Methods of Aqueous Treatments: The Last Resort for Badly Damaged Iron Gall Ink Manuscripts

Véronique Rouchon, Marthes Desroches, Valéria Duplat, Marine Letouzey, Julie Stordiau-Pallot

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Methods of Aqueous Treatments

The Last Resort for Badly Damaged Iron Gall Ink Manuscripts*

The calcium phytate treatment has given rise to intensive research. Despite its efficiency largely having being demonstrated, this treatment is not used on badly damaged manuscripts mainly because water is used as a solvent. Indeed, exposing badly damaged paper to aqueous solutions is paired with a substantial risk of causing additional splits and losses, which has so far hindered the use of calcium phytate on the most vulnerable manuscripts. This work investigates ways of manipulating badly damaged manuscripts during aqueous treatments. It was first tried out on test samples then originals. The results showed that mechanical risk is greatly minimized when the treatment is performed by flotation and with specific handling and drying precautions.

Wässrige Behandlung von Tintenfraß: Letzter Ausweg für schwer geschädigte Eisengallusmanuskripte
Die Calciumphytatbehandlung ist Gegenstand intensiver Forschungsarbeit. Obwohl ihre Wirksamkeit hinlänglich erwiesen ist, wird sie bei stark geschädigten Handschriften nicht angewandt, da Wasser als Lösungsmittel zum Einsatz kommt. Tatsächlich kann eine wässrige Behandlung zu weiteren Rissen und Substanzverlust im behandelten Papier führen. Daher wurde bislang bei derart gefährdeten Handschriften meist auf eine Phytatbehandlung verzichtet. Die vorliegende Arbeit untersucht verschiedene Methoden, stark geschädigte Manuskripte wässrig zu behandeln. Die Tests wurden zunächst an Probepapieren dann an Originalen durchgeführt. Die Ergebnisse zeigten, daß sich das Risiko mechanischer Schäden deutlich verringert, wenn man das Papier schwimmend wässert und besondere Vorkehrungen bei seiner Handhabung und Trocknung trifft.

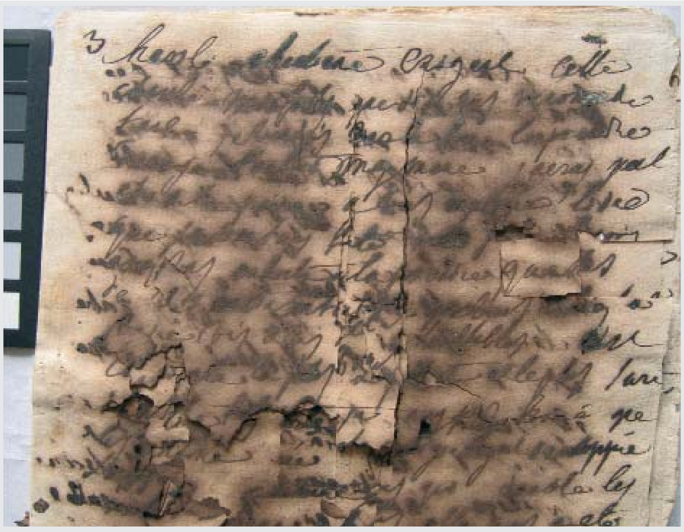
The conservation of manuscripts damaged by iron gall inks remains a true challenge. Despite intensive research over the last two decades devoted to the development of treatments, the conservation community has so far adopted no universal solution, probably because the reality of collections is their diversity and so multiple criteria must be taken into account when choosing a conservation treatment.

Of all solutions investigated to delay iron gall ink corrosion, the calcium phytate treatment proposed in the 1990s (Neevel 1995; Reissland and Ligterink 2011) has been the most extensively tested: its safety regarding paper has been demonstrated (Botti et al 2005) and its ability to limit the degradation of damaged paper has been measured several times on laboratory samples (Reissland and De Groot 1999; Neevel 2000; Kolar et al 2000, 2005 and 2007; Zappala and Stefani 2005; Henniges and Potthast 2008; Orlandini 2009; Rouchon et al 2011). The calcium phytate treatment involves immersing the object in several aqueous solutions for about one hour. The use of water as a solvent has pros and cons (Reissland 1999a, 1999b and 2000; Rouchon et al 2008 and 2009): water helps to delay the paper deterioration because it enables the partial removal of iron, sulphates, acids and degradation by-products, while organic solvents do not. When other parameters are taken into account, such as the aesthetic aspect or the historical aspect of the paper composition, the use of water as a solvent produces negative effects: the dissolution of water soluble products drastically modifies the chemical composition of the paper/ink and significantly changes the appearance of the object; the ink takes on a cooler colour and the paper lightens considerably. The calcium phytate treatment of objects of aesthetic value, such as drawings, is also not recommended.

