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An Investigation into Application Methods of Localised Dye in Textile Conservation

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## **ABSTRACT**

Localised dyeing is used by textile conservators to create visual infills for areas of loss with support fabrics. This dissertation reviewed and compared three resist methods for painting Lanaset® dyes on silk support fabric using dyes thickened with sodium alginate (SA) and sodium carboxymethyl cellulose (SCMC). Three methods of controlling dye application were used - Melinex® stencils, gutta, and cyclododecane - and the results evaluated for smooth colouring, level of definition, ability to prevent wicking, and depth of shade compared to immersion dyeing. The ease of application, preparation time, safety issues, and any special requirements were also considered.

Both thickeners helped to control wicking of the dyes. SA thickened dyes were easier to apply as they had higher viscosity, but also were more likely to result in blotchy colour or retain brushstrokes. SCMC thickened dyes created smoother, more even colour, but had a lower viscosity, making them more prone to wicking.

The investigation allowed for the formation of clear guidelines for the selection and preparation of materials, the mixing of dyes and thickeners, selecting depth of shade, and the fixing process to allow textile conservators to replicate the processes and achieve consistent and desirable outcomes.

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## TABLE OF CONTENTS

LIST OF FIGURES.....	8
LIST OF TABLES AND GRAPHS.....	9
INTRODUCTION .....	10
<b>Overview of Infills and Overlays.....</b>	<b>10</b>
<b>Research Questions, Aims and Objectives.....</b>	<b>13</b>
Research Questions.....	13
Aims and Objectives.....	13
<b>Background.....</b>	<b>14</b>
<b>CHAPTER 1: LOCALISED APPLICATION OF DYES FOR VISUAL INFILLS AND OVERLAYS OF TEXTILES: A LITERATURE REVIEW.....</b>	<b>15</b>
<b>1 Survey of visual infills and overlays : Four groups of treatments.....</b>	<b>15</b>
<b>2 Localised Application of Dyes on Substrates.....</b>	<b>19</b>
<b>3 Six Major Components of Localised Application of Dye.....</b>	<b>21</b>
3.1 Thickeners Used with Dyes.....	21
3.2 Dye Stock Solutions.....	21
3.3 Additives and fixatives.....	22
3.4 Stencils/ Guides/ Resists.....	22
3.5 Ways of Fixing.....	22
3.4 Removing Excess Dye After Fixing.....	24
<b>4 Conclusion.....</b>	<b>24</b>
<b>CHAPTER 2: DYES.....</b>	<b>26</b>
<b>2.1 Introduction.....</b>	<b>26</b>
<b>2.2 Lanaset® Dyes.....</b>	<b>26</b>
2.2.1 Fibre Reactive Dyes.....	26
2.2.2 Premetalised Dyes.....	27
2.2.3 Acid Dyes.....	27
<b>2.3 Qualities of Lanaset® Dyes.....</b>	<b>28</b>
<b>2.4 Silk and the Process of Dyeing.....</b>	<b>29</b>

2.4.1	Preparation of Silk for Dyeing.....	29
2.4.2	Additives.....	29
<b>2.5</b>	<b>Uses of Dyes in Textile Conservation.....</b>	<b>30</b>
<b>CHAPTER 3: MATERIALS</b>		
<b>3</b>	<b>Introduction.....</b>	<b>32</b>
<b>3.1</b>	<b>Materials and Preparation of Replicates.....</b>	<b>32</b>
3.1.1	Dyes.....	32
3.1.2	Thickeners.....	33
3.1.3	Method for Preparing Dyes.....	34
3.1.4	Substrate.....	34
3.1.5	Pattern.....	34
3.1.6	Brushes.....	36
3.1.7	Method of Preparing Silk.....	37
3.1.8	Applying Dye to Replicates.....	38
3.1.9	Steam Setting.....	39
<b>CHAPTER 4: EXPERIMENTAL PHASE ONE- THICKENERS.....</b>		<b>41</b>
<b>4.1</b>	<b>Sodium Alginate (SA).....</b>	<b>41</b>
<b>4.2</b>	<b>Sodium carboxymethyl cellulose (SCMC).....</b>	<b>41</b>
<b>4.3</b>	<b>Preliminary Trials.....</b>	<b>42</b>
<b>4.4</b>	<b>Methodology.....</b>	<b>43</b>
<b>4.5</b>	<b>Results.....</b>	<b>44</b>
<b>4.6</b>	<b>Conclusion.....</b>	<b>45</b>
<b>CHAPTER 5: EXPERIMENTAL PHASE TWO – BARRIER METHODS.....</b>		<b>46</b>
<b>5</b>	<b>Introduction.....</b>	<b>46</b>
<b>5.1</b>	<b>Aims.....</b>	<b>46</b>
<b>5.2</b>	<b>Methodology.....</b>	<b>46</b>
<b>5.3</b>	<b>Barrier Materials.....</b>	<b>47</b>
5.3.1	Melinex® .....	47
5.3.2	Gutta Resist.....	47

5.3.2.1	Process of applying gutta and setting dyes.....	48
5.3.2.2	Materials Testing.....	50
5.3.3	CDD.....	51
5.3.3.1	Application of CDD.....	52
5.3.3.2	Safety.....	54
5.3.3.3	Conclusion.....	55
<b>5.4</b>	<b>Procedure for Applying Dyes to Silk Using Barriers.....</b>	<b>55</b>
5.4.1	Melinex® .....	55
5.4.2	Gutta Resist.....	56
5.4.3	CDD.....	56
<b>5.5</b>	<b>Results.....</b>	<b>57</b>
5.5.1	Melinex® .....	57
5.5.2	Gutta Resist.....	59
5.5.3	CDD.....	60
<b>CHAPTER 6: EXPERIMENTAL PHASE 3 - DEPTH OF SHADE.....</b>		<b>64</b>
<b>6</b>	<b>Introduction.....</b>	<b>64</b>
<b>6.1</b>	<b>Aims.....</b>	<b>64</b>
<b>6.2</b>	<b>Methodology.....</b>	<b>64</b>
6.2.1	Immersion Dyed Samples.....	64
6.2.2	Localised Application of Dye.....	65
6.2.3	Comparison Between Immersion Dyed and Localised Application of Dye.....	67
<b>6.3</b>	<b>Results.....</b>	<b>67</b>
<b>6.4</b>	<b>Conclusion.....</b>	<b>70</b>
<b>CHAPTER 7: DISCUSSION.....</b>		<b>72</b>
<b>7.1</b>	<b>Aim 1.....</b>	<b>72</b>
<b>7.2</b>	<b>Aim 2.....</b>	<b>73</b>
<b>7.3</b>	<b>Aim 3.....</b>	<b>74</b>
<b>CHAPTER 8: CONCLUSION AND FURTHER RESEARCH.....</b>		<b>76</b>
<b>8.1</b>	<b>Conclusion.....</b>	<b>76</b>

<b>8.2 Further Research</b> .....	<b>77</b>
<b>BIBLIOGRAPHY</b> .....	<b>22</b>
<b>Appendix I - Materials and Suppliers</b> .....	<b>83</b>
<b>Appendix II - Risk Assessment and COSHH forms</b> .....	<b>83</b>
<b>Appendix III - Preliminary Experiments</b> .....	<b>103</b>
<b>Appendix IV - Immersion Dyeing: Steps for Dyeing the control samples of silk</b> .....	<b>105</b>
<b>Appendix V - Immersion Dyeing Sheet</b> .....	<b>106</b>
<b>Appendix VI – Material Safety Data Sheet for pēbēo water-based colourless gutta</b> .....	<b>107</b>
<b>Appendix VII - Materials testing of pēbēo</b> .....	<b>112</b>
pH Test.....	112
Oddy Testing.....	112
Azide Test.....	117
FTIR.....	117
<b>Appendix VIII – Material Safety Data Sheet for cyclododecane</b> .....	<b>119</b>
<b>Appendix IX - Oddy testing procedures</b> .....	<b>123</b>
<b>Appendix X - Photographs of metal coupons used in Oddy Test</b> .....	<b>124</b>
<b>Appendix XI - Photographs of Replicates in the study after steam setting</b> .....	<b>127</b>



## LIST OF FIGURES

- Figure 1: Example of conserved textile from the Karen Finch Reference collection
- Figure 2: Conserved areas with dyed fabric infill
- Figure 3: Conserved area with net overlay
- Figure 4: Timetable for dyeing silk with Lanaset®
- Figure 5: Four dye colours prepared with both thickeners
- Figure 6: Pattern used as a template
- Figure 7: Application of dyes with two thickeners
- Figure 8: Method of laying silk crepeline onto masking tape
- Figure 9: Method of attaching the silk to the Correx® frame
- Figure 10: Materials needed for applying dye to replicates
- Figure 11: Checking water boiler for condensation
- Figure 12: Wiping away excess condensation on the silk and frame
- Figure 13: Dye with sodium alginate at 3% w/v
- Figure 14: Dye with sodium carboxymethyl cellulose at 6% w/v
- Figure 15: Area where dye did not cover the entire weave structure
- Figure 16: Applying dye onto substrate with stencils
- Figure 17: Example of Serti style silk painting
- Figure 18: Applying pēbēo gutta onto silk crepeline
- Figure 19: applying dye onto substrate treated with gutta resist
- Figure 20: Structure of CDD
- Figure 21: CDD in solid form before heating
- Figure 22: Applying CDD to the substrate with a kitska
- Figure 23: Steaming samples with CDD under extraction
- Figure 24: Example of localised application where dye wicked under the stencil
- Figure 25: Dye extended over gutta resist
- Figure 26: White line between colours when gutta resist was used
- Figure 27: Substrate with dye and CDD before sublimation
- Figure 28: Area where CDD remained before applying dye
- Figure 29: Area where CDD was removed and dye reapplied before fixing with steam
- Figure 30: Effects of CDD at edges of fixed dye
- Figure 31: Immersion dyed samples of Lanaset® Blue 2RA

Figure 32: Hexagon pattern used in this research

Figure 33: Immersion dyeing

Figure 34: pH testing pēbēo gutta resist

Figure 35: the sealed test tubes after 'accelerated ageing'

Figure 36: Samples for Oddy test in Heraeus accelerating ageing oven

Figure 37: Coupons from silk with gutta - Front

Figure 38: Coupons from silk with gutta - Back

Figure 39: Details of corrosion on lead coupon

### **LISTS OF TABLES AND GRAPHS**

Table 1: Physical Properties of CDD

Table 2: Weight of dye with thickener required for 500 mm<sup>2</sup> surface area

Table 3: L\*a\*b\* readings comparing immersion dyed values with locally applied values

Table 4: Comparison of locally applied dye samples, washed vs. not washed in surfactant

Table 5: Oddy test results

Graph 1: Comparison of locally applied dye samples with sodium alginate and immersion dyed samples

Graph 2: Comparison of locally applied dye samples with sodium carboxymethyl cellulose and immersion dyed samples

Graph 3: Comparison of colour between samples with SCMC and SA in relation to immersion dyed samples

Graph 4: FTIR spectrum comparing pēbēo gutta to acacia gum

## INTRODUCTION

### Overview of Infills and Overlays

In textile conservation, support fabrics are used to provide strength and stability, compensate for loss, protect and restrain, and/or to impart a visual wholeness. These materials are chosen according to the function of the support and the method by which it is applied (i.e. stitched or adhered) but are broadly chosen to be sympathetic with the textile to be conserved in terms of fibre, structure, and weight.

Infills are fabrics used under the textile being conserved, as a patch or full support and are often but not always opaque. Overlays are placed over the top of the textiles, to protect and restrain the areas of weakness and are generally semi-transparent. Both types of support fabrics are dyed in order to render them visually unobtrusive and traditionally this colouration is achieved by immersion dyeing. Conservation dyeing follows well established methods and protocols using reliable dyes tested for chemically stability. In textiles where there is a pattern or areas of colour, often there is a need for local colouring of the support fabric, and the techniques available to textile conservators and choice of materials are less established.



Figure 1: Example of conservation involving overlay and infilled support fabric. Image by kind permission of the University of Glasgow.

Dyeing of fabrics allows textile conservators to integrate pieces of new stable support into the historic textile and as such, the 'visual' success of interventive treatment can depend on aesthetic choices. Overlays of silk crepe or nylon net can allow areas of damage or loss to be protected and contained while the fabric is sheer enough to allow the details of textile to be seen. The piece of crewelwork from the Karen Finch Reference collection in figure 1 displays both types of support fabrics: fabric infills placed under the embroidery, and sheer overlays of net. The solid support fabric closely matches the background in figure 2 and is unobtrusive while allowing the viewer to clearly tell it is not part of the original piece, and the dyed net overlay in figure 3 is a light neutral colour to support the crewelwork while blending with the background colour.



Figure 2: Arrow indicates conserved areas with dyed fabric infill. Image by kind permission of the University of Glasgow



Figure 3: Arrow indicates the edge of the net overlay. Image by kind permission of the University of Glasgow

The areas of embroidery covered by net in the figure 3 have a lighter overall appearance compared to the uncovered areas. With a localised application of dye to the net, the overlay could reveal the pattern of the embroidery more clearly by blending into the colours of the embroidery while remaining sheer enough to reveal the original handiwork of the embroidery. Localised application of dyes to support fabric can match areas of wear or ageing without causing the conserved area to appear conspicuously new or restored and without drawing attention to the loss.<sup>1</sup> In addition, infills in textile conservation are generally completed in a way that they can be identified by the viewer as additions and not lead to misreading the condition of the object. This is especially important in objects where the areas of loss or damage provide information about the textile's prior use or historical significance.<sup>2</sup>

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<sup>1</sup> Frances Lennard, Thórdís Baldursdóttir and Vicky Loosemore, "Using digital and hand printing techniques to compensate for loss: Re-establishing colour and texture in historic textiles," *The Conservator*, 31:1, (2010): 59.

<sup>2</sup> Lennard, Baldursdóttir and Loosemore, 56.

## **Research Questions, Aims and Objectives**

The overall aim of this research plan was to establish a clear protocol for the application of Lanaset® dyes to a silk crepeline substrate. This dissertation will impart information on technique of application, methods of colour delineation, and materials and supplies necessary. It was written in order to contribute to the body of knowledge about this process and make the process more accessible and efficient to practising conservators.

### RESEARCH QUESTIONS

What is the most effective method of controlling localised application of Lanaset® dyes applied to a silk crepeline substrate?

What are the optimal techniques of application in order to achieve reproducible results?

### AIMS AND OBJECTIVES

Aim 1: To evaluate mediums used by textile conservators to locally apply dye to support fabrics by:

- Reviewing existing practices of local application of dyes in textile conservation literature and building on previous research conducted at the Centre for Textile Conservation.
- Investigating thickeners used in textile conservation to control dye wicking when applying dye to support fabric.
- Visually evaluating processes used to locally apply and fix dyes on support fabric.

Aim 2: To explore barrier application methods that can be useful for future conservation treatments by:

- Comparing materials, utensils, and processes necessary for localised colouring.
- Experimenting with techniques borrowed from other areas of conservation or other disciplines.
- Using analytical techniques to determine the properties of barriers not currently used in textile conservation.

Aim 3: To identify a correlation between percentage of stock dye used in localised application and depth of shade produced by immersion dyeing.

### **Background**

This dissertation was a rare opportunity to build on the research of a previous Centre for Textile Conservation graduate, Caitlin Picard, who completed a dissertation in 2016 titled “Investigating the use of additives with Lanaset® dyes for localised colour application.” Her dissertation studied the lightfastness, wet and dry crocking- the tendency of the dye to rub off, and washfastness of this dye when fixed with steam. This dissertation continued her research by testing the ability to use this dye with barriers for multi-coloured applications of dye on silk, and prevent wicking or bleeding of dye using thickeners. Since lightfast testing, wet and dry crocking, and ageing tests were thoroughly conducted in Picard’s dissertation for these dyes, these experiments were not repeated due to time limitations. This investigation did not evaluate the uses of additives or fixatives when using localised dyes.

In addition to this source, a literature review of case studies using this technique established the current practices, and an evaluation of practical applications of this technique were conducted at the Centre for Textile Conservation. This research plan used quantitative and qualitative research methods to investigate the results of application and wicking properties of this dye when applied by brush and set with steam as measured by visual observation.

Research on how to optimise this process was conducted to inform conservation practice. The methods evaluated in this research could be useful to conservators treating an object such as a banner or flag to be viewed from both sides and allow for conservation treatment without obscuring either face. These methods also could be helpful when treating textiles such as costume or quilts containing patterning or different colours such that immersion dyeing cannot produce the desired details.

## **CHAPTER 1: LOCALISED APPLICATION OF DYES FOR VISUAL INFILLS AND OVERLAYS OF TEXTILES: A LITERATURE REVIEW**

This literature review surveys methods used in textile conservation to create infills and fabric overlays. As the existing literature on these methods is too vast to be completely discussed here, this review supplies a broad overview pertinent to understanding the techniques used to create infills which provide overall visual completion or continuity of pattern, and overlays that will protect damaged and fragile textiles while not diminishing the existing pattern. It begins with a chronological evaluation of published case studies and correspondence about the method of thickening dyes for localised application on support fabrics. It then compares practices from other adjacent fields that could inform this practice in textile conservation.

### **1 Survey of visual infills and overlays : Four groups of treatments**

There are several methods of creating support fabric that allow for the continuation of patterning and allow the conserved piece to be viewed as a whole. Two sources provided an overview of previous practices in textile conservation. Ballard's annotated bibliography provided thorough reviews of case studies from 1972-2008 creating visual infills and overlays including commercial fabric paints and inks, hand printing, use of thickened dyes, and inkjet printing.<sup>3</sup> Thüsing's 2000 dissertation was a thorough investigation of fabric paints, screen printing, and digital printing, and supplied useful correspondence from international conservators about their motivations and materials chosen as colouring media for localised colouration of support fabrics.<sup>4</sup> The reasons localised application of colour was chosen for treatments were because it was faster and less messy than dyeing, where discolouration or a previous repair mean a single colour will not minimise all areas of loss, when wear has created areas with different visual characteristics, and as an aesthetic approach to help a viewer read information from the object.<sup>5</sup> One conservator felt was 'on a par with the inpainting of losses in painting or objects conservation.'<sup>6</sup>

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<sup>3</sup> Mary Ballard, "Applying Colour Locally to Fabric for Use in Conservation: An Annotated Bibliography," *ICOM Textile Working Group Newsletter*, No. 25 (April 2008): 5-13.

<sup>4</sup> Kim Thüsing, "Camouflaging Areas of Loss in Patterned Textiles; Evaluating Textile Printing, Painting and Digital Imaging," (MPhil dissertation, Textile Conservation Centre), 64.

<sup>5</sup> Thüsing, 77.

<sup>6</sup> Thüsing, 72.



Objects treated by the conservators surveyed included double sided painted banners, delicate fabrics such as silk that could not be sewn, bedspreads, and objects where there were missing elements 'such as black dots from a calico.'<sup>7</sup> These conservators' main concerns about using localised application were about reversibility, long term stability of materials and testing for fading, off-gassing, crocking, and colour shifts,<sup>8</sup> and results that have a 'plastic like surface'.<sup>9</sup> The overall methods discussed in these and other textile conservation literature fell into four categories: digital printing, textile pigments, hand colouring methods and painting with dyes.

