INVESTIGATION INTO
THE REDUCTION OF
FOXING STAINS IN PAPER

Madison Brockman and Emily Farek
Advisors: Richard Wolbers and Joan Irving
Winterthur/University of Delaware Program in Art Conservation

ANAGPIC 2018, Queen’s University
Description

- Le Ballon (left) and Le Pigeon (right)
- Chine collé lithographs on wove paper
- 23.5 x 16.3 inches

Provenance

- 1870 - 1871
- Lithographs by Émile Vernier
- After paintings by Pierre Puvis de Chavannes
- Published by Lemercier & Cie, Paris
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BT Recto, normal illumination

S.O.S.!

- Pervasive foxing
- Risk of delamination

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**Technique:**

- Damp 2° support
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- Pasted verso of 1° support
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- 1° support placed on 2°
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• Damp 2° support
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• 1° support placed on 2°
• Plate printed on 1° as objects are pressed together
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- Damp 2° support
- Pasted verso of 1° support
- 1° support placed on 2°
- Plate printed on 1° as objects are pressed together

*Risk of delamination in aqueous treatment due to water-soluble adhesive*
THE ARTISTS

Pierre Puvis de Chavannes
1824 - 1898

Émile Vernier
1829 - 1887
Franco-Prussian War

- 19 July 1870 – 10 May 1871
- Paris under siege
- Messages carried by balloons and pigeons
- Images to uplift Parisian people
- Immediately reproduced for distribution

Images courtesy L'Histoire Pa L'Image
LE BALLON
LE PIGEON

BT Recto, normal illumination

BT Recto, UV illumination

BT Recto, transmitted illumination
WHAT IS FOXING?
WHAT IS FOXING?

Paper Support  Foxing
Detail: Foxing, normal illumination

Detail: Foxing, long-wave UV illumination
ART + SCIENCE

Treatments for 2nd year Paper Conservation

Aqueous Cleaning Methods Seminar with Richard Wolbers
First experiment on expendable foxed print:

- Drops of ferric chloride (iron (III) form) were applied to sample
- Agarose gel plugs containing ascorbic acid and EDTA were placed on the ferric chloride spots
- The gel was effective: proves iron (III) can be reduced and chelated
Other expendable examples of foxed prints were treated using various reducing agents, chelators, and enzymes.

**PRELIMINARY TESTING**

- Enzyme hydrolyzes poly-β(1→3)-glucose
- Lysing enzyme blend contains β-glucanase, cellulase, protease, and chitinase activities; industrial quality
- Lyticase is more targeted; premium quality

**Enzyme specificity**

**Ascorbic acid**

**Sodium hypophosphite**
Other expendable examples of foxed prints were treated using various reducing agents, chelators, and enzymes.
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The expendable examples are:
1. placed into a DI bath containing the reducing agent and chelator,
Other expendable examples of foxed prints were treated using various reducing agents, chelators, and enzymes.

The expendable examples are:
1. placed into a DI bath containing the reducing agent and chelator,
2. rinsed in DI water,

Richard Wolbers and Madison Brockman pour in the enzyme solution.
The expendable examples are:
1. placed into a DI bath containing the reducing agent and chelator,
2. rinsed in DI water,
3. then placed in a DI bath containing the enzyme.
Other expendable examples of foxed prints were treated using various reducing agents, chelators, and enzymes.

- Ascorbic acid
- EDTA
- Lysing enzymes
- Sodium hypophosphite
- DTPA
- Lysing enzymes
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- Ascorbic acid
- EDTA
- Sodium hypophosphite
- DTPA
- Lysing enzymes
- Lyticase
TREATMENT PROTOCOL

• Step 1: Pre-rinse
• Step 2: Reducing agent and chelator solution
• Step 3: Rinse
• Step 4: Enzyme solution
• Step 5: Final rinse
TREATMENT PROTOCOL

• **Step 1: Pre-rinse**
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- Step 5: Final rinse
International conference in conservation science

- Held in London, UK
- 24 countries represented
- 42 papers presented:
  - paper
  - paintings
  - objects
  - textiles
  - new research
- Numerous other posters presented
- Polysaccharide, polyacrylic, and novel gel systems
International conference in conservation science held in London, UK
WHY GEL?
WHY GEL?