These visual side effects may be acceptable on badly damaged manuscripts (e.g. Fig 1), especially when the text forms the main value of the object. When the paper can no longer be handled, the text becomes inaccessible and the object is considered as lost. In these specific cases, it would be useful to apply the phytate treatment before consolidating the support by conventional treatments such as lining. But here another problem arises: the exposure of a highly brittle paper to different aqueous solutions is accompanied by a substantial risk of mechanical stress, which might create new splits and losses.

These arguments lead to a paradoxical situation: the calcium phytate treatment, whose effectiveness is established, is not used on highly damaged documents, but only on documents in a reasonable condition that can still be handled in aqueous solutions without major mechanical risk.

In view of these considerations, it seemed useful to investigate an appropriate methodology for the application of aqueous treatment to badly damaged documents. The use of non-woven viscose fabric combined with a floating process has been proposed to minimize the mechanical risk (Huhsmann 2007). This option was complemented by the proposal of a work stand-



1 General view of the badly damaged document sacrificed in this study.

ard for the treatment of damaged manuscripts (Huhsmann 2008). This very detailed work is illustrated by the treatment of a moderately damaged document, which is held sandwiched between two fabrics during treatment. The employment of this universal process on badly damaged pieces (Fig 1) remains questionable. Moreover, this process does not include lining, which is unavoidable for badly damaged papers.

This study was performed in a laboratory context but was motivated by the presence of badly damaged pieces in the collections of the French National Library, which are impossible to handle safely. The aim is to find a 'last resort' for the conservation of these objects.

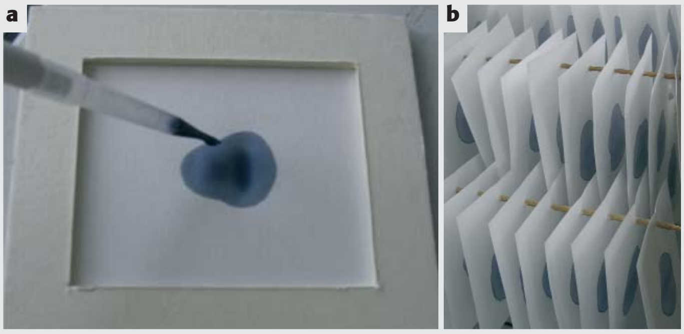
In the initial phase of this project, an attempt was made to assess the mechanical risk as objectively as possible. A specific methodology using test samples was formulated in order to semi-quantitatively evaluate the mechanical damage induced by aqueous treatments. This determined the most hazardous treatment steps as well as the safest. These results were then used in the second phase for the development of a treatment procedure that could be employed on real objects.

Methodology

Using Test Samples to Evaluate the Mechanical Risk

Test samples were made of a sheet of approx. 10 x 10 cm Whatman paper (Whatman) with a deposit of a 0.1 mL drop of iron gall ink ([1] Fig 2a), resulting in an ink spot of approx. 3 cm in diameter. The samples were then artificially aged at 85°C and 65 % RH for 13 days. After ageing, the inked areas were very fragile whereas the blank area remained in good condition. When submitted to an aqueous bath without particular care, these samples split into pieces.

As the samples were not strictly identical, it was necessary to develop a semi-statistical approach [2], which required the manufacture of a large number of samples (Fig 2b). Each treatment was evaluated on the basis of a set of 30 samples. These were divided in 5 groups of 6 samples, which were treated simultaneously. The aqueous treatments were conducted in a transparent container placed on a light-table (Fig 3). Several photographs were taken of all samples at different steps of the treatment in order to identify the moments when the splits occurred (Fig 3b). A semi-quantitative evaluation could then be achieved by counting the number of splits occurring on the entire set.