### 1.1 Digital Printing

Digital printing on fabric has been recently addressed in textile design and conservation as a method of creating clear and accurately patterned overlays for larger textiles where hand painting would be problematic.<sup>10</sup> This method was well documented by Hampton Court Palace conservators treating Queen Anne's cut and uncut velvet patterned state bed hangings.<sup>11</sup> The process of commercially printing a net overlay revealed the challenges of pattern distortion, colour matching by photograph and computer software upgrades. Additional costs, Oddy and lightfast testing of the inks was necessary, increasing the treatment time and equipment that is not always accessible to a textile conservator was required. Despite a long production time, there was less work done in the studio, and the use of computer aided design (CAD) successfully recreated the repeat pattern where it was lost or very worn. As CAD is already used in textile design, transferring these techniques to conservation could reduce 'weeks of skilled and tedious work to a matter of hours.'<sup>12</sup>

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<sup>7</sup> Thüsing, 74.

<sup>8</sup> Thüsing, 71.

<sup>9</sup> Thüsing, 72.

<sup>10</sup> Frances Lennard, Thórdís Baldursdóttir and Vicky Loosemore, "Using digital and hand printing techniques to compensate for loss: Re-establishing colour and texture in historic textiles," *The Conservator*, 31:1, (2010): 60.

<sup>11</sup> Maria Jordan and Libby Thompson, "Standing on the Shoulders of Others: Further Developments In Polychrome Patterned Nylon Net," in *Learning Curve: Education, Experience, Reflection, Forum of the ICON Textile Group*, London, 13 April 2015, ed. by Alison Fairhurst, 27 (London: ICON, 2015).

<sup>12</sup> Amanda Briggs and Gillian E. Bunce, "Breaking the Rules: Innovatory uses of CAD in Printed Textiles," *Ars Textrina* 24 (1995): 192-193.

## 1.2 Textile pigments

Pigments, provide a different attachment to fibres from dyes. Dyes bond chemically to the fibre whereas pigments are particles of colour that adhere to a substrate's surface via a binder which can be aqueous or solvent based.<sup>13</sup> Textile pigments have the advantage of being ready to use when purchased and the colours can be easily altered to match the object to be conserved<sup>14</sup> and Bricoprint (previously called Helizarin) is readily available and widely used in Britain.<sup>15</sup> Another advantage are the many methods of application – pigments can be silkscreened, spray painted, sponged, or brushed. The disadvantage of using pigments are the pigment and binder system can form a film which alters the drape of the fabric<sup>16</sup> and can yellow over time, become embrittled and possibly off-gas.<sup>17</sup>

## 1.3 Hand colouring methods

The third group of infill methods are manual applications of colour. These methods can also offer ease of colour selection and ability to create controlled and detailed patterning. Twenty-nine methods of application including photocopying and the use of artists' materials, such as acrylics, fabric paints, paint markers, and airbrushing were evaluated by Kaldany, Berman, and Sigurdardottir (1999) for their ability to create fine lines without feathering, and even and smooth coverage without stiffness. They were tested for crocking, lightfastness, washfastness, bleed resistance, and acidic off-gassing using AATCC procedures. Of the nine most promising methods, none performed consistently well in all stability tests, and it was concluded that each method would be useful for different treatments; for example use of photocopying as a patch where perfect patterning would be needed on an object that would never be subjected to cleaning with surfactant.<sup>18</sup>

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<sup>13</sup> Nancy Britton, "The Use of Textile Pigments in Conservation Applications," in *AIC Textile Speciality Group Postprints*, 7, ed. by P. Ewer and B. McLaughlin, 42 (San Diego, CA: AIC Textile Specialty Group, 1997).

<sup>14</sup> Britton, 43.

<sup>15</sup> Anna Harrison, Pippa Cruickshank, and John Fields, "Localised Colouring Agents for Textile Support Fabrics: An Investigation into their Colour-Fastness," *SSCR Journal* 12 (2001): 17.

<sup>16</sup> Britton, 42.

<sup>17</sup> Britton, 45.

<sup>18</sup> Mary Kaldany, Maria Berman and Sigurros Sigurdardottir, "Evaluating the Stability of Commercially Available Artists' Coloring Materials Used to Create Compensation Infills for Losses in Textiles," *Journal of the American Institute for Conservation*, 38 (1999), 455.

Frequently, acrylic paints are referenced as an immediate, low-cost manual application solution for painting on silk crepeline overlays.<sup>19</sup> This method successfully visually integrated losses on a 19<sup>th</sup> century quilt treated for display at Hampton House, a National Historic site in Maryland. The conservator was a trained artist with experience in a paintings conservation lab, so colour matching and mixing paints was a comfortable decision. She admitted that textile conservators might find painting an unconventional choice but assured readers that this treatment was easily reversible and necessary for the limited public display of this particular quilt.<sup>20</sup> This may be an ideal treatment for overlays on textiles with detailed patterning or for treatments on painted banners but can result in a stiffer texture which may not be appropriate in some pieces of costume where drape is essential to the design of the garment in addition the resulting treatment may supply a slight shine to the surface.<sup>21</sup>

#### 1.4 Applying dyes to substrates

Applying dyes to substrates such as silk can retain the flexibility and drape of the fabric and allow for patterning with several colours in one layer of fabric. The dye is absorbed by the fibres and is contained throughout the fabric and does not rest on the surface like acrylic paints or binder and pigment systems.<sup>22</sup> As the fabric is in direct contact with the object to be conserved, it is essential that the dyes used to colour the fabric are fast and physically and chemically stable. Using tried and tested conservation dyes has an added advantage of predictability in regard to colour fastness and chemical stability. Although there are more sophisticated methods used for visual infill such as digital printing, localised application of dyes on fabric may be more feasible since this process is not as expensive, does not require outside studios to complete the printing and can be completed using materials already

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<sup>19</sup> Joanne Hackett and Beth Szuhay, "Facing the future: The Use of Cyclododecane and Re-moistenable tissue paper in the Conservation of a painted silk flag," in *Tales of the Textile: The Conservation of Flags and other Symbolic Textiles*, North American Textile Conservation Conference 2003, *Preprints*, ed. by Jan Vuori, 167-175 (Albany, NY: NATCC, 2003).

<sup>20</sup> Susan Schmalz, "When patching is impractical: Nontraditional Compensation for Loss in a Quilt," *Journal of the American Institute for Conservation*, 38, No. 3 (1999), 388-389.

<sup>21</sup> Hackett and Szuhay, 167-175.

<sup>22</sup> Ágnes Tímár-Balázsy and Dinah Eastop, *Chemical Principles of Textile Conservation* (Oxford: Butterworth-Heinemann, 1998), 67.

existing in many conservation laboratories. Although this method is currently used, there is no standardised method of application.

## **2 Localised application of dyes on substrates**

As this dissertation will investigate the localised application of dyes, this section will look at the history of the literature specifically about this process. In localised application of dyes, a thickener is added to the dye to control wicking, and two products have been commonly used, sodium alginate (SA) and sodium carboxymethyl cellulose (SCMC). The literature shows that from 1995 to 2007 SA was the dominant method, and originated from Jan Vuori's work at the Canadian Conservation Institute.<sup>2324</sup> Her 1995 article "Painting Irgalan dyes onto silk crepe line" described her method of using a brush and fixing with steam, and is the most frequently referenced source about this method in publications by conservators in the United Kingdom, Germany, Canada and the United States.<sup>2526272829</sup> Many articles also look to Harrison, Cruickshank, and Field's 2001 treatment of an archaeological fragment of a baby's tunic at the British Museum using net overlays painted with SA thickened Lanaset® dyes for technical details on this method.<sup>30</sup> The most recent use of SA was in Germany in 2007 as thickener for Lanaset® in a study of lightfastness and wet and dry crocking.<sup>31</sup>

After 2002, SCMC was increasingly used more often than SA. Although Harrison and Vuori's articles are also cited in works from this time, the reason for selecting SCMC over SA is not explained. Conservators at the Australian War Memorial used SCMC as thickener when painting Lanaset® onto silk crepe line for overlays to contain and support fragile and faded

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<sup>23</sup> Jan Vuori, "Painting Irgalan dyes on to silk crepe line," *Textile Conservation Newsletter* 28 (Spring 1995): 5-8.

<sup>24</sup> Jan Vuori and Season Tse, "Lightfastness of Irgalan and Lanaset dyed silk: Immersion Versus Direct Application: Results of a Preliminary Study," *Textile Conservation Newsletter* 33 (1997): 14-18.

<sup>25</sup> Sarah Clayton et al., "Clear as Mud: How Cultural Significance Determines Preservation Choices," in *Tales of the Textile: The Conservation of Flags and other Symbolic Textiles*, North American Textile Conservation Conference 2003 *Preprints*, ed. by Jan Vuori, 28 (Albany, NY: NATCC, 2003).

<sup>26</sup> L. Becker and C. Hornig, "Partielles Färben von Protein und Cellulosefasern," *Zeitschrift für Kunsttechnologie und Konservierung*, Heft 1 (2007): 52-56 in Mary Ballard, "Applying Colour Locally to Fabric for Use in Conservation: An Annotated Bibliography," *ICOM Textile Working Group Newsletter*, No. 25 (April 2008): 5.

<sup>27</sup> Harrison, Cruickshank and Fields, 19.

<sup>28</sup> Helen Holt, "Further notes on painting with Irgalan dyes," *Textile Conservation Newsletter* 29 (1995): 3.

<sup>29</sup> Leslie Redman, "TCN Report on Painting Procedures," *Textile Conservation Newsletter* 28 (1995): 8.

<sup>30</sup> Harrison, Cruickshank, and Fields, 17.

<sup>31</sup> Becker and Hornig, 5.

silk ribbon bars on a military uniform. These materials were selected because they could be used for adhesive or stitch support and it was important to the integrity of the uniform to preserve and protect the colours of the ribbon bars.<sup>32</sup>

Most recently, in a 2008 article about painting an overlay for a ballet costume, Zagorska-Thomas used SCMC to thicken dyes, and attributed her approach to a 2007 unpublished student report by Alice Cole at the V&A Museum.<sup>33</sup> The methods described by Zagorska-Thomas have been influential in recent treatments, including a painted net overlay for Queen Anne velvet at Hampton Court Palace and Nora Meller's treatment of flags on a model ship at Royal Museums Greenwich.<sup>34</sup> The article was important to Picard's 2016 dissertation at the Centre for Textile Conservation (CTC) evaluating the stability of locally applied Lanaset® dyes on silk crepe-line by testing wet and dye crocking, wet bleeding and lightfastness.<sup>35</sup>

Many articles discuss localised application of Lanaset® on nylon net. These techniques apply to the application of this dye on silk, since Lanaset® can successfully dye both substrates.<sup>36,37</sup> The issues that arise concern the larger holes of the net as compared to the even and close weave of silk support fabric. This could account for differences in choice of concentration of thickener. One conservator at the Canadian War Museum experimenting on heavier fabric found penetration was more effective on thinner weight fabric.<sup>38</sup> In order to further evaluate the variables that influence the outcome of this process, it is useful to look more closely to the literature discussing the components used by conservators to locally apply dye.

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<sup>32</sup> Clayton et al., 28.

<sup>33</sup> Natalia Zagorska-Thomas, Natalia, "Bakst Where it Belongs: Reproducing Silk Painted panels of a Costume by L Bakst for S Diaghilev's Ballet Russes," in *Mind the Gap! Structural and Aesthetic Options for the Treatment of Loss in Textiles*, ed. by Alison Fairhurst, 76 (London: ICON, 2009).

<sup>34</sup> Private correspondence with Meller.

<sup>35</sup> Caitlyn Picard, "Investigating the Use of Additives with Lanaset® Dyes for Localised Colour Application," (MPhil Dissertation, University of Glasgow, 2016), 41-70.

<sup>36</sup> Harrison, Cruickshank and Fields, 16-20.

<sup>37</sup> Jordan, 27.

<sup>38</sup> Holt, 3.

### 3 Six major components of localised application of dye

Six components are mentioned that contribute to the success of the application: the thickener used with the dye, the stock solution of the dye, additives or fixatives, the physical application of the dye with any stencils or resists used to control the dye, fixing the dye, and rinsing excess dye from the substrate after fixing.

#### 3.1 Thickeners used with dyes

Most conservators have an established preference for using thickeners with dyes. Those that used SA most commonly used 3% w/v, with the exception of Harrison, Cruickshank, and Fields who preferred 4% w/v.<sup>3940</sup> Those that preferred SCMC used a range of 2-3% w/v with silk. All suggested the percentage needed to be adjusted to the thickness and weave of the fabric.<sup>41424344</sup> Authors suggested using as little thickener as possible so there was less to remove when rinsing.<sup>45</sup> When preparing the thickener and dye solution, most allowed the solution dissolve and thicken overnight.<sup>464748</sup>

#### 3.2 dye stock solutions

Lanaset® and Irgalan® were the most commonly used conservation dyes, and stock solutions ranging from 0.1% to 2% were used. Picard and Zagorska-Thomas used 0.1% w/v stock solution.<sup>49</sup> Zagorska-Thomas stated this produced the equivalent of a 1% immersion dyed depth of shade.<sup>50</sup> Meller determined 0.2 % w/v concentration with deionised water was ideal after experiments with 1% w/v resulted in the dye darkening at the edges of the painted design and leaving brushstrokes where the dye concentrated.<sup>51</sup> Vuori used a 2%

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<sup>39</sup> Harrison, Cruickshank and Fields, 17.

<sup>40</sup> Jan Vuori, "Painting Irgalan dyes on to silk crepe line," *Textile Conservation Newsletter* 28 (Spring 1995): 5-8.

<sup>41</sup> Jordan, 28.

<sup>42</sup> Personal correspondence with Meller.

<sup>43</sup> Picard, 29.

<sup>44</sup> Zagorska-Thomas, 76.

<sup>45</sup> Harrison, Cruickshank and Fields, 17.

<sup>46</sup> Ibid.

<sup>47</sup> Picard, 29.

<sup>48</sup> Personal correspondence with Meller.

<sup>49</sup> Picard, 29.

<sup>50</sup> Zagorska-Thomas, 76.

<sup>51</sup> Personal correspondence with Meller.

w/v stock solution, and after previous trials showed percentages lower caused the colour to dye unevenly, and Harrison also used 2%.<sup>5253</sup>

### 3.3 Additives and Fixatives

In most case studies, additives were not added to the dyes.<sup>5455</sup> In the work completed at the British Museum, sodium acetate (0.4% w/v) and acetic acid (0.8% w/v) were added to achieve pH,<sup>56</sup> and Picard's dissertation on the use of additives in localised application of dyes utilised Albeval Set (1%), sodium sulphate (2.5%) and acetic acid (1.5%).<sup>57</sup> The author's research concluded that additives were not required for long-term stability of Lanaset® dyes when locally applied and fixed with steam.<sup>58</sup>

### 3.4 Stencils/ Guides/ Resists

Few conservators mentioned stencils or resists. Many preferred to paint freehand onto the tensioned substrate<sup>59</sup> or used a template drawn onto Melinex® or Mylar® as a guide.<sup>6061</sup> Individual Melinex® stencils were used by Zagorska-Thomas for each colour and this method was chosen so the finished product would look hand painted like the originals.<sup>62</sup> Dye literature written for fibre-artists provided information on barrier products and resists including wax applied with a brush, or tjanting (a tool for batik) and gutta applied from a tube.<sup>63</sup>

### 3.5 Ways of Fixing

Several methods of setting the dyes were mentioned in the conservation literature about this process. Steam setting in either a horizontal or vertical position in a water boiler were

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<sup>52</sup> Jan Vouri and Season Tse, "Lightfastness of Irgalan and Lanaset dyed silk: Immersion Versus Direct Application: Results of a Preliminary Study," *Textile Conservation Newsletter* 33 (1997): 14-18.

<sup>53</sup> Harrison, Cruickshank and Fields, 17.

<sup>54</sup> Personal correspondence with Meller.

<sup>55</sup> Zagorska-Thomas, 76.

<sup>56</sup> Harrison, Cruickshank and Fields, 17.

<sup>57</sup> Picard, 30.

<sup>58</sup> Picard, 72.

<sup>59</sup> Personal correspondence with Anna Harrison.

<sup>60</sup> Kim Thüsing, "Camouflaging Areas of Loss in Patterned Textiles; Evaluating Textile Printing, Painting and Digital Imaging," (MPhil dissertation, Textile Conservation Centre), 64.

<sup>61</sup> Jordan, 28.

<sup>62</sup> Zagorska-Thomas, 76.

<sup>63</sup> Ann Milner, *The Ashford Book of Dyeing* (Christchurch: Shoal Bay Press, 1998), 96.

the most common methods of fixing the dyes.<sup>6465</sup> Using a large water boiler, the silk was mounted to a frame and rested horizontally on the top. In a Burshaw® water boiler or baby Burco, the silk on a frame is secured in a vertical position in the chamber.<sup>6667</sup> Concerns when steaming over the electric water boiler were the need to frequently blot the silk while steaming, the high temperatures of the steam, and the tendency of the dyes to run during fixing, especially when steaming vertically. Semi-permeable membranes of Goretex® and Sympatex® were attached to the inside of the lids to absorb steam and reduce excess moisture.<sup>6869</sup>

Other methods of fixing dyes were using a hand steamer,<sup>70</sup> an iron,<sup>7172</sup> a ‘pad-steam’ method, a pressure cooker,<sup>73</sup> and a steam box made of Coroplast® (corrugated plastic) with a nylon screen bottom which allowed the fabric to fix while loosely folded.<sup>74</sup> Additional ways of fixing mentioned during Thüsing’s survey included hot-air oven, laundry dryer at 70° C (158° F), vapour, and microwave.<sup>75</sup>

Steaming is also used in decorative arts and industrial printing. Silk painting literature detailed constructing a stovepipe steamer for steaming painted silk rolled up in newsprint, a method that is useful for silks of long lengths.<sup>76</sup> There was no mention of this method in conservation literature. Silk painting literature also recommended ironing, and microwaving as determined by the specific brand and type of dye used.<sup>77</sup> In industrial printing, ‘festoon’

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<sup>64</sup> Harrison, Cruickshank and Fields, 17.

<sup>65</sup> Personal correspondence with Meller.

<sup>66</sup> Jordan, 29.

<sup>67</sup> Zagorska-Thomas, 77.

<sup>68</sup> Harrison, Cruickshank and Fields, 17.

<sup>69</sup> Jordan, 29.

<sup>70</sup> Zagorska-Thomas, 77.

<sup>71</sup> Thüsing, 77.

<sup>72</sup> Becker and Hornig, 5.

<sup>73</sup> Ibid.

<sup>74</sup> Holt, 3.

<sup>75</sup> Thüsing, 77.

<sup>76</sup> Susan Louise Moyer, *Silk Painting: The Artist’s Guide to Gutta and Wax Resist Techniques* (New York: Watson-Guptill Publications, 1991), 134-136.