Three common polysaccharide gels

<table>
<thead>
<tr>
<th>Gel Type</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Agarose</td>
<td>5% (w/v)</td>
</tr>
<tr>
<td>Gellan Gum</td>
<td>5% (w/v)</td>
</tr>
<tr>
<td>Methylcellulose</td>
<td>4% (w/v)</td>
</tr>
</tbody>
</table>

Moisture Control

- Can act as a reservoir for solutions
- Can act as a poultice for degradation products

Contact angle tests on different papers

<table>
<thead>
<tr>
<th>Paper Type</th>
<th>Contact Angle</th>
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<tbody>
<tr>
<td>Whatman Filter Paper</td>
<td>&lt;&lt;90º</td>
</tr>
<tr>
<td>Handmade Rag Paper</td>
<td>90º</td>
</tr>
<tr>
<td>Machinemade Woodpulp Paper</td>
<td>&gt;90º</td>
</tr>
</tbody>
</table>
WHY GEL?

**Moisture Control**
- Can act as a reservoir for solutions
- Can act as a poultice for degradation products

**Physical Restriction**
- Can be cast in large sheets
- Gel sheet can cover entire object
- Weight may prevent layer separation

Image courtesy of Joan Irving
WHY AGAROSE?
WHY AGAROSE?

Solution Compatibility

- Agarose is a neutral gel
- Can carry ionic and enzymatic solutions
WHY AGAROSE?

Solution Compatibility
- Agarose is a neutral gel
- Can carry ionic and enzymatic solutions

Capillarity
- Pore size can be decreased with increasing concentration
- Pore size determines capillary force
- Ex: 5% w/v gel releases moisture slower than 1% w/v gel
“SEPARATED AT BATH”

SUCTION TABLE

TEK-WIPE
“SEPARATED AT BATH”

<table>
<thead>
<tr>
<th>COMPARISON</th>
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<tr>
<td>• Photodocumentation</td>
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<td>• Mend tears</td>
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<td>• Humidification/flattening as necessary</td>
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</table>
“SEPARATED AT BATH”

**SUCTION TABLE**

- All rinse solutions sprayed on object under suction
- Gels applied to object for 20 minutes:
  - Under suction for 5 minutes
  - No suction for 10 minutes
  - Under suction for 5 minutes

**COMPARISON**

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**TEK-WIPE**

- TEK-Wipe kept damp with rinse solutions
- Gels applied to object:
  - 30 minutes of contact time
  - No Mylar or other covering material
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OVERALL STEPS

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Created buffered solution as base for gel solutions using sodium phosphate and citric acid

OVERALL STEPS

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SUCTION TABLE

Added DTPA and NaPO$_2$H$_2$·H$_2$O to half of the buffered solution for the reducing/chelating gel

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Using a squeegee to ensure an even thickness of the agarose gel
Pre-rinsing using Dia sprayer
Solution: DI water with sodium citrate and citric acid, buffered to pH 6 and conductivity isotonic to print

OVERALL STEPS

- Photodocumentation
- Surface cleaning
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- **Pre-rinse with buffered solution, pH 6**
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Applying first agarose sheet atop *gampi* barrier layer

Solution: Buffered solution, DTPA, and NaPO$_2$H$_2$·H$_2$O adjusted to pH 7.5 with NaOH

### OVERALL STEPS

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### SUCTION TABLE

**Rinsing with Dia sprayer**  
Solution: DI water with sodium citrate and citric acid, buffered to pH 6

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Applying second agarose sheet atop *gampi* barrier layer
Solution: Buffered solution at pH 7.5 with lyticase enzymes
SUCTION TABLE