2 Preparation of test samples: application of a 0.1 mL drop of ink **(a),** samples hanging during drying **(b).**

For example, Fig 4 shows the average number of splits per sample occurring during a treatment performed in the same way on two sets of 30 samples. The results obtained on the two sets were very similar, showing that the methodology was satisfactorily reproducible.

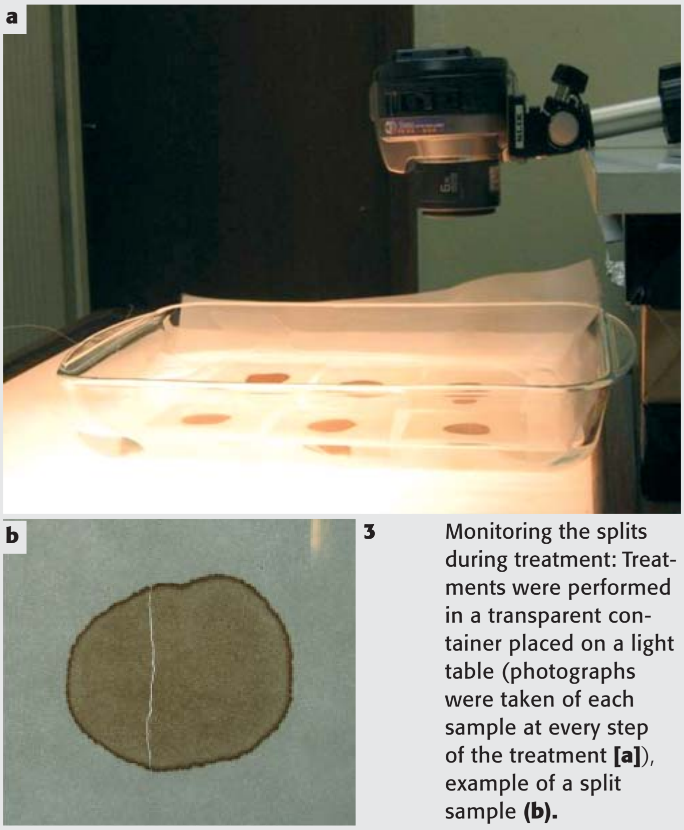
From Test Samples to Original Manuscripts

The use of test samples for the evaluation of the mechanical risk is useful to achieve a general idea of the most promising directions to investigate but is obviously limited. The manufacture of test samples saves originals, but cannot reproduce their complexity. It was therefore decided to apply the general conclusions derived from the test samples to the treatment of originals. The second part of this project was performed on a few badly damaged sheets with iron gall ink and dating from the 18th century (Fig 1).

Results

Evaluation of the Most Hazardous Sample Treatment Step

It was initially expected that the immersion of the sample would be very problematic: the swelling of moderately damaged paper is often said to be very hazardous for damaged inked areas (Reissland 1999b and 2000; Huhsmann 2007) because it may induce excessive strain on the most brittle part of the paper. Nevertheless, this seemingly logical argument was not confirmed here [3], as the samples were very easy to immerse and could remain for hours in the bath (Fig 4) with no visible damage. However the removal of the samples from the bath was very delicate. When conducted with tweezers without any particular care, it led to



3 Monitoring the splits during treatment: Treatments were performed in a transparent container placed on a light table (photographs were taken of each sample at every step of the treatment **[a]**), example of a split sample **(b).**

Tab 1 Description of the tested actions.