<sup>77</sup> Moyer, 137.



steamers or roller steamers are used for large-scale production and smaller works used smaller custom-built tower steamers, star steamers or flash 'agers.'<sup>78</sup>

### 3.4 Removing Excess Dye After Fixing

Some conservators preferred to wash the substrate after steam fixing. Picard washed the silk with Dehypon® LS45 surfactant after fixing to remove any residual dye.<sup>79</sup> She also ironed the silk to reveal any imperfections in the samples, but not to fix the dye.<sup>80</sup> Harrison, Cruickshank, and Field's methods included washing painted and fixed nylon net in Synperonic N and rinsing in distilled water as two colours of Lanaset® ran slightly without fixatives.<sup>81</sup> In silk painting literature, washing with synthrapol or professional dry cleaning was recommended to remove gutta or wax and bring back the pliability of the silk.<sup>82</sup> Other conservators like Zagorska-Thomas did not wash the silk after fixing, only rinsed with hot and cold water.<sup>83</sup>

## 4 Conclusion

The issue of patterning in infills or overlays has received little attention in the literature. Most articles covered the process of applying dyes to silk and pigments or dyes used. One article which addressed multicoloured infills for losses in wool clearly described using needle felting to physically create the missing pattern of a Hunkpapa Lakota dress,<sup>84</sup> but few others discussed the options available for recreating a pattern with dyes, although the need for multicoloured infills was apparent. Research into the technical processes required to create polychromatic patterning design is needed as it is a skill that could be utilised more often if conservators had all the information about these methods at hand.

Localised application of dyes has been chosen by textile conservators for creating infills and overlays because this method is more appropriate than immersive dyeing for double sided

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<sup>78</sup> Joyce Storey, *The Thames and Hudson Manual of Dyes and Fabrics* (London: Thames and Hudson, 1978), 163-167.

<sup>79</sup> Picard, 33.

<sup>80</sup> Ibid.

<sup>81</sup> Harrison, Cruickshank and Fields, 19.

<sup>82</sup> Moyer, 137.

<sup>83</sup> Zagorska-Thomas, 77.

<sup>84</sup> Anna Hodson, Susan Heald and Renate Maile-Moskowitz, "Hole-istic Compensation: Needle Felted Infills for Losses in Fulled Wool," *Journal of the American Institute for Conservation*, vol. 48, no. 1 (Spring 2009), 25-36.

and multi-coloured objects. It is possible with materials and supplies that are readily available in most conservation labs and does not require printing equipment or specialised knowledge of computer software and is less costly or time consuming. The specific methods of applying dyes to support fabric and sheer overlays is clearly a subject that requires further research, since very little has been published about the practical details of this useful method in conservation.

Differences in results using sodium alginate and sodium carboxymethyl cellulose as thickeners have also not been explored sufficiently in the literature despite the common use of one or the other by textile conservators when applying localised dyes. Questions remain about the application of dye and techniques involved. Established ratios of dye and thickeners would be beneficial to textile conservators to assist in colour and depth of shade as well as methods of creating barriers to prevent dye wicking in unwanted areas of patterning. Most conservators do not have the time or resources to closely evaluate the elements of this practice and devise optimal techniques for every treatment. The specific technical details are not generally discussed in published literature as conservators rarely publish all the trials and errors behind their treatments. This research corrects those oversights and argues there are benefits to bringing the practical and theoretical aspects of this technique together to inform future practices. This dissertation offers conservators details on thickeners, barrier methods, and techniques for localised application of dye which will allow for more treatment options.

## CHAPTER 2: DYES

This chapter explains the processes of immersion dyeing used in textile conservation and the qualities of Lanaset<sup>®</sup>, the dyes used in the experimental portion of this dissertation. Also addressed are the requirements for dyed support fabric used in conservation treatments in order to investigate the need for changes to existing processes.

### 2.1 Introduction

Dyeing fabric is a result of bonding coloured compounds to fibres. When organic fibres are immersed in water containing colouring matter they can absorb colouring matter, and when this colouring matter in a molecular or colloidal state of dispersion is heated, the matter will leave the water solution and attach to the fibres. The dyeing occurs as the colouring matter transfers to the fibres and the colour increases in the fibres while the dye bath becomes exhausted.<sup>85</sup> The quality of the dyed result is determined by its lightfastness, levelling (even penetration of colour), washfastness, and resistance to crocking. In textile conservation, the dye's chemical stability is also extremely important since the dye must not contribute to the conserved object's degradation.<sup>86</sup>

### 2.2 Lanaset<sup>®</sup> Dyes

The dye used in this dissertation is Lanaset<sup>®</sup> described as an 'intelligent mixture' by the manufacturer because it combines 1:2 premetalised, acid, and reactive dyes.<sup>87</sup> It is composed of two parts fibre reactive Lanazol<sup>®</sup> dye and one part Irgalan premetalised 1:2 dye.

#### 2.2.1 Fibre Reactive Dyes

Lanazol<sup>®</sup> dye is capable of brighter shades and very good fastness to light and wet treatments.<sup>88</sup> This fibre reactive part of Lanaset<sup>®</sup> dyes is capable of a solid chemical bond between the dye and fibre which makes it insoluble in water, hence the excellent

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<sup>85</sup>Max Simmons, *Dyes and Dyeing* (Melbourne: Van Nostrand, 1978), 2.

<sup>86</sup>Omar Abdel-Kareem, "Evaluation of the Dyes used in Conservation and Restoration of Archaeological Textiles," *Adumatu*, no. 20 (July 2009): 22.

<sup>87</sup>"Textile Effects 768002e: Lanaset<sup>®</sup> dyes," Huntsman, <https://www.textile-dyes.co.uk/lanaset.pdf> (accessed April 23, 2018).

<sup>88</sup>David G. Duff and Roy S. Sinclair, *Giles's Laboratory course in Dyeing*, (Bradford: Society of Dyers and Colourists, 1989), 25.

washfastness.<sup>89</sup> These dyes react as soon as the dye is adsorbed on the fibre and do not level in prolonged dyeing, therefore levelling auxiliaries such as Albegal B are necessary.<sup>90</sup> These auxiliaries coat the fibre and cause the dye molecule to move around the fibre looking for a dye site promoting even dyeing.<sup>91</sup>

### 2.2.2 Premetalised Dyes

The Irgalan dye portion is an acid dye that has been used on its own in conservation to dye support fabrics.<sup>92</sup> Premetalised dyes combine metal atoms with dye molecules. In the early 1900s, it was discovered that certain acid dyes mordanted with chrome before, during, or after the dye cycle improved washfastness. The mordant created a chemical attraction between the dye and the fibre which allowed the dye to bond with metal and fibre.<sup>93</sup>

From these mordant dyes evolved a dye that combined the mordanting and dyeing processes in one step. In these dyes, the dye and the chrome are combined in a water soluble compound. These dyes are described as premetalised (or metal-complex) since there is a metal ion attached to the acid dye molecule, and there are two types of premetalised dyes, 1:1 and 1:2, referring to the ratio of metal atoms to dye molecules.<sup>94</sup> These dyes allow the fibre to take up colouring in a single bath<sup>95</sup> and the advantages of such dyes are good washfastness, light fastness, ease of application, and equal colouring on silk and wool in the same dyebath.<sup>96,97</sup>

### 2.2.3 Acid Dyes

Acid dyes are so named because of the acid or acid-producing chemical added to the bath when dyeing protein fibres which results in the production of positive hydrogen ions (H<sup>+</sup>).

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<sup>89</sup> Linda Knutson, *Synthetic Dyes for Natural Fibers* (Colorado: Interweave Press, 1986), 31.

<sup>90</sup> Duff and Sinclair, 25.

<sup>91</sup> Ann Milner, *The Ashford Book of Dyeing: A Fibre-artists' Guide to the Chemistry of Colour*, (Ashburton, New Zealand: Ashford Handicrafts Ltd, 2007), 46.

<sup>92</sup> Vuori, 5.

<sup>93</sup> Knutson, 40.

<sup>94</sup> Knutson, 41.

<sup>95</sup> Sarah Foskett and Katharina Mackert, "Dyeing Techniques Manual," (Glasgow: University of Glasgow Centre for Textile Conservation, 2016), 37.

<sup>96</sup> Knutson, 150.

<sup>97</sup> Milner, 48.

The negative charge of the dye allows the acid's positive ions to attach to the negative portion of the fibre molecule. Then the negative dye molecule can bond with the positive ions on the fibre. This chemical bond is a salt link between the fibre molecule and the dye molecule.<sup>98</sup>

Acid dyes are the most commonly used dyes for protein fibres, and silk responds similarly to wool using these dyes with a few exceptions. Since there are fewer acid-combining sites  $\text{NH}_3^+$  on the silk molecule, silk will dye a lighter shade than wool if both are present in the same dyebath. Silk does not contain a cuticle like wool fibres and will respond more quickly to the dye molecule at lower temperatures. At higher temperatures in the dye bath, the dye molecule will transfer back into the bath, allowing for more intense colours of silk at lower temperatures. For this reason, it is not necessary to raise the temperature of the dye bath above 85° Celsius (185° F) when dyeing silk, which can preserve its strength and lustre.<sup>99</sup>

### **2.3 Qualities of Lanaset® Dyes**

Lanaset® dyes are currently produced by Huntsman Corporation, a manufacturer of chemicals including textile products and dyes. These dyes were originally produced by Ciba-Geigy and became available to the home dyer in 1985. They are now standard dyes used in textile conservation and are regularly used at the CTC. These dyes supply 'medium to high lightfastness, good wet fastness, a high exhaustion percentage and good reproducibility'<sup>100</sup> on protein fibres and nylon and come in 15 colours and 2 supplementary dyes that can be mixed to create any required shade.<sup>101</sup>

As each dye has different chemical variations, they have slight differences in properties of light- and wetfastness and solubility. For example, Red 2B is noted by Huntsman as sensitive to temperatures above 60° C (140° F),<sup>102</sup> and conservators have found that this dye was slightly faster on nylon net than other Lanaset® colours when steam setting without

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<sup>98</sup> Knutson, 29.

<sup>99</sup> Knutson, 28.

<sup>100</sup> Foskett and Mackert, 38.

<sup>101</sup> Huntsman, 37.

<sup>102</sup> Huntsman, 9.

a fixative in the rinse.<sup>103</sup> Yellow 2R has been known to separate and this dye stock solution should be stirred often before measuring or applying to prevent settling.<sup>104</sup> These differences between colours could affect the dyeing process.

## **2.4 Silk and the Process of Dyeing**

It is helpful to understand the structure of the substrate and the process of immersion dyeing in order to understand how silk will take localised dye application. Silk is a proteinaceous fibre composed of protein called fibroin. The fibroin of the silk fibre is made of twin filaments extruded by a silkworm when creating a cocoon.<sup>105</sup> Fibroin is composed of amino acids and contains  $\text{COO}^-$  and  $\text{NH}_3^+$  reactive groups.<sup>106</sup> Although fibroin contains the same reactive hydroxyl groups (-OH) as cellulosic fibres, these hydroxyl groups are located in the highly crystalline areas which are not as easily penetrated by dye as cotton or other cellulosic fibres. As a consequence, silk accepts dye differently than cotton or linen. The classes of dye that can be used on silk (and proteinaceous fibres) include the basic dyes, acid dyes, chrome mordant and premetallised dyes.<sup>107</sup>

### **2.4.1 Preparation of Silk for Dyeing**

In industrial dyeing, silk is degummed with Marseilles soap or enzymes before dyeing. This process removes the sericin and produces a soft supple feeling in the silk.<sup>108</sup> At the CTC, the silk is not washed or scoured before dyeing, and care is taken while dyeing to avoid too vigorous stirring, as agitation can damage the silk. Rough handling of silk can cause the twin filaments of silk to be ruptured and to project fibrils from the surface of the fibre causing patchy areas where light reflects unevenly from the surface of the fabric.<sup>109</sup>

### **2.4.2 Additives**

When immersion dyeing, additives are used, Albaflow<sup>®</sup> FFA-01 for wetting out, acetic acid to create the acidic environment in the dye bath, Albegal SET as levelling agent, and sodium

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<sup>103</sup> Harrison, Cruickshank and Fields, 19.

<sup>104</sup> Knutson, 154.

<sup>105</sup> Tímár-Balázsy and Eastop, 43.

<sup>106</sup> Knutson, 26.

<sup>107</sup> Knutson, 43.

<sup>108</sup> Abdel-Kareem, 23.

<sup>109</sup> Simmons, 21.

acetate to regulate dyebath pH. Glauber's salts (sodium sulphate) promote exhaustion and increase depth of shade by increasing the attraction between dye molecules and negatively charged fibres.<sup>110</sup> The timeline of dyeing silk with Lanaset® used at the CTC, is in figure 4 and the steps are described in greater detail in Appendix IV.

**DYEING SILK WITH LANASET**  
Silk has an isoelectric point of pH 5.1

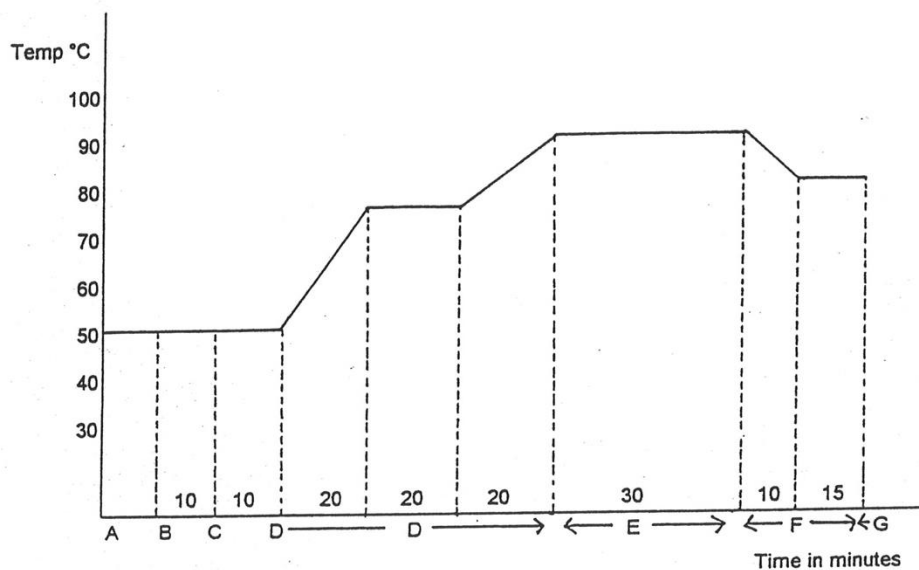


Figure 4: Time table for dyeing silk with Lanaset®<sup>111</sup>

**2.5 Uses of Dyes in Textile Conservation**

Although localised application of dyes is not a new technique, it does not have the established procedures or timeline of immersion dyeing. The same dyes may be used for both processes but when locally applied, they are used with thickening agents, and frequently additives and fixatives are not used.

Support fabrics created by both methods need to be evenly coloured, of a reproducible colour, fast to light and washing, and chemically stable. When support fabrics are immersion dyed, the dyes used have already been tested for wet and dry crocking to

<sup>110</sup> Picard, 18-19.

<sup>111</sup> Foskett and Mackert, 39.

prevent the possibility of harming a conserved object by adding a support layer or an overlay that transfers dye. New dyes (and all new substances) used in conservation in untried ways are subjected to testing to determine whether these substances will off-gas harmful chemicals or acidity that could have a detrimental effect on the object and those objects in its proximity.<sup>112</sup> Using dyes that have been tested for stability in immersion dyeing does not prove that these dyes will have the same properties when locally applied.

Although support fabrics are generally not washed, they still require washfastness since the object could be subjected to moisture after it is treated through humidification or a disaster such as a flood or fire sprinkler system. Picard's research into the stability of Lanaset® dyes locally applied evaluated this and three other areas: wet and dry crocking, and lightfastness concluding:

- Minimal to no dye transfer to white bleached cotton with and without additives during washfast testing. Red 2B without additives, and Blue 2R and Black B GR containing additives had greater changes after exposure to moisture as determined by colorimeter but changes were within an acceptable range between 4 and 5 on the CIE grading scale.<sup>113</sup>
- Wet and dry crocking tests showed minimal dye transfer from samples with and without additives within acceptable standards for conservation use.<sup>114</sup>
- These dyes with or without additives would not be recommended for localised application on support fabrics for long term display, since the colours shifted as a result of light ageing equal to 16 years of light exposure. Picard concluded that due to these dyes' sensitivity to light, they should only be used between 3-6 months for temporary exhibition between 5-10 years permanent display, if they are applied and set by steaming.<sup>115</sup>

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<sup>112</sup> Kaldany, Berman and Sigurdardottir, 452.

<sup>113</sup> Picard, 44-48.

<sup>114</sup> Picard, 49-58.

<sup>115</sup> Picard, 59-70.



## **CHAPTER 3: MATERIALS**

### **3 Introduction**

For this research, previous methods of localised application of Lanaset® dyes were reviewed and methods of the decorative arts of silk painting were investigated. Since the dyes used in silk painting are different from those used in this research and Lanaset® dyes with thickeners do not wick into the silk or bleed across the surface in the way silk painting dyes do, they do not allow for shading or layering of colour. Applying these dyes required trial and error.

Dyes were applied as the conservation literature on local application described and the procedures used by Picard were followed wherever possible, with a few exceptions. For example, the silk was not washed with surfactant after fixing, but instead the replicates were rinsed with soft tap water, and the dyes were applied with brushes rather than a roller to obtain detail.<sup>116</sup> As documented in Preliminary Experiments in Appendix III, there was no discernible difference in colour of locally applied Lanaset® dyes that had been washed in surfactant and those had not been washed. As there was no fugitive dye visibly released during the wash bath and the spectrophotometer showed no colour difference between the two sets, it was unnecessary to wash the silk after setting.

### **3.1 Materials and Preparation of Replicates**

This chapter outlines the materials, preparation techniques, and procedures for application of dyes related to this research.

#### **3.1.1 Dyes**

Four colours of Lanaset® dye were chosen to represent the colours produced by this line of dyes: a red, a blue, a yellow, and a black dye, since most colours can be derived from primary colours and the colours selected have slightly different molecular compositions from one another.

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<sup>116</sup> Picard, 31.

Two sets of dyes were prepared of the same colours used by Picard<sup>117</sup>:

- Blue 2 RA
- Yellow 2R GR
- Red 2B
- Black B GR

One set of dyes thickened with Sodium carboxymethyl cellulose (SCMC), shown in figure 5 in the larger beakers, used the same thickener as in Picard's research, but at a greater concentration of thickener, and the other set of four smaller beakers contained dyes thickened with Sodium Alginate (SA). This was done for ease of identifying while applying.



Figure 5: Each of the four colours prepared with thickeners

### 3.1.2 Thickeners

Two substances were chosen as dye thickeners for this research: SCMC and SA and to increase viscosity and hold the dye in place on the substrate. After preliminary experiments, 6% w/v of SCMC and 3% w/v of SA were chosen for the experiments for this project. The details are in Chapter 4: Experimental Portion: Phase One- Thickeners.

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<sup>117</sup> Picard, 29.

### 3.1.3 Method for Preparing Dyes:

1. Dyes were pasted cold with soft water for 0.1% w/v stock dyes (0.05g in 50ml) and left to thicken overnight.
2. Thickener was added slowly while stirring with a glass rod to prevent clumps.
  - a. 6 % w/v of SCMC (3 g in 50 ml) was added to each of the first group of four colours and stirred on the magnetic stirrer for 30 minutes.
  - b. 3% w/v of SA (1.5g in 50 ml) was added to the second group of four colours and stirred by hand with a glass rod. These were visibly thickened immediately.
3. Dye mixtures were left to thicken overnight.

