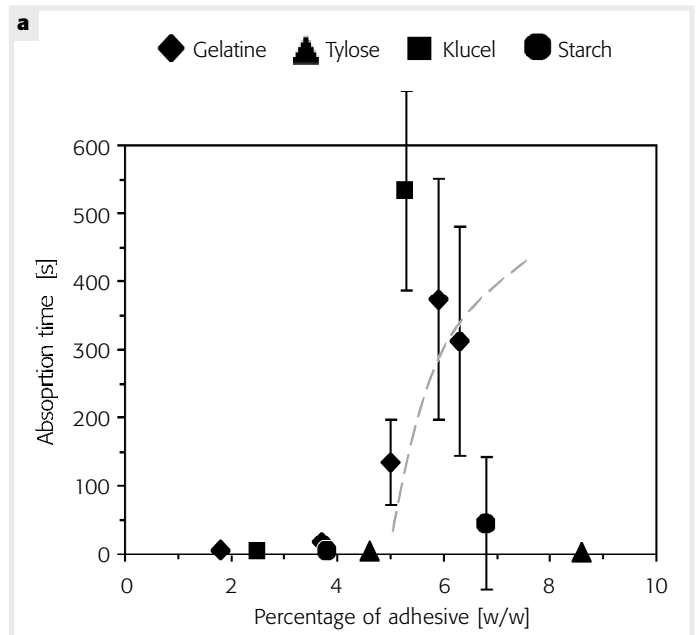
film that prevented the paper surface from absorbing water. Gelatine was the only adhesive that rendered the paper hydrophobic on both sides when its concentration was over 5 %.

The second method is based on capillarity measurements and is inspired by the Klemm test [12]. A paper strip, 1.5 cm wide and 10 cm long, is maintained vertically with its lower edge immersed in water. The capillary ascension length, measured after 5 minutes, showed that all the adhesives limit the water capillary ascension, but to different extents (Fig 8). Of these, gelatine appeared as the most effective adhesive in preventing water capillary ascension.

Of all the tests, gelatine was clearly the most capable of making paper hydrophobic while not significantly modifying



6 Water vapour absorption measurements at different humidity conditions. All solutions used to impregnate the paper were prepared at the same concentration (5 % w/v). The adhesive concentrations (w/w) deposited in the paper: starch, 5.8 to 7.7 %; Klucel, 6.3 to 8.5 %; Tylose, 6.5 to 8.4 %; Gelatine, 5.6 to 6.3 %.



7 Absorption time of 1 ml drop of water against the percentage of adhesive deposited in the paper: Each value corresponds to an average of ten measurements performed on the side of the application (a), or on the side opposite to the application (b). Error bars correspond to standard deviations.

the vapour absorption capacity. It was then investigated for use in lining of artefact.

Testing the Adhesives on the Replicas

The effectiveness of lining the replicas with gelatine and Japanese paper was tested. Klucel was also chosen as a reference for these tests as it offers an interesting alternative: it can be used in a non-aqueous solvent, and it can render at least one side of the paper hydrophobic. Calcium Phytate treated replicas were also lined with Japanese paper (RK1- 8 g/m²: Atlantis) using both adhesives. The adhesion and the appearance of the linings were satisfactory. However, after artificial ageing (35 days at 45 °C and 65 % of RH), some yellow bi-product migration was observed on the Japanese paper of the sample adhered with Klucel (Fig 9). This migration probably occurred in the film of Klucel deposited on the paper surface. The appearance of the gelatine lined sample remained unchanged and thus gelatine was chosen as the adhesive for the conservation treatment.

Conservation Treatment

Preliminary Precautions for the Aqueous Treatment

The dimensions of the artefact, detached pieces and fractures could be physically altered by immersion in water, causing further problems to be treated especially in its re-assembly. In order to limit such undesirable effects, the artefact was temporarily re-assembled before the treatment. The use of paper strip supports for this would probably have limited the ionic exchanges during the treatment and thus were not used. Cold-water insoluble gelatine joints were chosen to re-assemble the pieces and maintain the structural cohesion of the artefact during the treatment. A 4 % solution of gelatine was applied to the fractured edges in two phases separated by a drying period of 24 hours. Gelatine films (10 % solution w/v) were used to adhere the pieces together. A few small, thin, gelatine impregnated paper strips were placed locally at the beginning of the fractures to support the assembly

as a whole. Despite this preliminary consolidation, the artefact remained fragile and had to be handled with great care. A silk-screen was chosen to support the print when floating on the treatment solutions. It provided a rigid support combined with permeability for the liquid exchange necessary during the treatment.