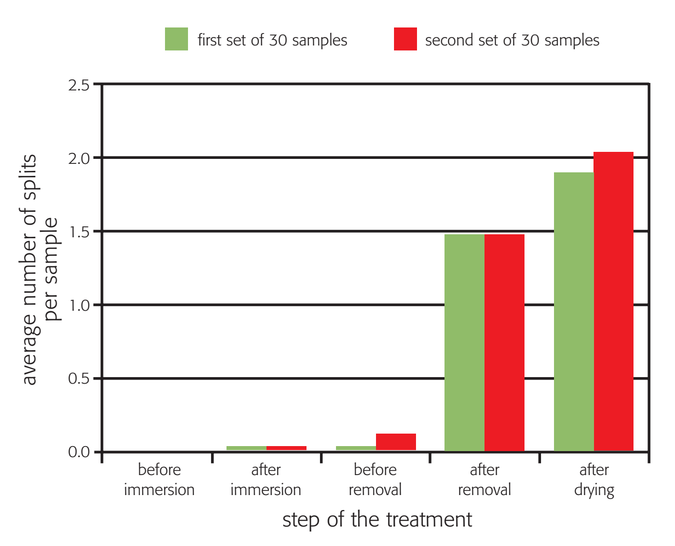
Gesture	*Description*
tweezers	No specific care. Tweezers were used to hold the undamaged part of the sample during immersion and removal from the bath with tweezers.
rigid grid	The samples were first placed on a polyester woven mesh, 43T (A. Buisine), covered with 1.5 x 1.5 mm flexible plastic net (BHV), then placed between two rigid metallic grids (BHV); easy to handle during immersion and removal from the bath.
mesh	The samples were placed between two fine polyester woven meshes, 43T (A. Buisine) and immersed in the bath. The woven mesh could be easily handled during removal without touching the samples.
frame	A 1.5 x 1.5 mm flexible plastic net (BHV) stretched on a rigid plastic frame was first immersed. It was covered with a fine polyester woven mesh, 43T (A. Buisine). The samples were immersed with tweezers, then removed from the bath using the frame.
plexi	The samples were immersed in the bath using tweezers, then removed using a Plexiglas® plate.
foam rubber	A foam rubber was first immersed. It was placed on a 1.5 x 1.5 mm flexible plastic net (BHV) which was stretched on a rigid plastic frame. This was covered with a fine polyester woven mesh, 43T (A. Buisine). The samples were immersed with tweezers and placed on the last layer. The frame was used for the removal of all layers.
siphon	Same as 'frame', but instead of removing the samples from the bath, the bath is emptied with a siphon.
floating	Same as 'frame', but instead of being immersed, the samples were simply floated on the solutions.
floating RV 1	Same as 'floating'. After removal from the solution, the samples were covered with a second polyester woven mesh, 43T (A. Buisine), held with a second rigid frame and turned over. Then the first polyester woven mesh and the first frame were removed and the verso side of the samples were exposed to the solution in the same way as the recto.
floating RV 2	Same as 'floating RV1', but without removing the first polyester woven mesh.

approximately 1.5 splits per sample, meaning that almost all samples were split.

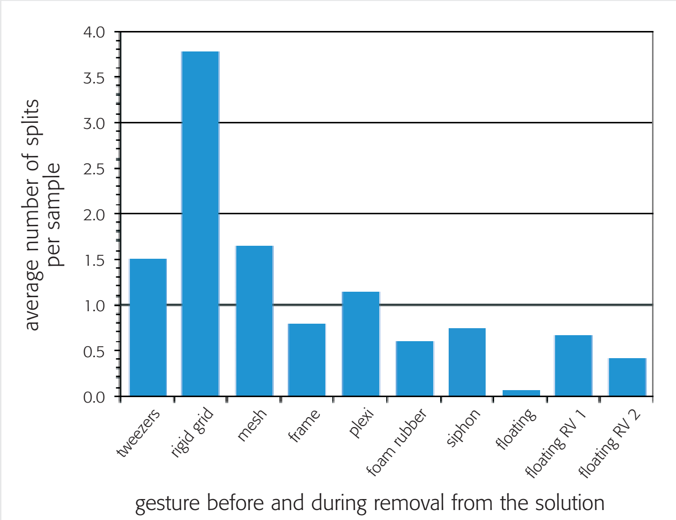
Optimizing the Sample Removal Method from the Solutions

Several techniques were also investigated to handle the samples during their removal from the bath. These techniques (Tab 1) were not exhaustive, but aimed to span the most typical actions that could be employed on damaged paper: sandwiched between two rigid grids or two pieces of woven mesh, placed on a rigid support, or left to float without any constraints, and so on. As shown in Fig 5 most of these actions had damaging results. Sandwiching the document between two rigid grids was by far the worst option (Fig 5: rigid grid). Generally, any water flow on the paper surface induced damage. Even using a siphon to empty the bath was not effective in preventing splits (Fig 5: siphon). Letting the paper float freely on the solutions appeared to be the safest option (Fig 5: floating). The paper could then be carried from one bath to another by using a fine mesh stretched on a rigid frame, the latter being slightly immersed in the solution during treatment.