### 3.1.4 Substrate

Since silk is a commonly used support fabric in textile conservation, silk crepe line, a very light weight loosely woven sheer silk, was selected as the support fabric studied in this research, as it can be used as support fabric with either adhesive or stitching treatments. Although silk can be scoured or prewashed without surfactant at 50° C (122° F) in softened water to remove any residue from the silk that may interfere with dyeing,<sup>118</sup> the silk used was not prewashed or scoured to preserve its quality.

### 3.1.5 Pattern

The pattern in figure 6 was used as a template for applying resists and for cutting stencils and was used to re-align the silks after steaming. When applying dye to this pattern, the top eight hexagons were painted with dye containing SA and the bottom eight with dye containing SCMC (see figure 7). This pattern was designed to include flat solid areas of dye and areas with no dye. Dye resistance was observed by comparing thicker and thinner lines of resist and areas where two colours were applied next to each other in the pattern. Variations of painting included solid planes of colour, straight edges, and sharp corners where the points of the hexagons met.

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<sup>118</sup> Ann Milner, *The Ashford Book of Dyeing* (Christchurch: Shoal Bay Press, 1998), 7.

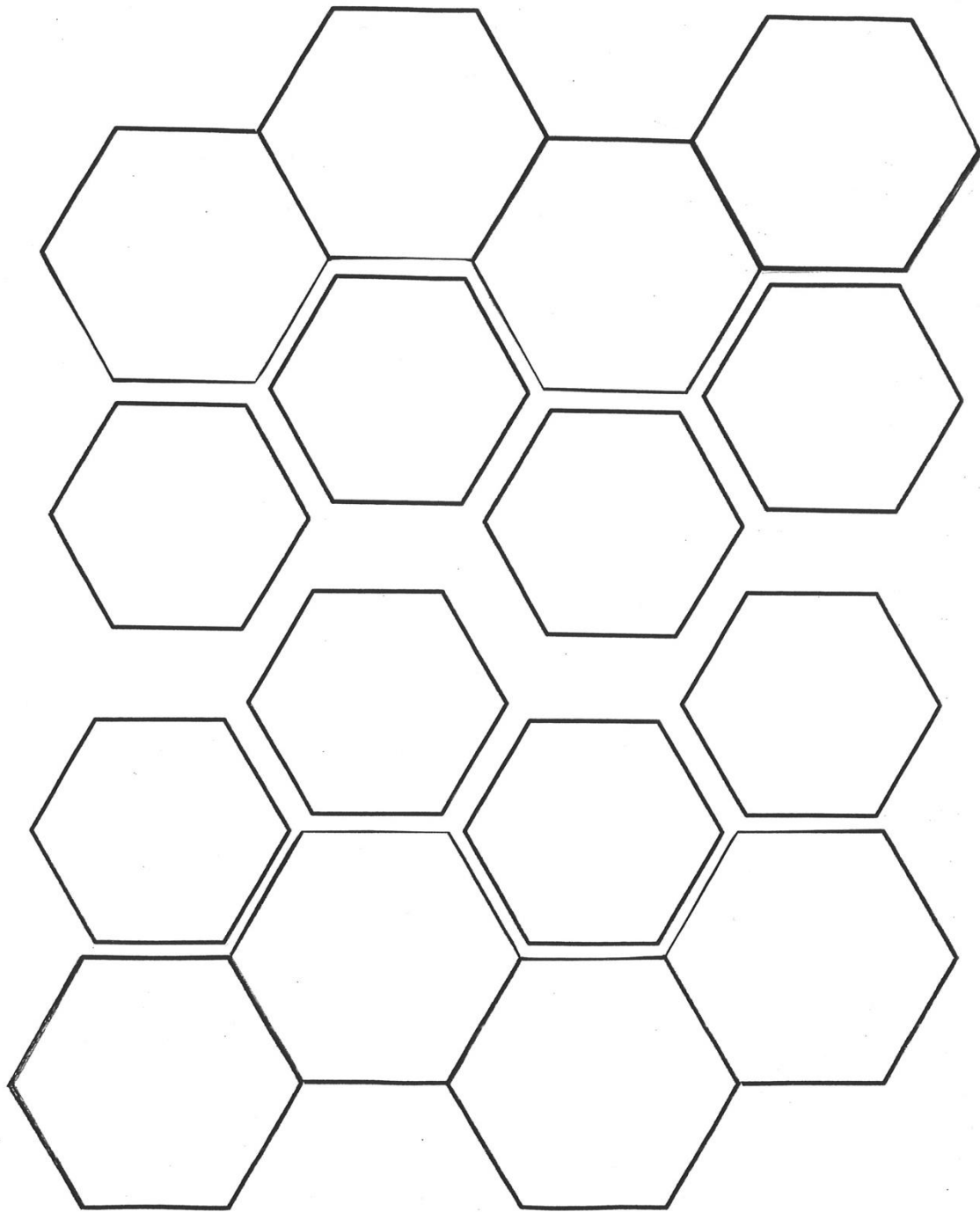


Figure 6: The pattern created for locally applying dye to silk crepe line. This was used as a template for lining up stencils and applying resists.

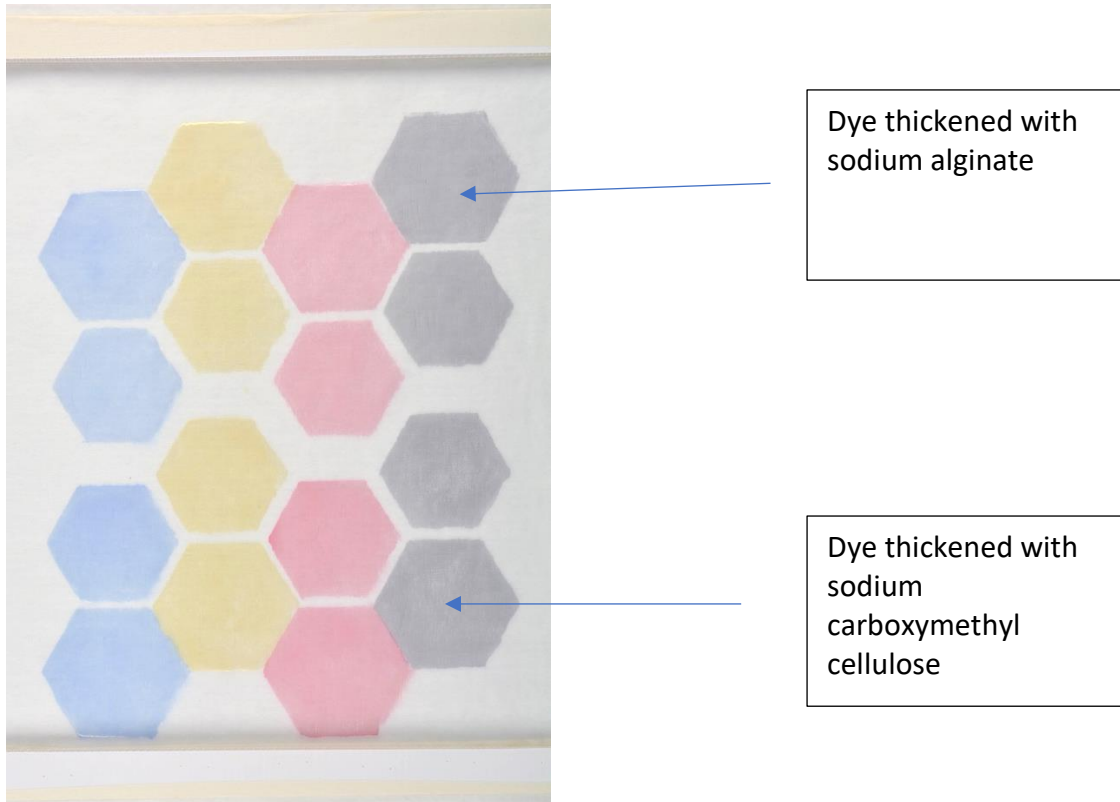


Figure 7: Application of dyes with two thickeners

### 3.1.6 Brushes

Squirrel hair brushes that come to a fine point were recommended in the silk painting literature for broad areas of colour. Small sable, prolene, or synthetic fibre watercolour round brushes were suggested for detail work since dye could be easily rinsed from the bristles.<sup>119</sup> Based on observations during preliminary experiments with natural and synthetic brushes, synthetic brushes were selected for this research. Round synthetic brushes with a pointed tip were used for applying dyes thickened with SCMC when using resists, and flat synthetic brushes were used for applying dyes thickened with SA. When applying dye using stencils with either thickener, flat synthetic brushes were used.

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<sup>119</sup> Susan Louise Moyer, *Silk Painting: The Artist's Guide to Gutta and Wax Resist Techniques* (New York: Watson-Guption Publications, 1991), 32.

### 3.1.7 Method of Preparing Silk



Figure 8: Laying silk crepeline onto masking tape

Method used to prepare silk crepeline. The silk was cut and stretched according to the guidelines in Moyers' *Silk Painting*.<sup>120</sup>

- A frame was cut from Correx® (corrugated plastic) with the outer measurement the size of the top of the electric water boiler and with 2-3 cm (.78 - 1.18 inch) borders.
- Silk crepeline was cut to fit the frame by pulling a thread across the warp and the weft to mark lines across the silk. The silk was cut along these lines to keep the silk squared on grain.
- Using 2.5 cm (1 inch) masking tape, four pieces of tape slightly longer than each side of the silk were placed sticky side up on the work surface.
- The silk was gently stretched and laid on the tape and smoothed from the middle out as seen in figure 8. The tape was released by cutting and applied to the opposite edge of the fabric.
- Each piece of tape was cut to the width and length of the silk and pressed to the Correx® frame starting from the centre of each side outward, adhering an adjacent side next, and going around the outside of the silk until it was stretched over the face of the frame smoothly . The process is shown in figure 9.

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<sup>120</sup> Moyer, 38-41.



Figure 9: Attaching the silk to the Correx® frame

### 3.1.8 Applying Dye to Replicates

Five samples of each method were completed to produce replicates for each of the dye applications in order to provide more statistically accurate information than a single sample would offer.<sup>121</sup> The completed dyed silk pieces were labelled and photographed after applying, and again after steaming. The materials assembled for applying dye to replicates are shown in figure 10.



Figure 10: Materials needed for applying dye to replicates

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<sup>121</sup> Terry J. Reddy and Chandra I. Reedy, *Principles of Experimental Design for Art Conservation Research* (Marina de Rey: Getty Conservation Institute, 1992), 44, <http://www.getty.edu/publications/virtuallibrary/089236243X.html>

### 3.1.9 Steam Setting

- In each method, the silk covered frame was placed horizontally on the top of a Grant SBB14 electric water boiler filled with 5 L of tap water and steam set over the opening for one hour.
- Although the original intention had been to keep the temperature under 85° C (185° F) to protect the silk, consistency with Picard's research and her findings for colour fastness and wet crocking were ultimately deemed more important. Therefore, the silk was steamed at 95-97° C (203-206.6° F). In the future it would be beneficial to steam at a lower temperature to protect the silk, and the resulting washfastness, lightfastness, and crocking of those samples should be tested and compared.
- Two small blocks of Plastazote® were placed on top of the frame under the lid in the front left corner and the rear right corner, as documented by Picard, to release steam and reduce condensation build up.<sup>122</sup>
- The lid remained on the water boiler while steaming but was lifted every 10 minutes and blotted for condensation gathering on the frame, silk, and lid, with care taken to prevent moisture from dripping onto the silk from the lid as seen in figures 11 and 12. After steaming, the silk was rinsed first with hot tap water to remove excess SCMC, SA, and gutta and then with increasingly colder water.
- The silk was dried while it was still stretched over the frame.

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<sup>122</sup> Picard, 32.





Figure 11: Removing the lid to check silk and remove condensation



Figure 12: Wiping away excess condensation on the silk and frame

## CHAPTER 4: EXPERIMENTAL PHASE ONE- THICKENERS

This chapter examines the two thickeners chosen for this research and information about the concentrations used.

### 4.1 Sodium Alginate (SA)

Sodium Alginate ( $\text{NaC}_6\text{H}_7\text{O}_6$ ) is derived from kelp and is the sodium salt of alginic acid.<sup>123</sup> It is used in conservation as an emulsifier of paints<sup>124</sup> and an adhesive, and industrially as a thickener and stabiliser in foods.<sup>125</sup> Alginates have been used in textile production as thickeners since the 1940s.<sup>126</sup>

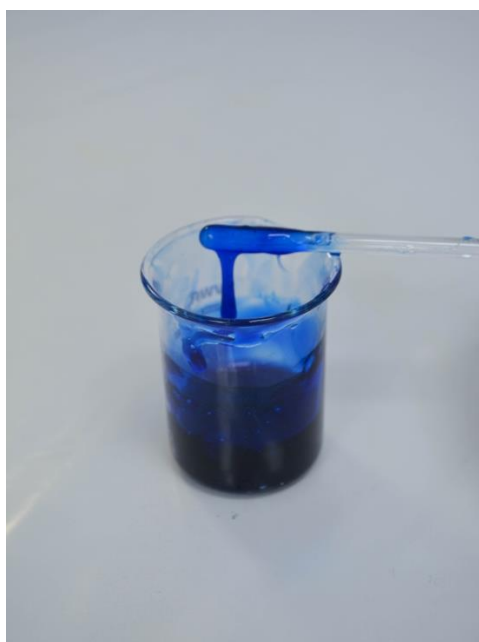


Figure 13: SA at 3% w/v

### 4.2 Sodium carboxymethyl cellulose (SCMC)

Sodium carboxymethyl cellulose is a water-soluble polymer composed of the sodium salt of alkaline modified cellulose. It is used in textile conservation as an adhesive and as a soil-

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<sup>123</sup>“Algin or Sodium Alginate,” AIC, [http://www.conservation-wiki.com/wiki/Adhesives\\_for\\_Paper#Algin\\_or\\_Sodium\\_Alginate](http://www.conservation-wiki.com/wiki/Adhesives_for_Paper#Algin_or_Sodium_Alginate) (accessed April 10, 2018).

<sup>124</sup> “Sodium Alginate,” CAMEO, [http://cameo.mfa.org/wiki/Sodium\\_alginate](http://cameo.mfa.org/wiki/Sodium_alginate) (accessed April 10, 2018).

<sup>125</sup> “Algin or Sodium Alginate,” AIC, [http://www.conservation-wiki.com/wiki/Adhesives\\_for\\_Paper#Algin\\_or\\_Sodium\\_Alginate](http://www.conservation-wiki.com/wiki/Adhesives_for_Paper#Algin_or_Sodium_Alginate) (accessed April 10, 2018).

<sup>126</sup> Storey, 95.

carrier during wet cleaning. It is also used in paper conservation as a sizing agent, mixed with cellulose powder for filling holes or losses, and as a deflocculating agent which promotes suspension of fibres in solution.<sup>127</sup> Industrially, it is used as a thickener of foods and detergents.



Figure 14: SCMC at 6% w/v

Both thickeners were mixed according to the suggestions found in the literature, and trial applications of dye were compared. As the SA formed a viscous paste immediately, only percentages of 3-4% were compared since the thickness suggested in the literature was satisfactory. The selected concentrations of thickeners are displayed in figures 13 and 14.

#### 4.3 Preliminary Trials

##### **Trial 1** - Comparison of 3% and 4% w/v SA

- Found 3% w/v used by previous conservators was sufficiently thick for use.

##### **Trial 2** - Comparison of 3%, 4%, 5%, 6% and 10% w/v SCMC

- 2-3% w/v were used by previous conservators.

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<sup>127</sup> Cathleen Baker, "Methylcellulose and Sodium Carboxymethylcellulose: Uses in Paper Conservation," *Book and Paper Specialty Group, AIC 10<sup>th</sup> Annual Meeting, May 26-30, 1982*, <https://cool.conservations-us.org/coolaic/sg/bpg/annual/v01/bp01-04.html> (accessed April 10, 2018).

- Trials used 3%, 4%, 5% and 6% to compare thickness. 10% was mixed to compare viscosity of a much higher concentration. Clearer definition with stencils was seen when higher concentrations of thickener were used.
- SCMC was always thickened overnight. No discernible difference in viscosity or texture was found between dyes thickened with SCMC and stirred for 30 minutes with the magnetic stirrer versus those stirred only by hand.

### **Trial 3 – Heavier Applications of Dye with SA and SCMC**

- SA resulted in blotchy and streaky colour fields because the thickener held the brush strokes. Areas where excess dye was scraped away with a small piece of Melinex® were smoother but still uneven in colouring after fixing.
- SCMC supplied a smoother application than SA, but the heavier application of SCMC pooled when applied. After steaming, red and black were slightly more blotchy than blue and yellow at a higher concentration of thickener.

### **Preliminary Trial Results**

During the three trial applications, the 2% SCMC recommended by the literature was very thin and watery even after stirring and thickening overnight. Higher percentages from 3-6% and 10% were tried and compared, as were different mixing methods (electric stirrer vs. by hand). A thicker consistency of SCMC than was recommended in the literature was needed to minimise the potential for dye bleed.

After the preliminary experiments, 3% w/v of SA and 6% w/v of SCMC were chosen for the experiments for this project. The 3% solution of SA had a thick texture like jam and the 6% solution of SCMC had a syrupy, more liquid, consistency.

### **4.4 Methodology**

Fifteen replicates containing both SA and SCMC were compared for the following:

- Texture of both thickeners
- Smooth, streaky, or blotchy colour
- Pooling of dye
- Differences between colours
- Details about effects of steaming process on dyes

## **4.5 Results**

Practice found that a thin layer of dye prevented pooling and reduced streaks or blotchiness. Allowing 30 minutes with a rotary fan for dye to dry before steaming was usually sufficient. Proper dryness was indicated by a shiny smooth surface on both sides of the substrate when peeled from the silicone release paper. The following observations were made:

- SA was more likely to leave streaky colour when applied too thickly - especially with Blue 2 RA, as it tended to retain the brushstrokes used when applying the dye.
- SCMC produced a smoother plane of colour overall but was more likely to pool in the centre of the shape, especially when Blue 2 RA and Red 2B were applied too thickly.
- Overall, Black B GR and Blue 2 RA were more opaque and smoother to mix. Red 2B and Yellow were more transparent and when mixed with SA, required more mixing.
- Red 2B and Black B GR had more watery consistencies with SCMC over all three batches of dye, which led to colour bleed.
- Dyes with both thickeners changed consistency only slightly when at a higher ambient temperature. Red 2B with SCMC wicked under two stencil samples and bled slightly with one gutta sample during application of dyes on the hottest days in the lab: 27 June at 23° C (73.4° F) with relative humidity of 35.6%, and 28 June at 27.6° C (81.7° F) with RH of 42.5% respectively when applied. In one sample with CDD applied on 28 June, Red 2B appeared watery in the centre and darker around the edges of CDD. On these same days, two applications of Black B GR thickened with SCMC, bled when applied inside a barrier of CDD.
- If the dye was applied to silk that was wet, the dyes bled regardless of the thickener used. Attempts to blot out areas where the dye had wicked were unsuccessful as the water used to wet out the unwanted dye allowed the adjacent dye to wick further out on the silk.
- Overall, SCMC and SA stayed in place when steaming. This was demonstrated in figure 15 in an area where the dye did not cover the entire weave structure but left blank spaces between two warps and two wefts in the weave, and these blank spaces remained after steaming. Due to the light depth of shade used, these areas were not very noticeable but did affect the outcome, as these areas were not completely filled in with colour.
- Both thickeners washed easily from the silk crepeline after steaming. Although the SCMC was used at a higher percentage than the literature recommended, it presented no difficulty in rinsing from the silk crepeline after steaming and five to six minutes of rinsing under hot then colder water cleared all excess dye and thickener from the silk.