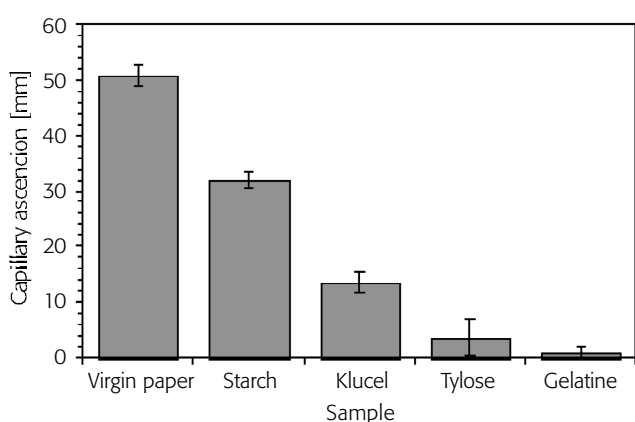
Anti-oxidant and De-acidification Treatment

The print was humidified for four hours at 85 % RH in order to relax the paper and reduce the undulations that would have hindered floating on the treatment solutions. This pre-treatment is also believed to limit stress and its resulting risk of new fractures that could occur when the paper is suddenly wetted.

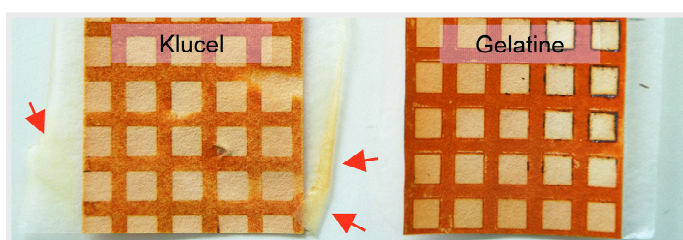
Prior to the chemical treatment, the artefact was floated recto up on a silk screen frame for 5 minutes on pure water in order to soak out the most soluble bi-products. Then it was treated with a Calcium Phytate solution of an approximate pH of 5.2 ± 0.2, followed by a calcium bicarbonate solution, both for 15 minutes. Contact with the solution was enhanced manually using a soft brush over the recto that was protected with a thin non-woven polyester fabric (Hollytex 17 g/m²: Hercules International). The bathophenanthroline test was used to check the phytate solution. No iron (II) was detected even after the floating treatment, indicating that the phytate solution was still active.

After drying, no visual changes were noticed on the artefact. The bathophenanthroline test showed that a large quantity of iron (II) was still present on the paper verso. The treatment duration, sufficient for the replicas, was apparently too short for the artefact, probably because of its hydrophobic nature. It was decided to repeat the treatment under identical conditions. The results of the next bathophenanthroline test on the verso of the paper were only slightly positive. However, it was decided to stop the treatment at this stage to avoid any visible changes occurring to the artefact. No visual change was noticed (Fig 1) and the decrease of the iron (II) content shows that the treatment was at least partially effective. Increasing its duration would mean taking the additional risk of altering the visual appearance of the artefact, for instance, lightening of unprinted areas.

All bathophenanthroline testing was performed on dry paper surfaces. They would certainly have been more sensitive if performed directly on the wet object, however, the results of this test were not considered to be the most decisive criteria for success. When monitoring the impregnation time, most of the attention was paid to avoiding visible changes as explained above. For



8 Water capillary ascension lengths on the papers impregnated with the different adhesives. All solutions used to impregnate the paper were prepared at the same concentration (5 % w/v). The adhesive concentrations (w/w) deposited in the paper: starch, 6.8 %; Klucel, 5.3 %; Tylose, 8.6 %; Gelatine, 6.3 %. Ten measurements were performed for each adhesive and only the average was taken.



9 The visual appearance of lined replicas after artificial ageing: replica lined with Klucel (left) and replica lined with gelatine (right).

similar reasons, no pH measurement was performed after treatment on the original although it would have been very informative.