As we were at first reluctant to apply the treating solution to one side only, it was attempted to turn the samples between two baths. Here again, damaging results were obtained (Fig 5: floating RV1 and floating RV2). Inverting the sample means removing it from the support, which is very hazardous when the paper is moist. The only way to remove the support with less risk was to let the sample at least partially dry but this was not in-



4 Evaluation of the reproducibility of the experiment on two sets of 30 samples. During immersion and removal from the bath, the samples were manipulated with tweezers without any specific care (Tab 1: 'tweezers'). They were dried in open air. The removal of the samples from the bath was the most delicate action.



5 Average number of splits appearing on the samples after removal from the bath. The tested actions are depicted in Tab 1.

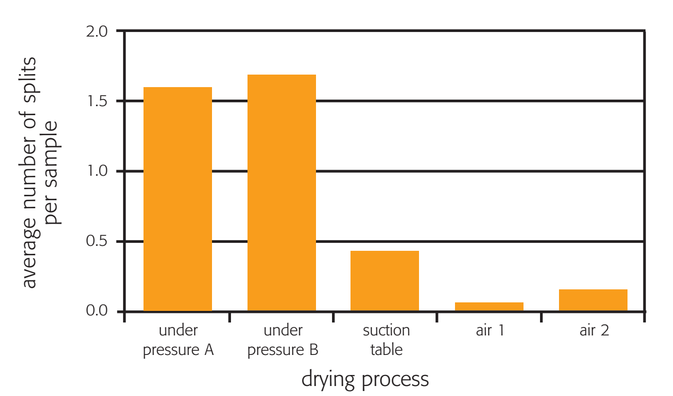
Tab 2 Description of the tested drying processes.

Name	*Description*
under pressure A	The samples were covered with a non-woven polyester film (Stouls) then placed between two blotting papers. The whole was held under pressure with a 2 kg weight.
under pressure B	The samples were immersed in ethanol before being dried in the same way as 'under pressure A'.
suction table	The samples were immersed in ethanol then placed overnight on a suction table.
air 1	The samples were immersed in ethanol then dried in open air on a rack.
air 2	The samples were dried in open air on a rack.

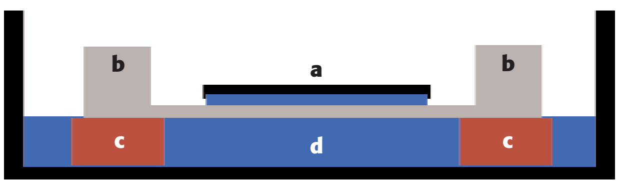
vestigated. In conclusion, it was decided that the best way to proceed was to float the paper on one side only, and to increase the duration of the treatment.

Optimising the Drying Process

Drying is the second most hazardous step of an aqueous treatment: as the inked areas remain less flexible than blank areas, they may not follow the dimensional changes of the sheet during drying. As a result, new splits may appear or existing splits may become longer. Several ways of drying the paper were also tested (Tab 2). Here again, the worst results resulted from the application of constraint to the paper (Fig 6: under pressure) whereas the best results were obtained on the samples that were left to dry in the open air (Fig 6: air).



6 Average number of splits appearing on the samples after drying. The drying processes are depicted in Tab 2.



7 Overall scheme of the treatment: The document **(a)** is placed on a silkscreen **(b)** supported by four blocks **(c)** at the corners. The tray is filled until the solution **(d)** reaches the screen. The weight of the document enables water to permeate through the mesh underneath the document only.

Investigating the Lining Process

Another important step of the universal treatment needed investigation: the phytate treatment delays the chemical degradation of the cellulose but does not reinforce the altered paper. It would seem necessary to consolidate a badly damaged document. Several types of lining were investigated: these treatments were performed using the same method [4] but with various types of adhesive (starch 4 % w/v, gelatine 4 % w/v, Tylose 4 % w/v and Klucel G in ethanol 4 % w/v). All adhesives led to an increase of approximately 0.2 to 0.4 splits per sample, showing that the composition of the adhesive is not a determinant factor for the risk of splits. The softness of the brush and the fluidity of the adhesive (which affects the ease of application) appeared to be more critical.