- Drips of moisture onto dye from the lid of the water boiler that were quickly blotted during fixing left no mark. However, areas where moisture from the steam in the boiler pooled on the dye left uneven colouring.
- Some distortion of the weave occurred on the outer areas of the silk during steaming with both water boilers. It was most visible in the blue and black hexagons and may have been a result of the temperature of the boiling water affecting the substrate.
- Dyes could not be layered (an extra layer of dye applied atop a previous application) for darker depth of shade, as the second layer did not adhere to the previous layer smoothly and resulted in blotchy uneven colour after steaming.

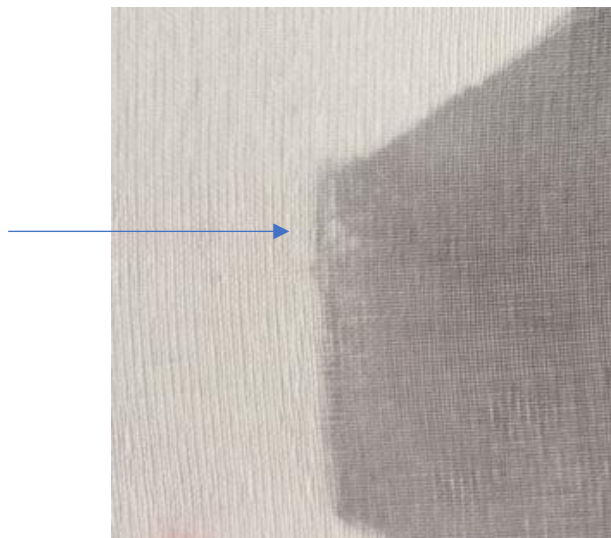


Figure 15: Area where dye did not cover the entire weave structure

#### **4.6 Conclusion**

Ultimately it was found that environmental factors such as temperature and possibly relative humidity can influence the consistency of thickened dyes, but by far the most important factor to the success of the end result was the appropriate thickness of dye to substrate and the use of a neat, careful application. The selection of brush was particularly significant with SA, as it was thicker and required a stiffer brush to apply into the fibres, where SCMC could be moved around the substrate easily with a softer brush since it had a lower viscosity. A higher concentration of SCMC in the dye could result in easier and still more controlled applications of dye.

## CHAPTER 5: EXPERIMENTAL PHASE TWO – BARRIER METHODS

### **5 Introduction**

When locally applying dye, many textile conservators follow a tracing of the area to be dyed under the substrate.<sup>128</sup> This method requires confident hand skills and comes with risks of applying dye in the wrong areas, uneven colour, or varying line quality. To make this process easier, others have created stencils made of Melinex® or other rigid materials to help guide their application.<sup>129</sup>

### **5.1 Aims**

In order to refine this process; i.e. make it easier and more efficient to replicate, three barrier methods were investigated for masking the areas which were not to be dyed - Melinex® stencils, gutta-resist, and cyclododecane (CDD).

### **5.2 Methodology**

Thickened dyes were applied with a paintbrush to fifteen pieces of silk crepeline stretched over Correx® frames – five using each barrier to create the same pattern. Each piece had four colours of both sodium alginate (SA) and sodium carboxymethyl cellulose (SCMC) thickened dye applied to it for comparison.

Each barrier method was evaluated based on:

- Ease of application
- Preparation time required
- Safety issues or special requirements
- Ability to have colours next to each other without bleeding into one another
- Ability to have thicker and thinner lines of resist with no dye bleed
- Level of definition in solid planes of colour including straight edges, and sharp corners where the points of the hexagons met.

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<sup>128</sup> Jordan and Thompson, 28.

<sup>129</sup> Zagorska-Thomas, 76.

### **5.3 Barrier Materials**

#### **5.3.1 Melinex®**

The first method of application was using a Melinex® stencil as per Thomas-Zagorska.<sup>130</sup> This is the only physical barrier that is not bonded with the substrate and there is potential for the dye to wick underneath it. Two stencils were cut with a craft knife following a printed out template to allow for half of the colours to be applied at a time, and the adjacent colours to be applied after the others had dried as seen in figure 16.

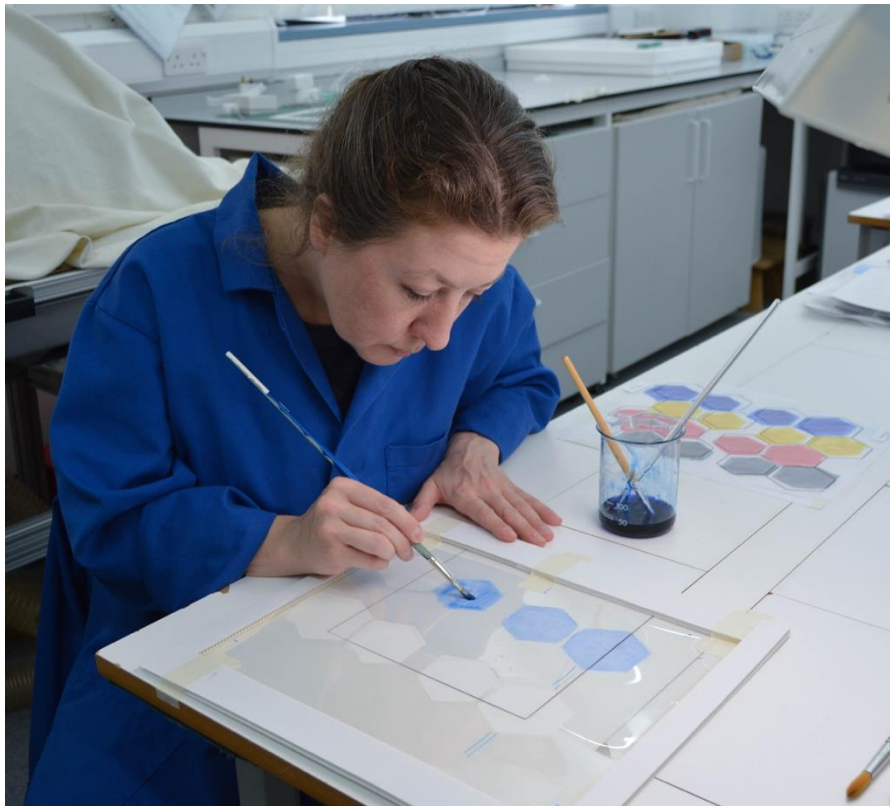


Figure 16: Applying dye onto substrate with stencils

#### **5.3.2 Gutta Resist**

Gutta resist is a commercially available paste that coats the fabric and creates a dye barrier. It is mouldable and pliable like rubber without its elasticity. It is produced for Serti style silk painting shown in figure 17, where it is painted onto silk to outline desired shapes and dye is

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<sup>130</sup> Zagorska-Thomas, 76.



allowed to bleed out to the line of resist, filling out the shape with colour. Many colours can be used successively since each area is enclosed completely by resist.

Figure 17: Example of Serti style silk painting

©Debby Hayes <http://paintandpattern.com/serti-silk-painting/>

Gutta resists are made from refined gutta-percha, a natural latex product from the coagulated milky sap of the palaquium tree native to Peninsular Malaysia, Sumatra, and Borneo. It has been used historically in golf balls, doll parts, dental fillings and electrical insulation.<sup>131</sup> Gutta as a resist is refined to a thin and rubbery texture which can be applied to fabric and dries as a dye-resistant and pliable line.<sup>132</sup> It has a thick rubbery texture, and must be removed from the silk after the painting process has been fixed. Many guttas can only be removed by solvent. For the purposes of this research a water-based gutta was selected so that it could be rinsed from the silk with warm water and will hold its thickness even when steam set.

#### 5.3.2.1 Process of applying gutta and setting dyes

*pēbēo* gutta was selected for this project due to its popularity with silk painters, and its global availability.<sup>133</sup><sup>134</sup> It has a narrow applicator tip built into the tube which allows for

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<sup>131</sup> "Gutta-percha," Getty Art and Architecture Thesaurus Online, <http://vocab.getty.edu/page/aat/300211567> (accessed June 10, 2018).

<sup>132</sup> Moyer, 25.

<sup>133</sup> Kaldany, Berman and Sigurdardottir, 445.

<sup>134</sup> Harrison, Cruickshank and Fields, 17.

simple and controlled application as seen in figure 18 and dye can be applied when the gutta is dry as seen in figure 19.

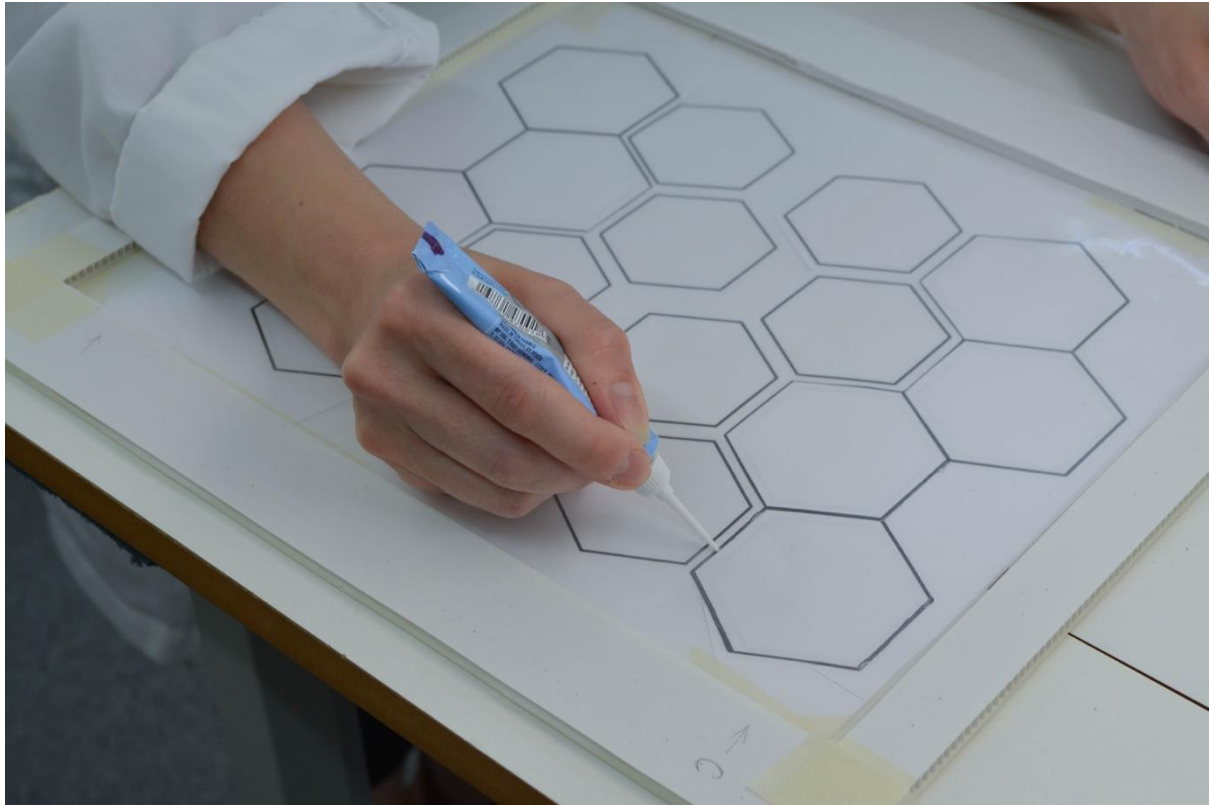


Figure 18: Applying pēbēo gutta onto silk crepeline

Colourless gutta was chosen for this research project as it leaves no residual colour on the substrate. As pēbēo is a commercial product, its contents are not 100% verified.

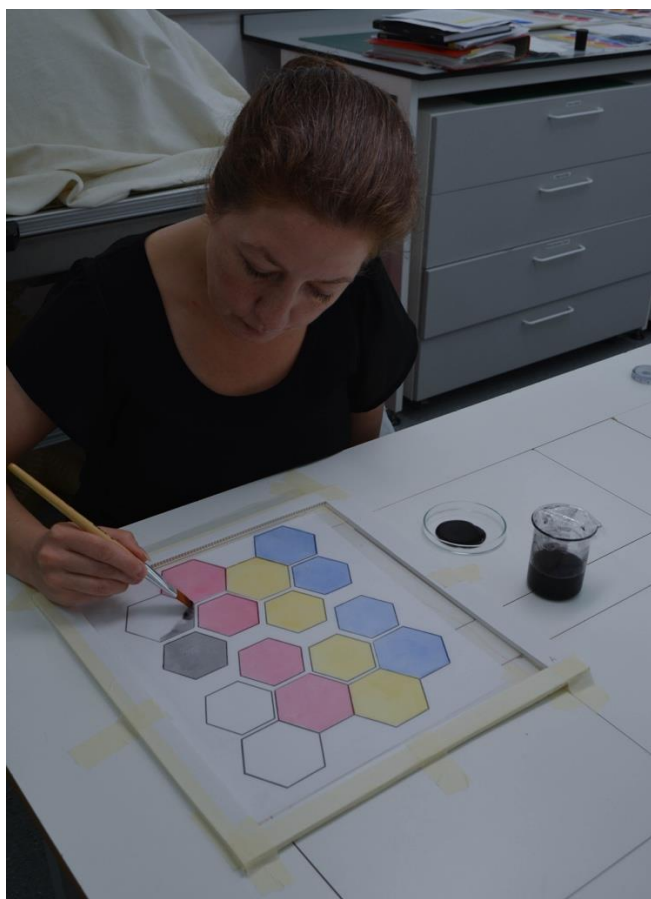


Figure 19: applying dye onto substrate treated with gutta resist

As gutta is not generally used in textile conservation, the chemical stability of pēbēo gutta was evaluated using pH, Oddy, and azide testing. It was also analysed with Fourier-transform infrared spectroscopy (FTIR) to determine what organic materials are present in the gutta before it is washed from the fabric.

#### 5.3.2.2 Materials Testing

As gutta is not generally used in textile conservation, the chemical stability of pēbēo gutta was evaluated using pH, Oddy, and azide testing. It was also analysed with Fourier transform infrared spectroscopy (FTIR) to determine what organic materials are present in the gutta before it is washed from the fabric.

It was determined that gutta is only slightly alkaline (within the recommended pH for textiles). However, both Oddy and azide testing suggests that there could be sulphides as well as other components present within the gutta that could potentially be emitted as

deterioration products, i.e. organic acids, with age. Finally, FTIR analysis suggests that pēbēo gutta is a natural gum product which could potentially age by hydrolysis and oxidation, becoming discoloured and brittle. It is therefore important that gutta is thoroughly rinsed from the silk to minimise the risk of its presence causing long-term deterioration. This testing and results are thoroughly discussed in Appendix VII: Materials testing of pēbēo gutta resist.

### 5.3.3 Cyclododecane

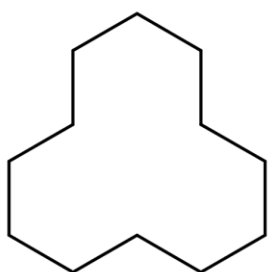


Figure 20: Structure of CDD

Cyclododecane (CDD) has been used in conservation since 1995 as a temporary adhesive, barrier, or consolidant to provide support for delicate objects. It is used as a masking material by paper and textile conservators to create a hydrophobic barrier over water-soluble inks and dyes to prevent bleed during wet cleaning. CDD ( $C_{12}H_{24}$ ), is a cyclic hydrocarbon that sublimates from a white waxy solid directly to a gas at room temperature over time leaving no visual residues and is considered by some to be totally reversible.<sup>135</sup> However, conflicting and unresolved information in the literature shows more research on CDD is needed to determine whether residue is actually left behind on the object after sublimation.<sup>136137</sup>

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<sup>135</sup> Sophie Rowe and Christina Rozeik, "Subliming Surfaces: The Uses of Cyclododecane in Conservation," *Studies in Conservation*, 53: sup 2 (2008): 17.

<sup>136</sup> Rowe and Rozeik, 28.

<sup>137</sup> Chris Waters, "Cyclododecane: A Closer Look at Practical Issues," *Japanese Institute of Anatolian Archaeology*, 16 (2007): 197.

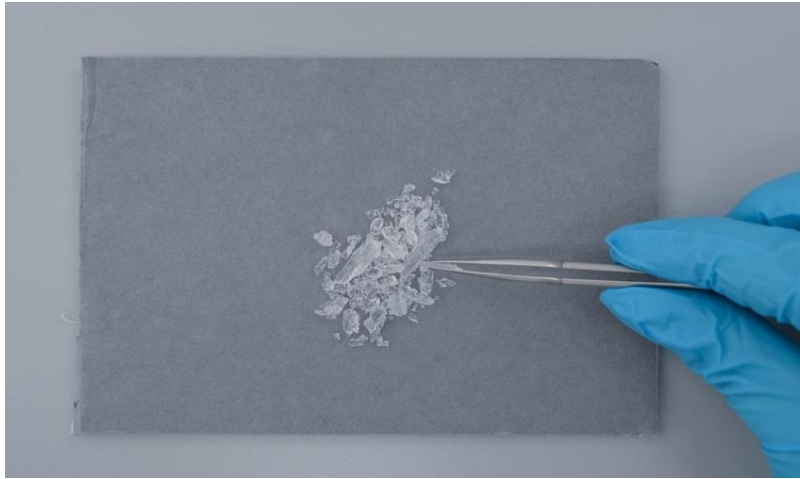


Figure 21: CDD in solid form before heating

There are three ways of applying CDD: as a melt, in a saturated solution, and as a spray from a heated spray gun. For this research, CDD was used as a melt to provide a barrier to locally applied dyes. It was applied to silk crepeline fabric with a kistka, an electric wax melting pen traditionally used for Ukrainian egg dyeing. The kistka was loaded with CDD (figure 21) via a metal chamber in the top of the pen. The substance melted as the metal heated and was released from the tip of the kistka like ink from a pen. It was applied as a narrow line of resist around the desired shape to prevent the applied dye from wicking. Once the CDD was applied, several colours of dye could be applied immediately and it was put in a fume cupboard to sublime once the dyeing was complete. Sublimation can take up to a week depending on the temperature, and Hangleiter showed that the rate of sublimation increases with temperature and occurs most quickly at temperatures over 30° C (86° F) with good airflow over the object.<sup>138</sup>

#### 5.3.3.1 Application of CDD

For CDD application on textiles, both Schraff and Nielsen's research and Majors' dissertation found the best results were obtained by applying a layer of melted CDD to a cold surface on the front of the textile and a second thinner application of melted CDD to a warm surface on

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<sup>138</sup> Rowe and Rozeik, 22.

the back.<sup>139</sup><sup>140</sup> Although this method was successful at protecting dyes from bleeding during wet cleaning, application on both sides of the textile was considered unnecessary for this research project. Due to the open weave structure of the silk crepe line, the CDD was able to penetrate through the fabric resulting in a layer on both the front and back.