Rinsing with Dia sprayer
Solution: Calcinated filtered tap water, adjusted to pH 8 with Ca(OH)$_2$

OVERALL STEPS

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- Humidification/flattening as necessary
### SUCTION TABLE

After final rinse, print was placed into a drying stack

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“Le Pigeon,” BT

“Le Pigeon,” DT after bathing
RESULTS

“Le Pigeon,” BT

“Le Pigeon,” DT after bathing
“Le Pigeon,” BT

“Le Pigeon,” DT after bathing
Surface cleaning with cosmetic sponges and white vinyl eraser crumbs

OVERALL STEPS

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Preparing the bathing chamber by saturating the TEK-Wipe in the pre-rinse solution

OVERALL STEPS

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TEK-WIPE

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Using a squeegee to ensure even saturation and planarity of the TEK-Wipe
Pre-rinsing using Dia sprayer
Solution: DI water with sodium citrate and citric acid, buffered to pH 6 and isotonic to print

OVERALL STEPS

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Applying first agarose sheet atop *gampi* barrier layer

Solution: Buffered solution, DTPA, and NaPO$_2$H$_2$·H$_2$O adjusted to pH 7.5 with NaOH

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Changing TEK-Wipe, saturated with rinse solution

(And having fun!)
Applying second agarose sheet atop *gampi* barrier layer

Solution: Buffered solution at pH 7.5 with lyticase enzymes

**OVERALL STEPS**

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- Final calcinated rinse, pH 8
- Mend tears
- Humidification/flattening as necessary
Changing TEK-Wipe, saturated with final rinse solution
Solution: Calcinated filtered tap water, adjusted to pH 8 with Ca(OH)$_2$

**OVERALL STEPS**
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- **Final calcinated rinse, pH 8**
- Mend tears
- Humidification/flattening as necessary
After final rinse, print was placed into a drying stack

OVERALL STEPS

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- Mend tears
- Humidification/flattening as necessary
RESULTS

“Le Ballon,” BT

“Le Ballon,” DT after bathing
RESULTS

“Le Ballon,” BT

“Le Ballon,” DT after bathing
"Le Ballon, DT after bathing
"Le Ballon, BT

"Le Ballon," DT after bathing
RESULTS
RESULTS

**Colorimetry**
Minolta CR-221

**Locations**
- 1° support
- 2° support
- 1° support foxing
- 2° support foxing
- $D_{\text{max}}$ image
- $D_{\text{min}}$ image
**RESULTS**

**Brightening**

Secondary support

- “Le Ballon” $\Delta L^* = 2.09$
- “Le Pigeon” $\Delta L^* = 2.03$
- “Le Ballon” $\Delta b^* = -2.10$
- “Le Pigeon” $\Delta b^* = -3.23$
RESULTS

**Brightening**

Secondary support
- "Le Ballon" $\Delta L^* = 2.09$
- "Le Pigeon" $\Delta L^* = 2.03$
- "Le Ballon" $\Delta b^* = -2.10$
- "Le Pigeon" $\Delta b^* = -3.23$

**Foxing Reduction**

Secondary support foxing
- "Le Ballon" $\Delta L^* = 4.94$
- "Le Pigeon" $\Delta L^* = 1.03$
- "Le Ballon" $\Delta b^* = -4.63$
- "Le Pigeon" $\Delta b^* = -0.91$
“Le Ballon”

“Le Pigeon”

UV

TEK-Wipe

Suction Table
DISCUSSION
<table>
<thead>
<tr>
<th>DELIVERY METHOD COMPARISON</th>
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<tbody>
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DELIVERY METHOD COMPARISON

SUCTION TABLE
- Quicker total treatment time

TIME

TEK-WIPE
- Longer total treatment time
DELIVERY METHOD COMPARISON

SUCTION TABLE
- Quicker total treatment time
- Conservator is active through the entire treatment

TEK-WIPE
- Longer total treatment time
- Conservator is active only for bath changes