Use on Highly Damaged Originals

Some tests were made on original samples, which lead to the determination of five additional factors that are critical in mechanical risk management:

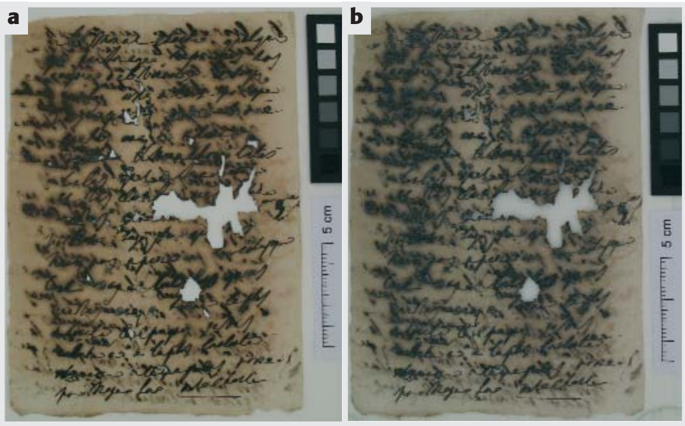
> The carrier should remain rigid while allowing water to permeate. A silkscreen (A. Buisine) constructed with a polyester woven mesh, 43 threads·cm^{-1}, stretched on an aluminium frame perfectly fits this criteria.

> The mesh should be positioned at the very surface of the bath so that with the weight of the paper, the application of a soft pressure on the mesh enables water to just permeate through the mesh. The water should come through the mesh ONLY underneath the document, not at the free margins of the screen (Fig 7).

> Bending the paper and touching its surface should absolutely be avoided during the treatment.

> Placing the document on a free mesh or sandwiched between two meshes during the floating process should be avoided. This seemed to have caused additional damage. It was decided to place the document directly on the silkscreen with no intermediate layers. It is then possible to undertake the lining in the meantime without waiting for the complete drying of the document. This point differs significantly from the previous recommendation (Huhsmann 2008).

> It was not possible to perform the complete drying in open air

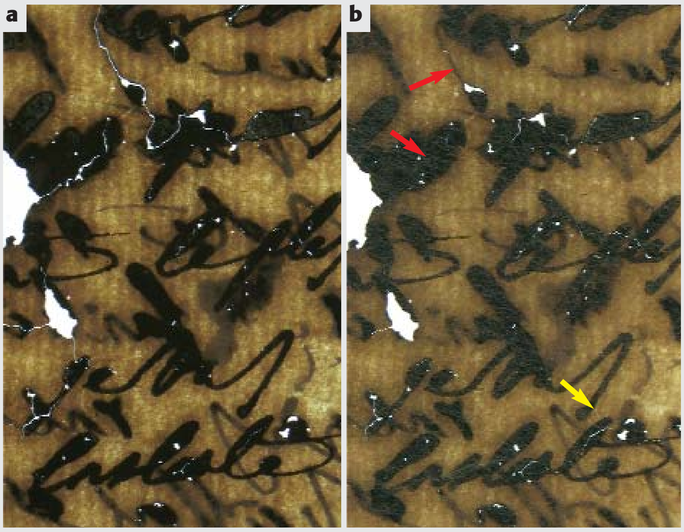


8 Overall view of an original document: before treatment **(a)**, after treatment **(b)**.Images of this document at each step of the treatment are available in the 'Instructables' section.

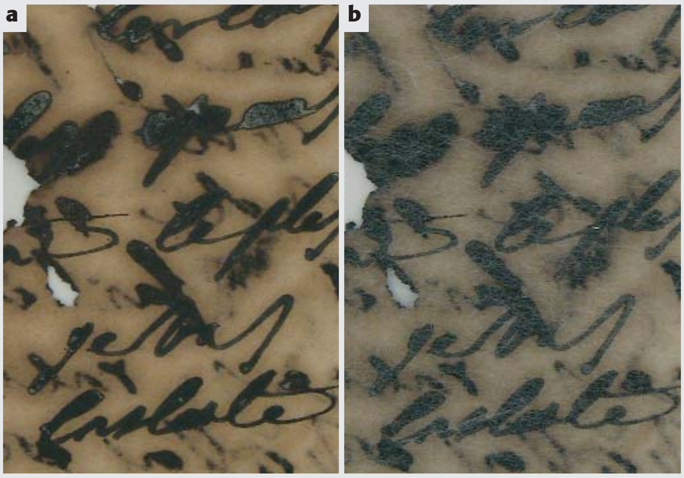
because the blank paper tends to undulate, which enlarges existing splits or causes new ones. The weight of a 1 cm thick piece of wood and a couple of woollen felts is enough to keep the paper flat while enabling an even air exchange during drying.