CDD can also be heated in a small bain marie and applied with a paintbrush to create a larger area of resist. Majors found that a brush could apply more resist and to a larger area, but application with a kitska was more effective at preventing dye bleed when used to control fugitive dyes.<sup>141</sup> For this research, the method used by Hackett proved to be the most successful - drawing the tip of the kitska 'along the silk 1-2 mm from the edge' to be protected forming a 2 mm wide line of CDD as seen in figure 22.<sup>142</sup>



Figure 22: Applying CDD to the substrate with a kitska

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<sup>139</sup> Annemette Bruselius Scharff and Ingelise Nielsen, "The Use of Cyclododecane for the Fixation of Bleeding Dyes on Paper and Textiles: A Critical Evaluation of Application Methods," In *Dyes in History and Archaeology* 19 (2000) ed. by Jo Kirby et al., 153 (London: Archetype Publications, 2003).

<sup>140</sup> Gennifer Majors, "Sublime Applications: Creating an Efficient Cyclododecane Barrier on Textiles" (MPhil dissertation, University of Glasgow, 2014).

<sup>141</sup> Ibid.

<sup>142</sup> Hackett and Szuhay, 169.

### 5.3.3.2 Safety

The health and safety information published concerning conservation use of CDD is conflicting and often vague. Conservators have acknowledged inconclusive toxicity information but have continued to use the product nonetheless, despite the lack of safety testing for conservation.<sup>143</sup> Few specific precautions are mentioned in published literature, despite the use of CDD on large-scale projects in high quantities<sup>144</sup> and its wide-spread use in conservation has been misrepresented as evidence of its safety.

The main concerns about the safety of CDD are its effects on human health and the environment. There is a risk of fire when using melted CDD, and the risks associated with exposure to solvents when used with a solvent. Physical properties of CDD vary from source to source and conservators are urged to err on the side of caution. Table 1 compiled for the “Subliming Surfaces: Volatile Binding Media in Conservation” conference at Cambridge shows contradicting flash points and ignition temperatures. In particular, the product sheet from Kremer, the CTC’s supplier of CDD, shows a flash point of 98° C (208.4° F) where Kremer’s MSDS shows the flash point of 114° C (237.2° F).

Table 1: Physical Properties of CDD from Material Safety Data Sheets and information provided by manufacturers and suppliers<sup>145</sup>

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<sup>143</sup> Hackett and Szuhay, 169.

<sup>144</sup> Rowe and Rozeik, 25.

<sup>145</sup> Rowe and Rozeik, 18.

The long-term health effects from exposure to CDD are essentially unknown and literature states it poses a risk of being bioaccumulative, although it has not been proven to be toxic to humans.<sup>146</sup> There is a lack of rigorous safety testing of CDD and there is concern that the use of CDD in conservation will not be included in industrial studies of CDD as the uses of the product are very different than the controlled environments of industry.

Another issue is the testing of CDD has occurred as a solid or in solution but little has been studied of its effects when inhaled.<sup>147</sup> As of the 2015 Conference at Cambridge, research into the safety of CDD vapour was being conducted at University of California - Riverside by Dr Paul Ziemann. Unfortunately, this research has not been completed but it will be beneficial to the field of conservation if published.<sup>148</sup>

#### 5.3.3.3 Conclusion

These discrepancies in health and safety encouraged greater precautions while using this product despite the small amounts used during this study. Steps were taken whenever possible during this project which were outlined in the COSHH form (See Appendix II)

### **5.4 Procedure for Applying Dyes to Silk Using Barriers**

This section outlines each barrier method and the procedures required for application. Additional materials testing or requirements are also provided.

#### **5.4.1 Melinex®**

1. Melinex® stencils were used to apply dye to the tensioned silk crepe line. Two stencils were used to facilitate the application of two colours per stencil and allow drying time between colours. Adjacent colours were not applied at the same time, the lightest colours were applied first, and 30 minutes were given for the dyes to dry between stencils. A flat synthetic bristle brush was used throughout. A smooth shiny surface on both the front and back of the silk indicated that the colours were dry which took approximately 30 minutes.
2. The dried sample was then set by steaming in the water boiler and rinsed in hot then cooler water.

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<sup>146</sup> Rowe and Rozeik, 26.

<sup>147</sup> Ibid.

<sup>148</sup> Rowe and Rozeik, 27.



#### 5.4.2 Gutta Resist

1. A layer of silicone release paper was placed over the printed design template. The tensioned silk crepeline was laid on top and the gutta applied. A fan was used to expedite drying.
2. Each colour was applied after 30 minutes of drying gutta and a fan was again used to expedite drying.
3. The dried sample was then set by steaming on the water boiler and rinsed in hot then cooler water.

#### 5.4.3 CDD

1. A layer of silicone release paper was placed over the printed design template. The tensioned silk crepeline was laid on top and CDD was applied using a kitska. The application of CDD and dye was done under a Nedermann extraction hood as a safety precaution (see COSHH form in Appendix II).
2. After application, the silk was left in a fume cupboard for the CDD to sublime. Once it had completely visually sublimed,<sup>149</sup> the silk was steamed on the water boiler as above under extraction and rinsed in hot then cooler water.

This procedure is different from the other applications as CDD has a lower melting point than the temperature of the water boiler when fixing. The only way to know if the CDD had completely sublimed would be to weigh the silk before and after application of CDD.<sup>150</sup> Therefore, to prevent accidental inhalation of steam containing CDD, an extraction hood was used to remove any remaining CDD that sublimed during the fixing process as seen in figure 23.

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<sup>149</sup> Judged via visual assessment only.

<sup>150</sup> "Volatile Binding Media," AIC, [http://www.conservationwiki.com/wiki/Volatile\\_Binding\\_Media#How\\_do\\_you\\_know\\_the\\_CDD\\_is\\_gone.3F](http://www.conservationwiki.com/wiki/Volatile_Binding_Media#How_do_you_know_the_CDD_is_gone.3F) (accessed April 10, 2018).



Figure 23: Steaming samples with CDD under extraction

## **5.5 Results**

### **5.5.1 Melinex®**

Overall, Melinex® stencils worked well when used as a guide but not as a true stencil due to the fact that when/if the brush touched the Melinex®, the dye wicked underneath the stencil. With a careful, neat application of the dye, taking care not to brush too close to the stencil, this method could provide clear patterning. The thickness of the dye was an important factor for this method as SCMC wicked under the stencil more readily than SA. Figure 24 shows the result when dye was of a lower viscosity and wicked under the stencil as seen in the areas of red and black. This method does not require specialist equipment and is easy to carry out without former experience, unlike with gutta and CDD. There are also no health or safety issues.



Figure 24: Dye wicked under the stencil

However, this method is not the quickest to carry out - cutting stencils can be time consuming and their quality has a significant impact on the clarity of the resulting image. Intricate designs could require elaborate stencils. Furthermore, taping areas with masking tape to reinforce fragile areas did not work since the tape left residue and interfered with the application process.

Dyeing a pattern with Melinex<sup>®</sup> stencils was successful when colours were applied directly next to each other. Colour bleed was dependant on the thickness of the dye and careful application, preventing the brush from touching the edge of the stencil.

Melinex<sup>®</sup> stencils were capable of producing straight edges, sharp corners and curved lines dependant on careful application and dye of sufficient thickness. Thin lines of no colour were difficult with this method because of the readiness of the dye to wick under the Melinex<sup>®</sup> stencil, but thicker areas of no colour were successful as there were larger spaces between the areas of application.

Another drawback to using Melinex<sup>®</sup> is the possibility that stencils can be easily misaligned, resulting in overlapping colours and distortion of the pattern. In addition, the stencils need to be slightly larger than the desired shape if the dye is not going all the way to edge of the stencil. This can be resolved by keeping a printed pattern under a piece of silicone release paper below the substrate as dye is applied to guide the correctly sized application.

Stencils may be beneficial to a conservator to use as an aid to the free hand process of locally applying dyes to a substrate but, due to the wicking properties, they may not work well on their own as true stencils when made of Melinex®.

### 5.5.2 Gutta

Gutta resist successfully stayed intact during steaming and completely washed out in hot water afterward. It was moderately successful as a barrier material and had a medium ease of application, but it required practice and a steady hand. Application (straight from the tube) took 20-25 minutes and required no special safety precautions. However, there was a faint white line with feathery edges around many shapes where the dye was applied over the resist, which could be a concern. There were two difficulties with gutta: if the dye was not thick enough, it was easy to apply it past the resist. As it blended into the substrate it was difficult to see when applying dye, which encouraged the application of dye over the line of resist instead of up to it (figure 25). A thinner application made it easier to apply dye to the silk but it was difficult to apply a thin and even layer of gutta because of its viscosity.



Figure 25: Dye extended over gutta resist



Figure 26: White line between colours when gutta resist was used

Use of gutta resist allowed colours to be applied next to each other but there was some colour bleed for the reasons outlined above. With an application of 1-2 mm wide, only a slight white line between colours showed where the gutta had been, as seen in figure 25. As it is applied from a tube, a straight, even line can be difficult to achieve, as in figure 26. Thin and thick lines of resist were more easily obtained with gutta resist than with stencils but the crispness of line was difficult to control. Lines made on silk crepe line with gutta left a softer edge and less sharp definition than when using Melinex<sup>®</sup> to resist dyes thickened with SA and SCMC.

Unlike with stencils, small spots of excess dye could be wiped away before drying with a clean dampened paintbrush, and the dye from the shape did not bleed since it was retained by the gutta. Larger areas of excess dye were not removable as they had already permeated the fibres of the substrate. Without a resist such as gutta, the dye can wick out as a result of capillary action.

Further chemical testing of gutta resist should be conducted before wide-spread use in conservation as it is not entirely known what chemicals in this product could degrade the dyed fabric or transfer to the conserved object if left on the support fabric. Long-term effects such as yellowing are not known for this product, and users should be careful to rinse it completely from the fabric before drying and using for support fabrics.

### 5.5.3 CDD

CDD was successful at resisting the dye but it was difficult to apply. The rate of CDD released from the kitska was hard to control, and it set up very quickly, thus requiring very efficient application. The total time of application required was approximately 45 minutes including time to set up special safety preparations and equipment.

Applying CDD required practice to be able to move the kitska quickly without directly touching it to the silk. A wider application of CDD left chunks of CDD which distorted the intended pattern and took longer to sublime. Dye applied to areas with thick layers of CDD

also left odd marks in the dyed areas adjacent to the CDD. Figure 27 shows the substrate with CDD and dye before sublimation.



Figure 27: Substrate with dye and CDD before sublimation

An additional concern with CDD was that it tended to spread out beyond the line applied, creating a thicker application which resulted in a smaller area covered by dye because the resist covered more substrate, thereby distorting the pattern.

An even application of CDD was difficult and there is a risk of dripping CDD on the substrate. After repeating the process, it was found that drips could be removed after CDD dried by breaking the unwanted spot into pieces with tweezers and brushing it from the weave with a paintbrush before applying dye, leaving no sign of the spot after application. In areas where the dye was applied over a drip and allowed to sublime, a white spot remained where there was no dye ( figure 28). Attempts to apply dye into this area before steaming successfully eliminated the white spot but left the area slightly uneven in colouring (figure 29).

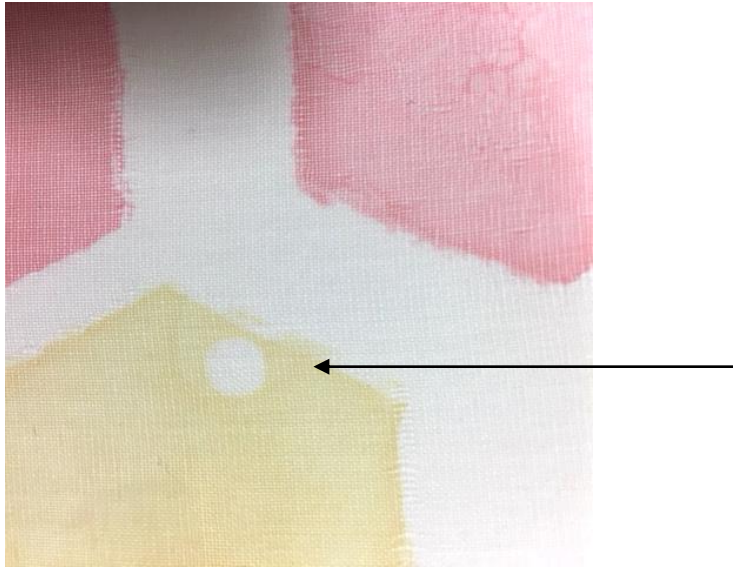


Figure 28: Area where CDD remained before applying dye

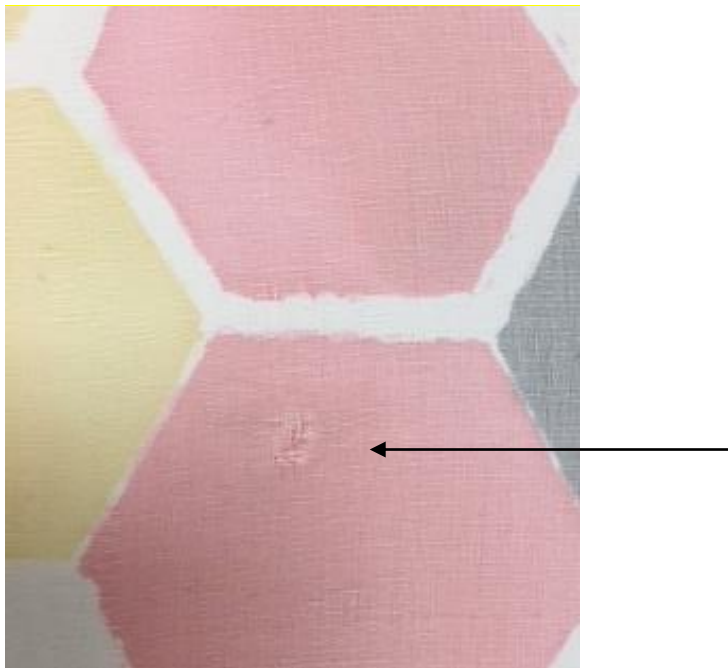


Figure 29: Area where CDD was removed and dye reapplied before fixing with steam

Creating a smooth line of CDD required practice since too much CDD released from the kitska expanded past the area of application. A 2 mm application with the smallest tip worked best for drawing lines but it was difficult to control the amount of CDD that was released from the tip. The application often distorted the weave of the silk when it bulked up on the substrate and pulled threads in the weave together. When the kitska was held

just above the substrate while applying CDD and the tip of the kitska was not allowed to touch the fabric but skimmed over the surface swiftly, a smooth even line was possible.

In terms of patterning, CDD was successful at separating colours next to each other but a thin line of no colour remained where the CDD had been. Thin and thick lines were possible but as the application was difficult to control, the edges of lines were often jagged and uneven. CDD also often left a brushy batik-like effect at the edges of coloured areas as seen in figure 30.



Figure 30: Effects of CDD at edges of fixed dye

Providing sharp corners and straight edges with CDD was possible but completely dependent on the conservator's skill, and curved lines would be difficult as it is hard to control the application of CDD. For a pattern requiring small circles or dots of resist, CDD applied with a kitska is an excellent choice, as discovered by the accidental drips on the silk crepeline.

In addition to the awkwardness of the application and the difficulty of controlling the flow of CDD from the kitska, CDD can spread out from the application and cover more substrate than desired which results in errors in the design/patterning. Because there is uncertainty about CDD's long term safety and effects on human health and environment, it should be used with precautionary measures such as ventilation and extraction.



## CHAPTER 6: EXPERIMENTAL PHASE 3 - DEPTH OF SHADE

### **6 Introduction**

When immersion dyeing, previous dye recipes are used to determine the desired colour and depth of shade. This can be problematic when locally applying dyes, because there is no clear or reliable conversion of recipes for this process. The results of this research could simplify the process of selecting colour and depth of shade for localised application using existing immersion dyed recipes. This chapter compares immersion dyed applications to localised applications of the same Lanaset® dye colour and investigates the correlation between depth of shade and percentage of dye stock used in localized application. The purpose of this analysis is to provide conservators with a guideline for selecting dye recipes for localised application.

#### **6.1 Aims**

To determine if a dye concentration of 0.1% in water applied locally produces approximately 1% depth of shade when immersion dyed as was suggested by Zagorska-Thomas in her treatment of a Ballet Russes costume.<sup>151</sup>

#### **6.2 Methodology**

Silk crepe line was immersion dyed at six depths of shade: 0.25%, 0.5%, 1%, 1.5%, 2%, and 2.5% using a 1% dye stock of Lanaset® Blue 2RA. A Konica Minolta Spectrophotometer CM-2600d was used to determine the colour of each of the immersion dyed samples (figure 31) and these values were then compared with samples of thin applications of dye. All samples were on white blotter paper background when readings were taken.

##### 6.2.1 Immersion Dyed Samples

Six depths of shade were immersion dyed following the steps outlined in Appendix IV and the dye recipe sheet is located in Appendix V.

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<sup>151</sup> Zagorska-Thomas, 76.

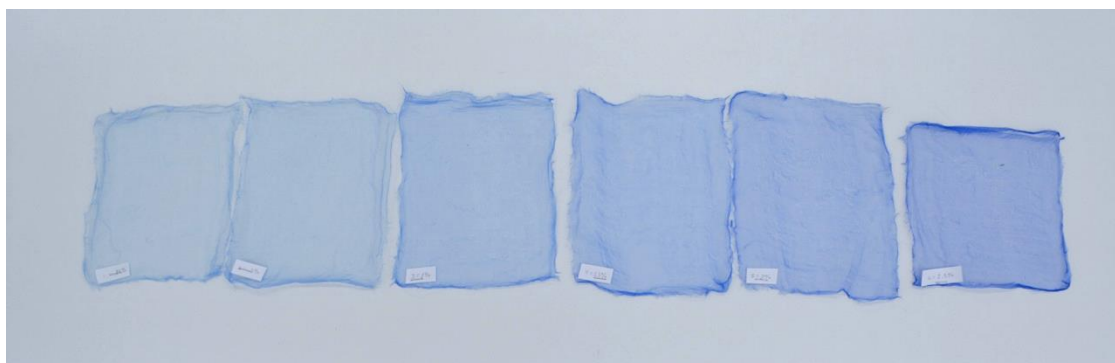


Figure 31: Immersion dyed samples of Lanaset® Blue 2RA

### 6.2.2 Localised Application of Dye

A thin application was defined as an application covering the surface of the substrate completely with no excess dye or pooling. For the pattern used in this research, 0.4-0.6 g of dye stock with thickener was required for each colour application as outlined in table 2, consisting of a pair of hexagons with surface area of 500 mm<sup>2</sup> as illustrated in figure 32. The pattern used in this research is discussed in detail in Chapter 3 Materials. This sample was fixed by steaming as previously explained in the same chapter.