Consolidation and Lining

After the aqueous treatment, the artefact was left on the silkscreen and placed on blotting paper to absorb the excess water. The polyester non-woven fabric covering the recto was removed and first replaced by two Japanese paper sheets (RK1, 8g/m²: Atlantis). Later, near the end of the drying process when the paper was only slightly damp, these were replaced by two pieces of felt. This method applied a soft pressure to the artefact during the final stages of the drying process. Flatness of the paper was maintained while preserving its surface texture. Being able to move the silkscreen without touching the artefact provided a safe and easy way to regularly replace the blotting papers.

After drying, the artefact was turned over and laid on a piece of felt, its recto side now in contact with two new Japanese papers. The fractures and fragments were consolidated on the verso using Japanese paper strips (Fig 10a). In order to limit the addition of water during this treatment, the Japanese paper was first impregnated with a 4 % w/v gelatine solution then dried and cut into strips. The adhesive was reactivated during the application of the strip with a warm, diluted gelatine solution (2 % w/v).

Lacunae were filled using pieces of tinted paper pulp then

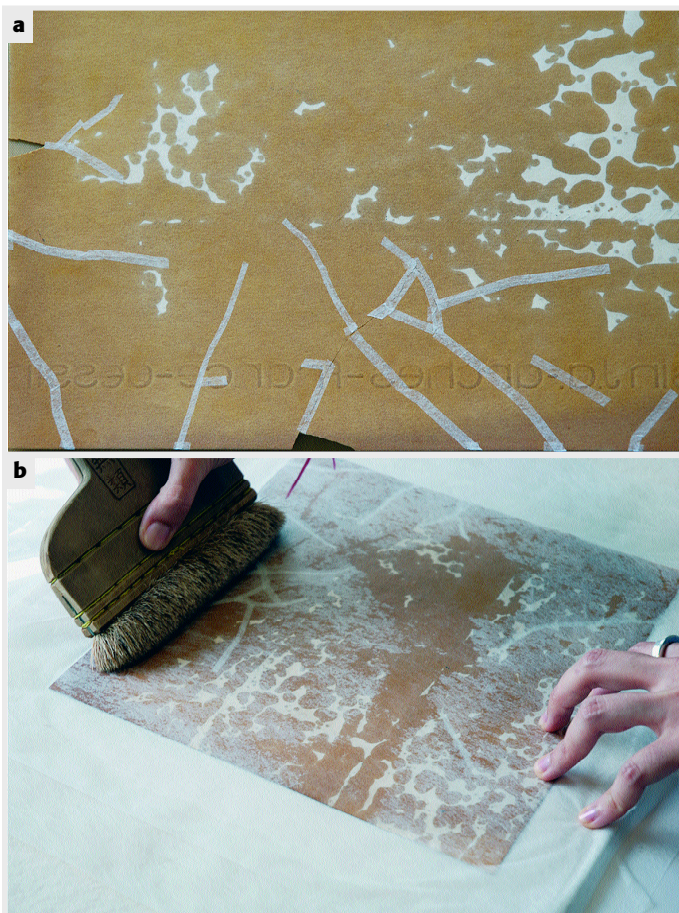
the artefact was lined with gelatine impregnated Japanese paper. This was reactivated in a similar way to the previously used paper strips (Fig 10b and 11). The artefact was then covered by two pieces of felt that applied a soft pressure during the drying process.

Framing

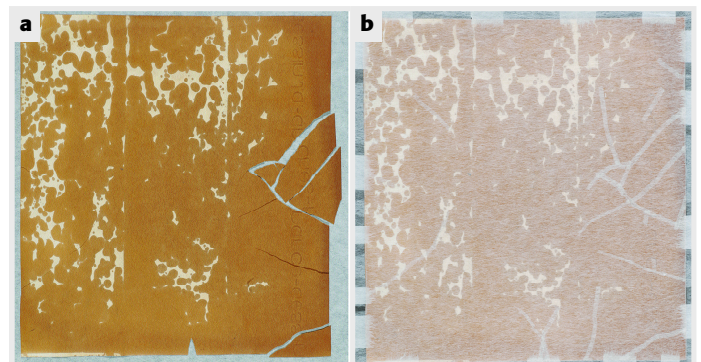
The conserved artefact was to be displayed but any added stress had to be avoided due to its remaining fragility. First it was mounted on a piece of cardboard of the same size (the support). This construction was fixed to a second, larger piece of cardboard (the background), thus bringing the artwork to the fore (Fig 12). The glazing consists of a Plexiglas® sheet the same size as the background board with a built-in spacing of 2 cm between from the artefact and the Plexiglas®. This provides physical protection without impeding visual access. A wooden frame with a honeycombed polycarbonate backboard completes the framing assembly.