Finally a method of handling the paper sheet from beginning to end of the conservation treatment was developed. This process, illustrated in (Fig 7) and fully described in the 'Instructables' section of this issue, enables the treatment of a paper that is so brittle that manipulation is almost impossible. In Fig 8 for example, such a paper was treated without resulting in any substantial mechanical damage. This is particularly clear when examining the sample on a light table (Fig 9). No new splits could be found. Some existing splits were slightly enlarged (Fig 9: yellow arrow) while some others were no longer visible (Fig 9: red arrows), probably because the two edges of the split rejoined.

An initial concern was that a floating process would make the treatment less efficient because the chemical exchanges between paper and solution could be limited to one side only. The final colour of the paper (Figs 8, 10) indicates that this is not the case: the paper lightens significantly on both sides and turns a cooler hue similar to an immersion result. It would appear that the floating process does allow chemical exchange between the paper and the solution and does not jeopardize the overall efficiency of the treatment. It is however advised to extend the duration of the treatment (compared with immersion treatments) since the floating process may delay chemical exchanges.



9 Light table image of the same document as in Fig 8 (detail): before treatment **(a)**, after treatment **(b)**.



10 Daylight picture of the same document as in Fig 8 (detail): before treatment **(a)**, after treatment **(b)**.

Loss of legibility is the main drawback of the method, mainly due to the slight opacity of the Japanese paper. This aspect may not be noticed with a first impression of the document (Fig 8), but becomes obvious when focussing on details (Fig 10). Dyeing the Japanese paper with diluted acrylic colours was attempted with encouraging results (not shown): further development in that direction requires an investigation into the stability of the dye when in close contact with the manuscript.

Conclusion

This work aimed to formulate a treatment of badly damaged papers embrittled by iron gall ink corrosion. Firstly the most damaging steps, the removal of the paper from the baths and its drying, were determined using test samples. Several methods were attempted to reduce the mechanical risk: letting the paper float on the solution was by far the best option and the best way to dry the paper was to apply a minimal constraint. These approaches were adapted to the treatment of badly damaged original documents, which led to the development of a treatment protocol. It is hoped that this protocol will allow a more specific use of the calcium phytate treatment, which remains to date the most widely documented of anti-oxidant treatments. Obviously this method is not suitable to treat kilometres of documents. It was designed in a laboratory context to address hopeless documents that can no longer be manipulated, even with the greatest of care. The method is time consuming (probably more than one hour per sheet) and is also restricted to some specific cases where no additional split is acceptable and where the time spent corresponds to the value of the object.

Acknowledgements

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Endnotes

* A summary of this work is available in French: Rouchon, V., Letouzey M., Desroches, M., Duplat, V., Duranton, M., Pellizzi, E., and Stordiau-Pallot, J. (2011): Traitement de restauration des manuscrits endommagés par les encres ferrogalliques: atouts et limites du traitement au phytate de calcium. In: Support Tracé, Vol 11, pp 106-115.

[1] Composition of the ink: iron II sulphate heptahydrate (11.6 $g{\cdot}L^{-1}$), gallic acid (1.5 $g{\cdot}L^{-1}$), gum arabic (34.1 $g{\cdot}L^{-1}$; all Sigma-Aldrich).

[2] A full statistical approach would require a sampling of more than 100 test samples per treatment which would be much too time consuming. We opted for a semi-statistical approach and sampled approximately 30 laboratory tests per treatment. This lead to what was considered to be a satisfactory reproducibility. The ex-

perimental error relative to the average number of splits remains difficult to determine, but this aspect was not considered to be limiting since this approach enabled the distinction between treatments that are damaging and those not.