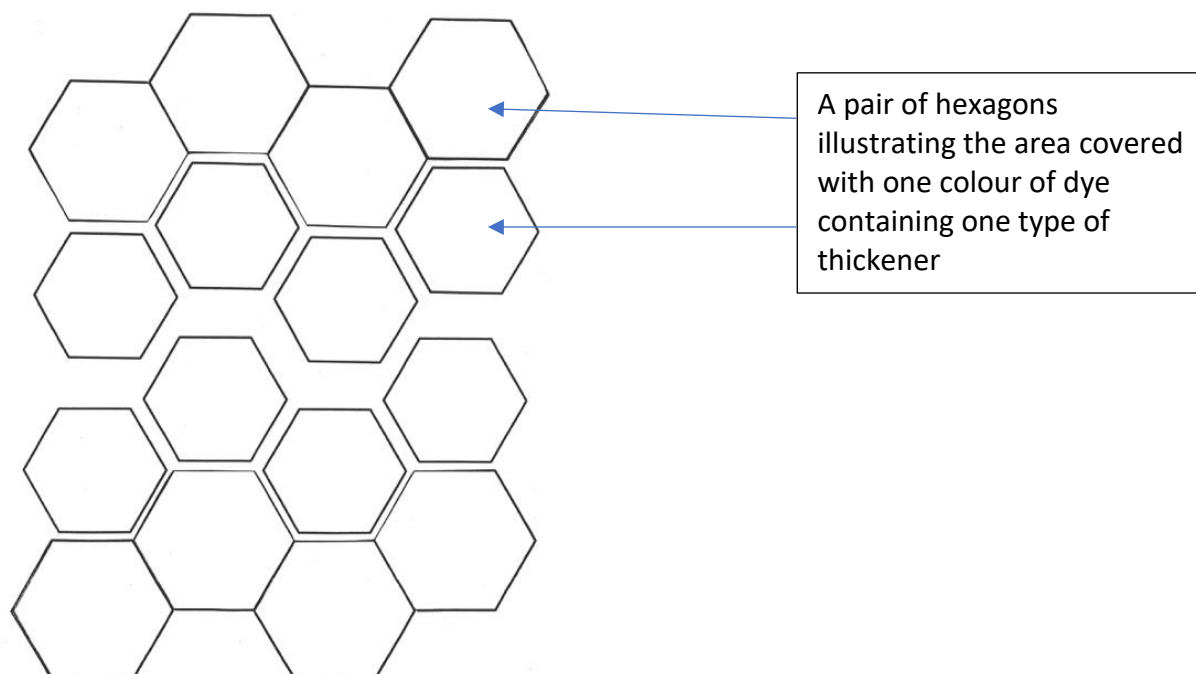


Figure 32: Hexagon pattern used in this research

Colour	Dye and SA	Dye and SCMC
Red 2B	0.509 g	0.577 g
Blue 2RA	0.482 g	0.583 g
Yellow 2R GR	0.556 g	0.559 g
Black B GR	0.619 g	0.623 g

Table 2: weight of dye with thickener required for 500 mm<sup>2</sup> surface area



Image 33: Immersion dyeing

### 6.2.3 Comparison between immersion dyed samples and the localised application

The spectrophotometer measured all samples as L\*a\*b\* coordinates according to the CIELAB colour-difference equation. This system approximates the differences in colour as seen by the human eye and depends on three variables: lightness, hue, and saturation.<sup>152</sup>

The L\* value represents the degree of lightness/darkness and ranges from 0, which is absolute black, to 100, which is absolute white. The a\* value represents the red-green axis, and b\* represents the blue-yellow axis.<sup>153</sup> The saturation is represented by  $a^{*2} + b^{*2}$ , which is the distance from the centre of the axis.<sup>154</sup> To study the difference between samples, Spectra Magic NX software was used to capture values of the immersion dyed samples and values of localised applications of Lanaset® Blue 2RA to compare them. The equation used for calculating the difference is  $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$  where  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$  represent the differences between those units in each sample.<sup>155</sup>

## 6.3 Results

- Three readings of each of the six depths of shade were taken, and values for each were averaged for an overall L\*a\*b\* value of each depth of shade. Three readings were taken and averaged from the locally applied samples with SA and with SCMC. Spectrophotometer readings found that despite the different thickeners, dyes with SA and the dyes with SCMC had the same depth of shade.
- Comparisons showed the locally applied colours were between 1% and 1.5% depth of shade with the averaged values closer to 1% depth of shade. The  $\Delta E$  value for 1% depth of shade compared to the locally applied dye samples was closest to zero (which would be no difference between colours) for both thickeners as seen in table 3. If  $\Delta E$  is lower than 3, the difference between colours is considered not visibly noticeable.
- Supporting Zagorska-Thomas' statement, the local application of 0.1% dye stock with thickener produced a colour closest to 1% depth of shade when immersion dyed using a

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<sup>152</sup> Brigitte Oger, "Fastness to light and washing of direct dyes for cellulose textiles," *Studies in Conservation*, 41 (1996): 130.

<sup>153</sup> Guff and Sinclair, 11.

<sup>154</sup> Oger, 130.

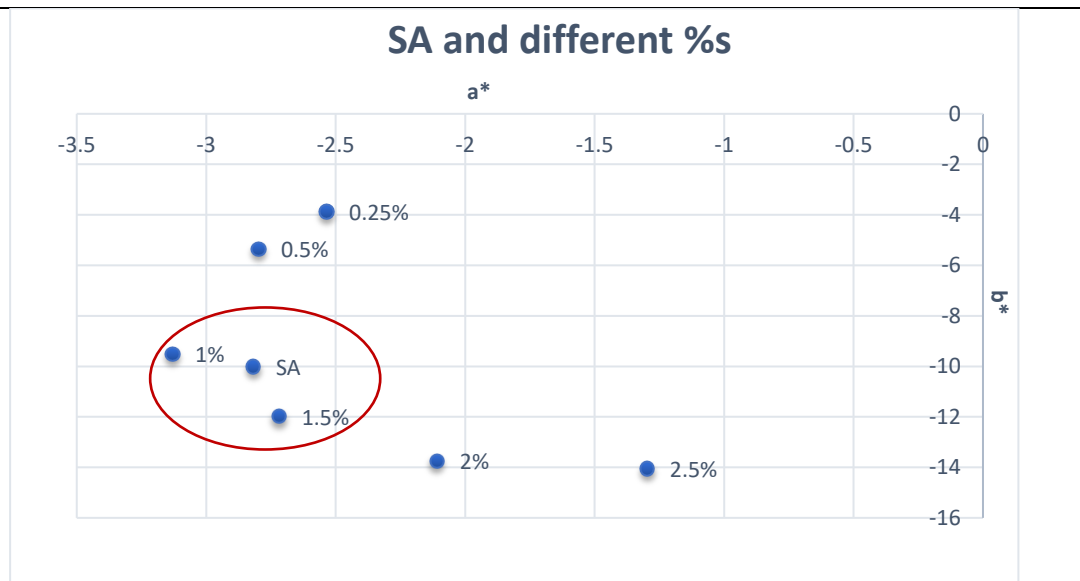
<sup>155</sup> Wilfred Ingamells, *Colour for Textiles: A User's Handbook*, (Bradford: Society of Dyers and Colourists, 1993), 152.

0.2% dye stock. The values shown in table 3 for SA vs 1% show the locally applied dye with SA had a -1.065 difference in lightness values (L\* reading) and was 0.312 less green (a\* reading), and 0.49 more blue (b\* reading) than the immersion dyed samples at 1% depth of shade.

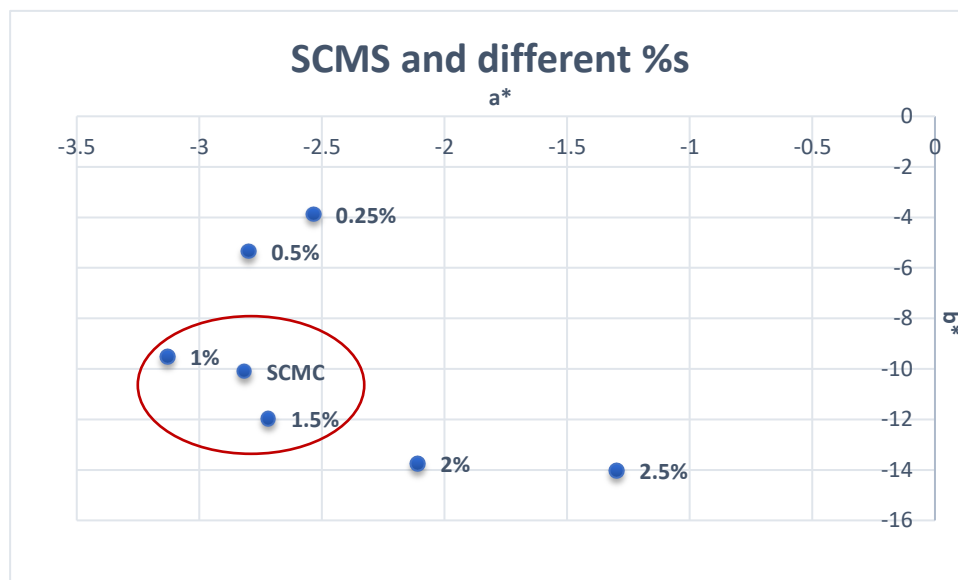
Comparison	$\Delta L^*$	$\Delta a^*$	$\Delta b^*$		$\Delta E$
SA vs .25%	-5.58667	-0.283	-6.127		8.296449
SA vs .5%	-4.555	-0.01967	-4.66033		6.51668
SA vs 1%	-1.065	0.312	-0.49033		1.213258
SA vs 1.5%	1.451667	-0.09967	1.969667		2.448848
SA vs 2%	3.266667	-0.70967	3.741333		5.017202
SA vs 2.5%	4.135	-1.52133	4.036333		5.975338
SCMC vs .25%	-6.05867	-0.281	-6.219		8.686908
SCMC vs .5%	-5.027	-0.01767	-4.75233		6.917782
SCMC vs 1%	-1.537	0.314	-0.58233		1.673343
SCMC vs 1.5%	0.979667	-0.09767	1.877667		2.120122
SCMC vs 2%	2.794667	-0.70767	3.649333		4.650655
SCMC vs 2.5%	3.663	-1.51933	3.944333		5.593184

Table 3: L\*a\*b\* readings comparing immersion dyed values with locally applied values

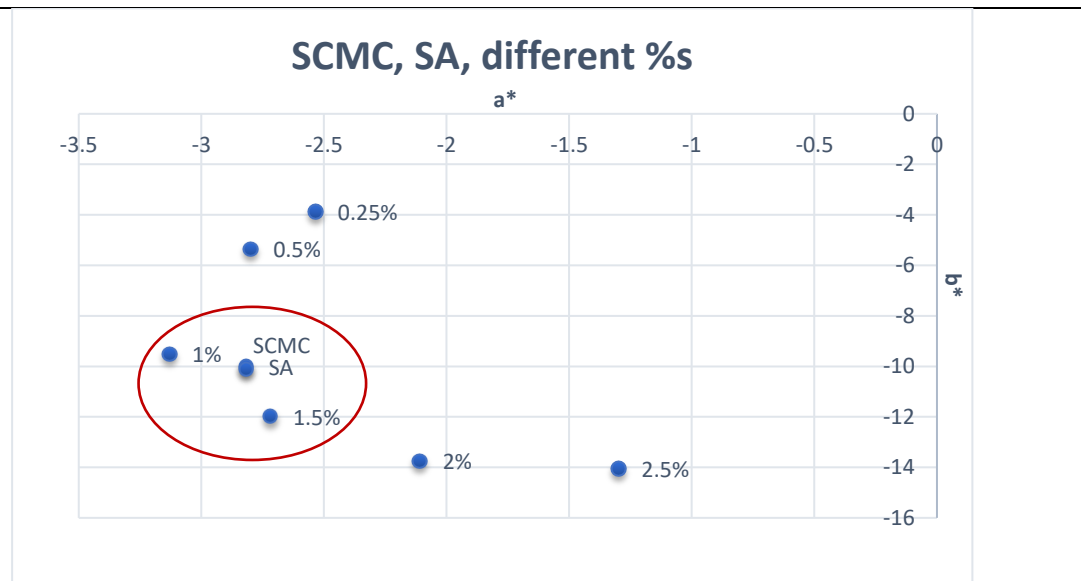
The immersion dyed samples for 1% and 1.5% are the closest to the locally applied samples in darkness/lightness and the closest on the red-green and blue-yellow axis as shown in graphs 1 and 2. From graph 3, it is clear that SCMC and SA have practically the same hue.



Graph 1: Comparison of colour between locally applied dye samples with sodium alginate and immersion dyed samples



Graph 2: Comparison of colour between locally applied dye samples with sodium carboxymethyl cellulose and immersion dyed samples



Graph 3: Comparison of colour between samples with SCMC and SA in relation to immersion dyed samples

#### **6.4 Conclusion**

The positive results of this research could simplify the process of selecting colour and depth of shade for localised application (without additives or fixatives) using existing immersion dyed recipes. A Lanaset® dye recipe (using a 0.2% stock) can be made up for localised application by using 1/10 of the % of desired depth of shade and colour for a close match. Based on these results, a desired depth of shade could be created by preparing 0.1% localised dye stock and it would be closest to 1% depth of shade of the immersion dyed samples. If 2% depth of shade was required, a 0.2% localised dye stock could be used.

The depth of shade could still be altered by thickness of application but a thinner application of dye is ideal as it results in smoother more even colouring. Application could be perfected through practice. Other colours would need to be tested since the steam fixing process could have different effects on the depths of shade of individual dyes.

The choice to use additives or fixatives would also influence the outcome of colour and depth of shade. Two issues identified by Picard were change in pH of Lanaset® dyes had

direct impact on depth of shade, and the colours were stronger in the samples with additives.<sup>156</sup>

It would be advisable for conservators to apply and set selected colours on a test piece of substrate before the actual piece to confirm that the match is accurate, since dyes thickened with SA and SCMC have a shiny surface before fixing with steam that could make it difficult to visually compare colours.

This guideline for colour selection could make localised application of dyes an easier and less time-consuming process when using Lanaset<sup>®</sup> because existing immersion dyed recipes could be used instead of guesswork when selecting a dye. The desired colour could be selected from previous immersion dye recipes and the amount of dye needed for localised application for that colour would be easily calculated by mixing a dye stock that is 1/10 the concentration of the immersion dye recipe (if the stock was prepared at the customary 0.2%).

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<sup>156</sup> Picard, 39.



## CHAPTER 7: DISCUSSION

This chapter addresses each aim presented in the Introduction and discusses the findings of this research in relation to each aim.

### 7.1 Aim 1:

Evaluate mediums used by textile conservators to locally apply dye to support fabrics.

After reviewing the current and previous practices in textile conservation literature on applying dye locally and conducting practical research into the two most commonly used thickeners, sodium alginate (SA) and sodium carboxymethyl cellulose (SCMC), it was clear that both thickeners reduced wicking of the dye into silk crepe line but had different textures. Having a choice of thickener allows the conservator to determine the viscosity and texture of the dye applied to the substrate and the quality of line or solid plane of colour.

SA had an almost grainy paste-like texture that was thicker than the SCMC and was easier to control. At 3% w/v, it was the consistency of fruit jam. This thickener stayed in place when it was applied by brush, so using a firmer and flatter brush assisted in applying straight lines and sharp corners. Due to its thicker texture and viscosity, it is possible SA could be used for more detailed painting or stamping at other concentrations. Research conducted by Jan Vuori using SA suggested the following concentrations could be used to create finer patterning with dyes:

- 2g in 125ml was sufficient for hand painting (1.6%)
- 4g in 125ml created a hard line good for stamping or printing (3%)
- 6g was good for painting (4.8%)<sup>157</sup>

Overall, SA thickened dyes would be more likely to hold brushstrokes and have streaks in planes of colour.

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<sup>157</sup> Private correspondence with Picard.

SCMC thickened dye created a more fluid line. At 6% w/v, it had a syrupy consistency much like a children's school glue. For conservators who prefer painting free hand, dyes with SCMC can be used for this purpose mixed to the texture of a thickened watercolour which does not wick into silk crepeline if steamed carefully and used at a concentration appropriate for the substrate. It created a smoother plane of colour overall.

The thickness of the application had an effect on the smoothness and depth of shade. A thicker application resulted in a darker colour after steaming and was more likely to be streaky or uneven. Certain colours provided difficulty during application with SCMC. Red 2B, for instance, wicked under the stencil in 2 of 5 applications, which may have been an effect of the steaming temperature. Red 2B and Yellow 2RGR were less opaque and some of the yellow dye appeared to settle to the bottom.

The process of fixing the dye by steaming over a water boiler was successful. After fixing the dyes, samples produced no dye bleed or visible colour loss when the substrate was washed in surfactant (in Appendix III). This process reduced the time required to dye fabric to just the time required for application of dye and an hour to fix by steaming. This eliminated the need for standing over the water boiler for 2 hours wiping constantly, since condensation cleared from the inside lid and silk every 10 minutes was sufficient. This was a simpler process than immersion dyeing and a more efficient use of studio time. Constant stirring, calculating additives, and careful monitoring of temperature were not required as one temperature was used for the entire fixing process and no additives were used.

## **7.2 Aim 2:**

Explore barrier application methods that can be useful for future conservation treatments.

Melinex® stencils, gutta resist and CDD were compared. It was found that Melinex® had the greatest ease of application despite the time required for preparing stencils since it was the easiest barrier method to control and required no previous experience, special equipment, or safety preparations. All barriers could allow for colours to be directly next to each other

without dye bleeding if the dye was thickened effectively. However, Melinex® had the highest amount of colour bleed due to application when the dye wicked under the stencil but, with a controlled application, this could be reduced. All methods were able to create thin and thick unbroken lines where there was no dye but the quality of line and level of definition varied between barriers. With practice, gutta resist could be a useful dye barrier if further testing of the long term effects of this product were conducted.

Analytical techniques of FTIR attempted to determine what organic materials are present in the gutta before it is washed from the fabric but were unable to determine how this commercial product will age or degrade over time if small amounts remain in the support fabric when used for localised application of dyes.

### **7.3 Aim 3:**

Develop tools and explore techniques of painted application, including depth of shade.

Materials, utensils, and processes necessary for localised application of dye were evaluated, and techniques from other areas of conservation and the field of silk painting were utilised. Steps were outlined for successful application of dyes and choice of materials including substrate, brushes, and preparation of substrate on a frame, and the mixing of dyes and thickeners. The method of steaming over a water boiler used by Picard was optimised and it was found helpful to leave the top 5 cm (2 inches) of the silk free of pattern since moisture collected on the frame at the back of the electric water boiler and drips from this collection could cause the dyes to run. The addition of Plastazote® pieces under the lid allowed excess steam to escape, and very little moisture gathered on the tensioned silk if the lid was removed and wiped every 10 minutes instead of Picard's method of every 15 minutes.<sup>158</sup>

Determining approximate depth of shade using one colour of dye was possible using immersion dye recipes and this method can reduce the effort required to determine colour if the conservator has previously immersion dyed the same fabric with Lanaset® dye.

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<sup>158</sup> Picard, 32.

The environmental conditions of humidity and temperature and any irregularities in the dyeing process were recorded since they may have influenced the performance of the thickened dyes or resists and were considered when concluding the research.

## CHAPTER 8: CONCLUSION AND FURTHER RESEARCH

### 8.1 Conclusion

The first research question investigated the most effective method of controlling localised application of Lanaset® dyes when applied to silk crepe line. This research found that both thickeners provided good control of wicking when applied locally and that dyes containing sodium alginate (SA) were easier to apply than those with sodium carboxymethyl cellulose (SCMC), as SA had a higher viscosity but was more likely to result in blotchy colour or to retain brushstrokes. SCMC provided smoother, more even colour but had a lower viscosity, making it more likely to run and wick under stencils and over barriers.