TIME
INTENSITY
DELIVERY METHOD COMPARISON

SUCTION TABLE
- Quicker total treatment time
- Conservator is active through the entire treatment
- Gel functions as a reservoir

TIME

INTENSITY

MOISTURE CONTROL

TEK-WIPE
- Longer total treatment time
- Conservator is active only for bath changes
- Gel functions as a poultice
DELIVERY METHOD COMPARISON

SUCTION TABLE
- Quicker total treatment time
- Conservator is active through the entire treatment
- Gel functions as a reservoir
- Weight of gel and pull from suction

TEK-WIPE
- Longer total treatment time
- Conservator is active only for bath changes
- Gel functions as a poultice
- Weight of gel alone

TIME

INTENSITY

MOISTURE CONTROL

PRESSURE
DELIVERY METHOD COMPARISON

SUCTION TABLE
- Quicker total treatment time
- Conservator is active through the entire treatment
- Gel functions as a reservoir
- Weight of gel and pull from suction
- Potential for more rinse solution to be delivered in spray form

TEK-WIPE
- Longer total treatment time
- Conservator is active only for bath changes
- Gel functions as a poultice
- Weight of gel alone
- Each rinse solution application limited to amount needed to saturate TEK-Wipe

TIME

INTENSITY

MOISTURE CONTROL

PRESSURE

DEGREE OF RINSING
RESULTS

"Le Ballon," DT after bathing

"Le Pigeon," DT after bathing

TEK-Wipe

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<td>• Saturated TEK-Wipe provides more even wetting</td>
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PRACTICALITY COMPARISON

SUCTION TABLE

INSTITUTIONS

PRIVATE PRACTICE

TEK-WIPE
PRACTICALITY COMPARISON

SUCTION TABLE
• Requires investment in large, expensive equipment

EQUIPMENT

TEK-WIPE
• Requires smaller, more easily available supplies
PRACTICALITY COMPARISON

SUCTION TABLE
- Requires investment in large, expensive equipment
- Greater physical restraint and control of moisture

TEK-WIPE
- Requires smaller, more easily available supplies
- Gentler to delicate objects or those with dimensionality

EQUIPMENT

OBJECT NEEDS
PRACTICALITY COMPARISON

SUCTION TABLE
- Requires investment in large, expensive equipment
- Greater physical restraint and control of moisture
- Conservator must act quickly and know the object can withstand the pull of suction

TEK-WIPE
- Requires smaller, more easily available supplies
- Gentler to delicate objects or those with dimensionality
- Conservator has more time to monitor treatment and can safely treat delicate objects
PRACTICALITY COMPARISON

SUCTION TABLE
- Requires investment in large, expensive equipment
- Greater physical restraint and control of moisture
- Conservator must act quickly and know the object can withstand the pull of suction
- Requires electricity. TEK-Wipe can be used instead of blotter.

TEK-WIPE
- Requires smaller, more easily available supplies
- Gentler to delicate objects or those with dimensionality
- Conservator has more time to monitor treatment and can safely treat delicate objects
- TEK-Wipe is reusable and thus less expensive than blotter
Successful overall aqueous treatment of foxed chine collé

Sodium hypophosphite reducing agent

Lyticase enzyme to lyse fungal cell wall

Use of gel reservoir under suction
Characterization of Foxing

- XRF with small spot size
- Fluorescent stain for chitin on surrogate

Using XRF to detect iron presence

Foxing with fluorescent stains

Image courtesy Richard Wolbers
**FUTURE RESEARCH & APPLICATIONS**

**Treatment Protocol Modifications**

- Full immersion bath
- Local gel application on suction table
- Blotter washing
- Use of enzymes with higher activity
- Repeated steps/applications
- Hybrid TEK-Wipe/suction table treatment

Full immersion bathing

More materials prep for repeated steps
FUTURE RESEARCH & APPLICATIONS

Other Analysis

- Residue studies
- Chromatography
- Artificial aging
- Mechanical testing
- Conductivity tests
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