[3] This observation may be due to the fact that the samples remained water-permeable on their whole surface whereas highly damaged originals are often less permeable on inked areas than on blank areas.

[4] After the flotation, the samples remained on the same carrier for some hours in order to initiate drying. When the paper was no longer wet but only damp, it was covered with a thin Japanese paper (RK00, 3.6 $g \cdot m^{-2}$, Atlantis France). The adhesive was applied through the Japanese paper with a soft brush. In the case of Klucel G, the samples were placed in an ethanol bath before lining in order to accelerate the drying process.

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Suppliers

A. Buisine, 78 rue Felix Faure, 92700 Colombes, France, Tel +33-141-192970, Fax +33-147-858210, www.serigraphie-boutique.fr (polyester woven mesh, 43 threads$\cdot cm^{-1}$; silkscreen fabric).

Atlantis France, 35, rue du Ballon, 93160 Noisy le Grand, France, www.atlantis-france.com, Tel +33-148-155151, Fax +33-148-155151 (Japanese paper, Nao, RK00; Klucel G; Zin Shofu starch).

BHV, 52 rue de Rivoli, 75 189 Paris Cedex 4, France, Tel +33-977-401400, Fax +33-142-749679, www.bhv.fr (1.5 x 1.5 mm flexible plastic net; 1 x 1 cm rigid metal grid)

Sigma-Aldrich Chemie S.a.r.l., 80 rue Luzais, 38070 Saint-Quentin Fallavier, France, Tel +33-474-822888, Fax +33-474-956808, www.sigmaaldrich.com (gallic acid monohydrate, 398225; iron II sulphate heptahydrate, 215422; gum arabic, G9752)

Stouls, 9-11 rue de l'Orme Saint-Germain, 91165 Champlan Cedex, France, Tel +33-1-69101-070, Fax +33-1-69101-079, www.stouls.com (Tylose MH300P; Reemay 17$g \cdot m^{-2}$; blotting paper 250 $g \cdot m^{-2}$).

Whatman olc, Springfield Mill, James Whatman Way, Maidstone, Kent ME 14 2LE, United Kingdom, Tel +44-1622-676670, Fax +44-1622-691425, www.whatman.com (Whatman No 1).

Authors

Véronique Rouchon has an engineering background (Ecole Polytechnique, Palaiseau) and a PhD in Material Sciences (University of Paris VII). She was appointed lecturer at the University of La Rochelle and joined the CRCC in 2006 in order to focus on paper conservation science.

Véronique Rouchon, Centre de Recherches sur la Conservation des Collections (CRCC), MNHN, CNRS, MCC, 36 rue Saint Hilaire, 75005 Paris, France, Tel +33-1-40795303, rouchon@mnhn.fr

Marthe Desroches completed a master degree in paper conservation at the Institut National du Patrimoine (INP) in 2007. She held a temporary position in Paris (French National Library) to work on the project and now runs her own private conservation workshop.

Marthe Desroches, 26 rue du Peintre Lebrun, 78000 Versailles, France, Tel +33-672-850187, marthedesroches@hotmail.fr

Valeria Duplat completed a master degree in paper conservation at the Institut National du Patrimoine (INP) in 2007. In 2008, she had a temporary position in Paris (French National Library and CRCC) and in Copenhagen (Royal Library) to work on iron gall ink corrosion. She now runs her own private conservation workshop.

Valeria Duplat, 9 rue Etex, 75018 Paris, France, Tel +33-1-664211459, valeriaduplat@gmail.com

Marine Letouzey completed a master degree in book and paper conservation at the University of Paris I (Panthéon Sorbonnes) in 2006. She had a temporary position at the CRCC in 2006 to work on the project. She is now co-founder of the private conservation workshop Art&.

Marine Letouzey, Art&, 25 rue Campo Formio, 75 013 Paris, France, Tel +33-677-810140, marine.letouzey@free.fr

Julie Stordiau Pallot completed a master degree in paper conservation at the Institut National du Patrimoine (INP) in 2005. She had a short-term position at the French National Library in 2006 to work on the project at the CRCC. She now works as a paper conservator.

Julie Stordiau-Pallot, 102 mail du Neutrino, 01280 Prévessin Moëns, France, Tel +33-670-562666, julie.stordiau@hotmail.fr