Conclusion

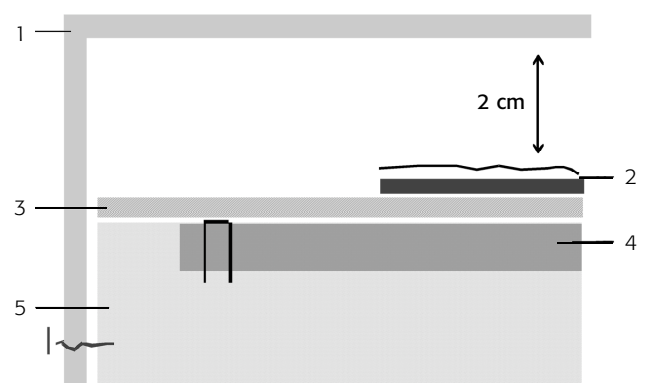
Contemporary graphic art uses the paper sheet as an experimental ground, sometimes combining chemically incompatible materials. This is certainly the case with Bernard Pagès' artwork. As a result, his 'Empreinte de trois plaques de fer rouillées' was in a state of advanced degradation. To explore the conservation possibilities, an anti-oxidant treatment originally developed for



10 Consolidation of the verso: Placement of thin Japanese paper strips (a); reactivation of gelatine impregnated Japanese paper with a diluted solution of gelatine (b).



11 The verso of the artefact: before (a) and after intervention (b).



12 The framing of the artefact (cross section): Plexiglass® glazing (1); artefact mounted on the first (support) board (2); background board (3); honeycombed polycarbonate backboard stapled to the wooden frame (4); wooden frame (5).

iron gall ink corroded manuscripts was tested on replicas and appeared to be effective in preventing further paper degradation. However, with the original, although performed twice, the treatment was probably only partially effective because some free iron (II) remained in the paper. As visual changes to the work were unacceptable, it was decided to stop the anti-oxidant treatment at this point.

The selection of adhesive to be used for the physical consolidation of the artefact was also questionable. The solvent (water) used for the preparation of these adhesives was not regarded to be a decisive factor as the artefact had already been treated in aqueous solutions so most of water soluble compounds should have previously migrated out of the paper. On the other hand, the impact the adhesive could have on the water-to-paper attraction was considered to be a decisive factor as it could influence future cellulose hydrolysis for the worse. The comparative study performed on replicas showed that gelatine was clearly the most appropriate for rendering the paper hydrophobic and was therefore chosen for the consolidation treatment.

This study did not aim to be exhaustive. In particular, the addition of ethanol to the treatment solutions was only partly explored, as it was not possible to produce replicas that were as hydrophobic as the original. However, it was clearly shown that the Calcium Phytate treatment could be used on rust prints without damaging the rust deposit. It also provides some objective comparisons between the properties of the major adhesives used in the paper conservation field.

This approach illustrates the necessary compromise that has to be made between effectiveness and side effects when applying a chemical treatment. It also shows the necessity of close collaboration between scientists and conservators when selecting an appropriate conservation treatment.

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Endnotes

- [1] A detailed description of the method is available at <www.knaw.nl/ecpa/ink/CaPhytate.html>.
- [2] This treatment has been performed at the National Archives of the Netherlands, the Conservation Centre for Art and Historic Artefacts (Philadelphia, USA), the Congress Library (USA) etc.
- [3] The rust deposits were analysed by X-ray micro-diffraction. They are all iron (III) oxy-hydroxides products but have varying crystalline forms. The Goethite form was detected mainly on the origi-

nal. The Lepidocrocite and Akaganeite forms were detected mainly on the replicas.

- [4] A one micro-litre drop, formed with a micro-syringe, is deposited on the paper surface. The absorption measurement corresponds to the time taken to penetrate the paper at 23 °C and 50 % RH. Penetration is achieved when the secular refraction of a raking incident light becomes negligible.
- [5] This adhesive was initially prepared with a concentration of 10 % w/v, using 25 g of starch in 250 ml of water. During the preparation, approximately 40 % of the water evaporates and the concentration rises to approximately 16 % w/v. The starch gel was then diluted in order to obtain a final concentration of approximately 5 % w/v.
- [6] These two gels were prepared in the desired concentration 24 hours in advance. Klucel was prepared in pure alcohol, Tylose in water.
- [7] The Gelatine 225LH30 was left for one hour in cold water then warmed to 50 °C for 30 minutes
- [8] In order to determine the initial dry mass (m_1), the papers were dried for one hour at 105 °C then weighed. The adhesives were applied with a large brush on one side only. The excess of adhesive was removed by placing the six sheets between two blotting papers, then under a piece of glass (21 x 29 cm²) with a 2 kg weight for a short time. After approximately one minute, the mass of the sheets (m^2) was measured again. The percentage of dry adhesive in the paper (Adh%) was then estimated based on the initial concentration of the adhesive used (c): $Adh\% = c (m_2 - m_1) / m_1$.
- [9] The dry mass of the sized sample (m_{dry}) takes the dry mass of the adhesive into account: $m_{dry} = m_1 (1 + Adh\%)$. The measured sample weight (m) leads to the determination of the percentage of absorbed water, (water%): $water\% = (m - m_{dry}) / m_{dry}$
- [10] Standard NF EN ISO 483: 'Petites enceintes de conditionnement et d'essai utilisant des solutions aqueuses pour maintenir l'humidité relative à une valeur constante, septembre 1999'. Potassium acetate saturated solutions were used to obtain 23 % RH and potassium chloride saturated solutions to obtain 85 % RH. The artwork was displayed in an air-conditioned room at 50 % RH.
- [11] These measurements were performed on the prototype Varimass apparatus available at the Laboratoire Génie des Procédés Papiers, Pagora (previously Ecole Française de Papeterie de Grenoble), 461 rue de la Papeterie, BP 65, 38402 Saint Martin d'Hyères Cedex.
- [12] Standard NF ISO 8787, mai 1987, AFNOR: 'Détermination de l'ascension capillaire. Méthode Klemm'.

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Suppliers

Atlantis France, 35 rue du Ballon, 93160 Noisy le Grand, France, Tel +33-1-48155151, Fax +33-1-48155150, www.atlantis-france.com (Zin shofu and RK1 Japanese paper Nao, Kozo fiber).

Canson SAS, BP 139, 07104 Annonay Cedex, France, Tel +33-4-75698800, Fax +33-4-75698999, www.canson.fr (Johannot paper, 125 g.m⁻², cotton linters fibre, gelatine sized).

Hercules International Limited LLC, Aqualon division, Veraartlaan 8, 2288 GM Rijswijk, The Netherlands, Tel +31-70-4134341, Fax +31-70-3191187, www.aqualon.com (Klucel G, viscosity 125-450 mPa.s at 25 °C and 20 g.L⁻¹, Hollytex 17 g/m²), provided by the French supplier Stouls, 9/11 rue de l'Orme Saint Germain, 91165 Champlan Cedex, France, Tel +33-1-69101070, Fax +33-1-69101079, www.stouls.fr.

Rousselot, 10 avenue de l'Arche, 92419 Courbevoie Cedex, France, Tel +33-1-46678700, Fax +33-1-46678701, www.rousselot.com (Gelatine 225LH30; pH 5.6; viscosities of 6.7 % and 60 °C: 5.5 mPa.s; Bloom 225).

ShinEtsu Tylose GmbH & Co KG, Rheingastr. 190-196, 65203 Wiesbaden, Germany, Tel +49-611-9628786, Fax +49-611-9629364 (Tylose MH 200 KG4, DP 500, viscosity 200 mPa.s at 20 °C and 20g.L⁻¹ in water), provided by the French supplier Stouls (contact dates see Hercules International) under the reference Tylose MH300P^a.

Whatman plc, Springfield Mill, James Whatman Way, Maidstone, Kent ME14 2LE, United Kingdom, Tel +44-0-1622-676670, Fax +44-01622-691425, www.whatman.com (paper no 1).

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