Steam fixing techniques using a water boiler were evaluated and optimised for future use. The most successful methods of preparing the substrate, frame, and pattern, and the selection of tools including brush selection were outlined. A successful method of selecting colour and depth of shade was determined for Lanaset® dyes used in localised application.

The second research question evaluated techniques of application of localised dyes in order to achieve optimal and reproducible results. This research found that application using Melinex® stencils and gutta resist provided the most efficient and easily reproducible results. When using a Melinex® stencil or gutta, a higher concentration of thickener may be less likely to wick under the Melinex® or over the gutta resist. A sharper and cleaner line resulted from careful application using stencils, and the gutta provided a softer edge to dyed areas and both methods would provide a helpful guide for textile conservators when locally applying dye by hand.

Cyclododecane (CDD) was difficult to control without extensive previous experience in applying the substance, and its application often resulted in distortion of the weave of silk crepe line. It is possible this could be reduced by use of a tighter weave substrate such as silk habotai. This barrier also required additional safety precautions. Research into its long-term effects to human health and the environment has been incomplete and inconclusive in the conservation field and should be evaluated before it is used in textile conservation as a

barrier method for localised application of dye. For these reasons, the other methods are preferred.

## **8.2 Further Research**

Chemical stability and effects of steam setting for each colour of dye could be further tested beyond this twelve week research. Picard's research evaluated the stability of Lanaset® dyes thickened with SCMC, but these dyes thickened with SA have not been recently evaluated.<sup>159</sup> Her experiments could be conducted again with remaining colours to determine if all Lanaset® colours are stable when steam-set, and if any of the colours have unusual sensitivity to light or moisture. The temperature when steam fixing could be lowered to protect the condition of the silk and possibly reduce the distortion of the weave. This would require additional testing to see if the reduction in temperature affects the washfastness, lightfastness, or wet and dry crocking.

A number of questions remain and merit further attention. It is difficult to address these questions in the long term or to know if the observations about dye stability are only valid over a short duration. In addition, changes could occur after completion of this research project to the areas of these samples where gutta and CDD have been used. In order to determine what could happen over time, it is suggested these samples be re-evaluated after 1 year, 5 years, and 10 years to determine if areas of resist yellow with age and if localized application of these dyes maintain their chemical stability over time. A sample of each barrier method has been retained with this dissertation.

Other areas for further research would be looking at alternate materials for barriers such as stencils that would have reduced rigidity and the use of a roller sponge when applying dye with stencils and resists, to prevent dyes from wicking under the stencil and to streamline application. Gutta resists from other companies could be compared and application to both sides of the substrate could be investigated. In addition, the method used of steam fixing over a water boiler only allows for pieces as large as the top opening. Alternate methods of

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<sup>159</sup> Jan Vuori and Season Tse, "Lightfastness of Irgalan and Lanaset dyed silk: Immersion Versus Direct Application: Results of a Preliminary Study," *Textile Conservation Newsletter* 33 (1997): 14-18.

fixing such as ironing, hand steaming, and the use of a steam box or a stovepipe steamer could be investigated when a larger piece of overlay or infill is needed.

Although some questions remain, this research has shown that both thickeners, sodium alginate and sodium carboxymethyl cellulose, are useful in the preparation of Lanaset® dyes for localised application. When using barriers for multi-coloured dye application, Melinex® stencils and gutta resist are the preferred of the three methods investigated. This dissertation offers clear guidelines for the selection and preparation of materials including substrate, brushes, and tensioning of the substrate on a frame. It details information on the mixing of dyes and thickeners, selecting depth of shade, and the fixing process to reduce ambiguities faced by textile conservators when applying dyes locally to support fabrics. With this report, textile conservators in a variety of contexts will be able to approach their work with greater confidence in their ability to achieve consistent and desirable outcomes.

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## **Appendix I Materials and Suppliers**

Sodium alginate

### **Special Ingredients**

Sodium carboxymethyl cellulose

### **Aldrich (US)**

Cyclododecane

### **Kremer Pigmente GmbH & Co**

Pēbēo water-based gutta  
Paint brushes

**Miller's – the City Art Shop Ltd**  
28 Stockwell Street  
Glasgow G1 4RT

Silk crepeline

### **Le Lievre (UK) Ltd.**

Lanaset® dyes  
Additives

**Huntsman UK**  
Adlington Court  
London Road  
Poyton SK10 4NL

Correx® Corrugated plastic sheeting  
FischerBrand pH strips  
Oddy testing supplies  
Decon 90  
Beakers  
Cotton wool  
tweezers  
Test tubes and stoppers  
Lead, silver, and copper coupons  
Acetone  
Glass brushes for each metal  
Melinex® polyester film  
Plastazote®

**Preservation Equipment Ltd**  
Vinces Road  
Diss  
Norfolk IP22 4HQ

Kitska electric wax pen

### **WaxArtSupply.com**

Nederman extraction hood  
Konica Minolta Spectrophotometer CM-2600d  
Heraeus Accelerated Ageing Oven  
Grant SBB14 electric water boiler

## **Appendix II Risk Assessment and COSHH forms**

R54/C1 Applying Cyclododecane to Fabric Substrates COSHH










R54/C2 Using pēbēo water-based colourless gutta for painting on silk COSHH

R54/C3 Immersion dyeing and local application of Lanaset® dyes on silk crepeline COSHH







R54/C4 Oddy Testing pēbēo gutta on silk crepeline COSHH

R54/C5 Azide Test of pēbēo gutta for sulfides COSHH

**Assessment Title: Applying Cyclododecane to fabric substrates**
**Assessment Reference Number: R54 /C1**
**School / Service / Location: Centre for Textile Conservation, Robertson Building, level 3, room 314**
**Safety Coordinator: Sarah Foscett**
**Details of Hazardous Substances (Please attach safety datasheets where available)**

Name of Substance (Include all substances used or produced)	Quantity kg / g / ml	Physical Form	GHS Hazard Classification (Tick all that apply)									
												
1. Cyclododecane (CDD)	< 200 g	Amorphous Solid	X							X		X

**Special Hazards (\*Separate risk assessment may be required)**

	Details: N/A		Details: N/A		Details: N/A
	Details: N/A		Details: N/A		Details: Dust can form explosive mixtures with air

**Further Details / Other Special Hazards:** Acute oral toxicity / when disposing, this chemical should be treated as toxic waste

**Exposure to Hazardous Substances**

Substance	Possible Exposure Route (Please tick)					Workplace Exposure Limits	
	Inhalation	Ingestion	Skin	Injection	Other (State)	8h TWA	15min STEL
1. Cyclododecane (CDD)	X		X			None specified	None specified

**Description of Activity (Continue on a separate sheet if required)**










- CDD will be heated to 60-90 degrees Celsius using heated metal tools and an electric double-boiler and applied to silk crepeline under extraction.
- CDD will be allowed to sublime at room temperature in a fume cupboard or open environment until it is no longer present.
- CDD will be tested using painted application of dye and stereomicroscopic analysis.
- After sublimating, silk that had been painted with CDD will be steamed in an electric water boiler under extraction.

**Persons at risk:**

Students, Staff, cleaners, visitors

**Summary of Control Measures**

Assessment of risks and any existing control measures	<ul style="list-style-type: none"> <li>- Exposure to CDD via various routes, including inhalation, and skin contact.</li> <li>- Handling and use of CDD only involves the use of small amounts</li> <li>- During flammable chemical use, no ignition sources will be present in the area.</li> <li>- Tie back hair</li> <li>- Ensure good hygiene, wash hands before handling food stuffs or doing other activities.</li> <li>- No food or drink to be consumed in the workroom/lab</li> <li>- PPE as outlined below, avoid contact with eyes, on skin or on clothing.</li> <li>- Ensure adequate ventilation.</li> <li>- Use good workroom practice.</li> </ul>		
Risk Rating (Before Control)	High	Medium x	Low
Procedural Controls (e.g. lone working, hygiene)	- Use of appropriate PPE when heating and applying CDD		
Engineering Controls	-Ensure adequate ventilation, avoid dust build up		

(e.g. fume cupboard)	<b>-Allow to sublime in fume cupboard</b>			
<b>PPE Requirements</b> (Please give details)  **Face fit testing required	 <b>Dust Mask**</b>	Dust Mask is not required for this amount of CDD but it will be worn during this activity	 <b>Gloves</b>	Rubber heat resistant gloves
	 <b>Respirator**</b>	NIOSH/MSHA or European Standard EN 149 approved respirator if exposure limits are exceeded or if irritation or other symptoms are experienced.	 <b>Footwear</b>	Closed toe shoes/boots
	 <b>Eye Protection</b>	Safety Goggles With protective shields	 <b>Protective Clothing</b>	Long sleeved lab coat
	 <b>Face Shield</b>		 <b>Other (Specify)</b>	Extractor will be used when Steaming this
<b>Instruction and Training</b>	Student has undertaken safety training for solvents and other chemicals but not for this chemical specifically.			
<b>Supervision Required?</b>	Yes, course tutor will be present			
<b>Other safety precautions:</b> (Including specialist first aid requirements)	Precautionary measures against static discharges will be taken: wearing non-synthetic clothes, cotton lab coat, and leather soled shoes.  <b>First aid:</b> -Inhalation: move to fresh air, if irritation persists consult physician -Skin contact: wash instantly with water and soap and rinse thoroughly -Contact with molten material: cool affected skin with water -Eye Contact: rinse open eye and under eyelids for at least 15 minutes under running water and consult physician -If swallowed: do not induce vomiting, give water to rinse out mouth, seek medical advice			
<b>New Risk Rating</b>	High	Medium x	Low	
<b>Supporting Information Checklist</b> (Include details for each where relevant)				
<b>Waste Disposal</b>	-Treat as toxic waste according to local laws and regulations. -Do not discharge into drains or surface/ground water -Dispose in suitable waste container			
<b>Emergency Procedures</b> (including spill / leak control)	-Any spills will be cleared away with blue roll and solvent will be allowed to evaporate in fume hood.			
<b>Atmospheric Monitoring</b>	-Avoid open heat, open fire and other ignition sources -Avoid strong oxidising agents			
<b>Health Surveillance</b>	N/A			
<b>Supporting Risk Assessments</b> (Please attach where relevant)	Biological N/A	DSEAR N/A	Radiation N/A	
<b>Assessment Details</b>				
<b>Assessed By: Kathleen Martin</b>			Date: 31/5/2018	
 <b>Approved By: Sarah Foscett</b>			Date: 31/5/2018	
Date of next review: 23/5/2019				
<b>Description of Activity</b> (Continuation sheet)				

Continuation sheet number:

### CoSHH Assessment Acknowledgement

By signing this document I acknowledge that I have read and understood the attached CoSHH assessment and have familiarised myself with the safety control measures and protective equipment necessary to carry out the task safely. I hereby agree to follow the safe system of work required and implement the required safety procedures fully.

Full Name	Signature	Date Completed
Kathleen Martin		31/5/2018



# CoSHH Assessment

Assessment Title: **Using Pēbēo Gutta for painting on silk**

Assessment Reference Number: **R54 /C2**

School / Service / Location: **Centre for Textile Conservation, Robertson Building, level 3, room 314**

Safety Coordinator: **Sarah Foscett**

**Details of Hazardous Substances (Please attach safety datasheets where available)**

Name of Substance (Include all substances used or produced)	Quantity kg / g / ml	Physical Form	GHS Hazard Classification (Tick all that apply)													
Pēbēo water based gutta – Colourless 001	20ml	Liquid	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

**Special Hazards (\*Separate risk assessment may be required)**

	Details:		Details: eye irritant		Details:
	Details:		Details:		Details:

Further Details / Other Special Hazards: **N/A**

Exposure to Hazardous Substances						Workplace Exposure Limits	
Substance	Possible Exposure Route (Please tick)					8h TWA	15min STEL
	Inhalation	Ingestion	Skin	Injection	Other (State)		
Pēbēo water based gutta – Colourless 001	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	N/A	N/A

**Description of Activity (Continue on a separate sheet if required)**










- Applying this product to silk crepe line, painting dyes on to this fabric, steaming the fabric then washing out this product with water.

Persons at risk: **Students and Staff**

**Summary of Control Measures**

Assessment of risks and any existing control measures	<ul style="list-style-type: none"> <li>-Contact with skin – lab coat, nitrile gloves, eyewear with side shields and closed toe shoes as outlined below.</li> <li>-Avoid ingestion and inhalation. Ensure adequate ventilation.</li> <li>-Acids are flammable - avoid heat, flame sparks and electrical equipment.</li> <li>-Use good workroom practice.</li> <li>- No eating or drinking in areas where mixture is used.</li> </ul>		
Risk Rating (Before Control)	High	Medium	Low x
Procedural Controls (e.g. lone working, hygiene)	<ul style="list-style-type: none"> <li>- Cleaning away any spills immediately</li> <li>- Use of appropriate PPE</li> <li>- Handle in well-ventilated area.</li> </ul>		
Engineering Controls (e.g. fume cupboard)			



<b>PPE Requirements</b> (Please give details)  **Face fit testing required	 <b>Dust Mask**</b>		 <b>Gloves</b>	Latex, nitrile, PVC or butyl rubber gloves
	 <b>Respirator**</b>		 <b>Footwear</b>	Closed toe shoes/boots
	 <b>Eye Protection</b>	Safety goggles	 <b>Protective Clothing</b>	Lab coat
	 <b>Face Shield</b>		 <b>Other (Specify)</b>	
<b>Instruction and Training</b>	Trained in lab procedures, not trained specifically for this product			
<b>Supervision Required?</b>	Yes, tutor must be present in the building			
<b>Other safety precautions:</b> (Including specialist first aid)	-In the event of splashes or with eyes, wash thoroughly with water for 15 minutes holding the eyelids open. -In the event of swallowing, rinse mouth with water, do not induce vomiting and seek medical attention. -Store in an upright position			
<b>New Risk Rating</b>	High	Medium	Low x	
<b>Supporting Information Checklist</b> (Include details for each where relevant)				
<b>Waste Disposal</b>	Do not dispose of in drains or waterways			
<b>Emergency Procedures</b> (including spill / leak control)	Any spills will be cleared away with blue roll			
<b>Atmospheric Monitoring</b>	Avoid high and freezing temperatures			
<b>Health Surveillance</b>	N/A			
<b>Supporting Risk Assessments</b> (Please attach where relevant)	Biological N/A	DSEAR N/A	Radiation N/A	
<b>Assessment Details</b>				
<b>Assessed By:</b> Kathleen Martin			<b>Date:</b> 31/5/18	
<b>Approved By:</b> Sarah Foskett 			<b>Date:</b> 31/5/18	
<b>Date of next review:</b> May 2020				
<b>Description of Activity</b> (Continuation sheet)				
<b>Continuation sheet number:</b>				

### CoSHH Assessment Acknowledgement

By signing this document I acknowledge that I have read and understood the attached CoSHH assessment and have familiarised myself with the safety control measures and protective equipment necessary to carry out the task safely. I hereby agree to follow the safe system of work required and implement the required safety procedures fully.

Full Name	Signature	Date Completed
Kathleen Martin		31/5/2018











**Assessment Title: Immersion dyeing and local application of Lanaset® dyes on silk crepe line fabric**

**Assessment Reference Number: R54 /C3**







**School / Service / Location: Centre for Textile Conservation, Robertson Building, level 3, room 314**

**Safety Coordinator: Sarah Foskett**

**Details of Hazardous Substances (Please attach safety datasheets where available)**









Name of Substance (Include all substances used or produced)	Quantity kg / g / ml	Physical Form	GHS Hazard Classification (Tick all that apply)															
																		
1. Lanaset dye Black B GR	<20g	powder																
2. Lanaset dye Blue 2R	<20g	powder						X										X
3. Lanaset dye Yellow 2R GR	<20g	powder						X										
4. Lanaset dye Red 2B	<20g	powder																X
5. Albaflow	<50ml	liquid						X										
6. Sodium acetate	<50ml	liquid																
7. Acetic Acid	<50ml	liquid		X						X								
8. Albege Set	<50ml	liquid						X										
9. Sodium sulphate	<50ml	liquid																
10. Formic Acid	<50ml	liquid		X						X		X						
11. Sodium Alginate	<20g	powder																
12. Sodium carboxymethyl cellulose (SCMC)	<20g	powder																


**Special Hazards (\*Separate risk assessment may be required)**

	Details:		Details:		Details:
<b>Carcinogenic Substance</b>		<b>Skin Sensitiser</b>		<b>Respiratory Sensitiser</b>	
	Details:		Details:		Details:
<b>Biological Material*</b>		<b>Radioactive Substances*</b>		<b>Explosive Atmosphere*</b>	

**Further Details / Other Special Hazards: N/A**

Substance	Possible Exposure Route (Please tick)					Workplace Exposure Limits	
	Inhalation	Ingestion	Skin	Injection	Other (State)	8h TWA	15min STEL
1. Lanaset dye Black B GR	X		X			N/A	N/A
2. Lanaset dye Blue 2R	X		X			N/A	N/A
3. Lanaset dye Yellow 2R GR	X		X			N/A	N/A
4. Lanaset dye Red 2B	X		X			N/A	N/A
5. Albaflow			X			N/A	N/A
6. Sodium acetate			X			N/A	N/A
7. Acetic acid	X		X			10ppm	N/A
8. Albege Set			X			N/A	N/A
9. Sodium sulphate			X			N/A	N/A
10. Formic acid			X			5ppm	15ppm
11. Sodium alginate	X		X			N/A	N/A

12. Sodium carboxymethyl cellulose (SCMC)	X		X			10mg/m <sup>3</sup>	N/A
<b>Description of Activity</b> (Continue on a separate sheet if required)							
<ul style="list-style-type: none"> <li>- Measuring and mixing dye powder into solution in 250 ml medical flat bottles</li> <li>- Measuring dye additives into small beakers</li> <li>- Dyeing of support fabric using dye solution and additives</li> <li>- Painting dyes on support fabric and steam setting</li> </ul>							
<b>Persons at risk:</b> Students and Staff							
<b>Summary of Control Measures</b>							
<b>Assessment of risks and any existing control measures</b>	<p>-Possible inhalation of dye powders and acetic acid vapours – dust mask to be worn and adequate ventilation in workspace.</p> <p>-Contact with skin – lab coat, nitrile gloves, eyewear with side shields and closed toe shoes as outlined below.</p> <p>-Avoid ingestion and inhalation. Ensure adequate ventilation.</p> <p>-Acids are flammable - avoid heat, flame sparks and electrical equipment.</p> <p>-Use good workroom practice.</p>						
<b>Risk Rating (Before Control)</b>	High		Medium		Low x		
<b>Procedural Controls</b> (e.g. lone working, hygiene)	<ul style="list-style-type: none"> <li>- Cleaning away any spills or broken glassware immediately</li> <li>- Use of appropriate PPE when measuring dye and mixing dye stock</li> </ul>						
<b>Engineering Controls</b> (e.g. fume cupboard)							
<b>PPE Requirements</b> (Please give details)	 Dust Mask**	Dust Mask		 Gloves	Nitrile Gloves		
**Face fit testing required	 Respirator**			 Footwear	Closed toe shoes/boots		
	 Eye Protection	Safety Glasses with side shields		 Protective Clothing	Lab coat		
	 Face Shield			 Other (Specify)			
<b>Instruction and Training</b>	Yes, dye course undertaken November 2016						
<b>Supervision Required?</b>	Yes, tutor must be present in the building						
<b>Other safety precautions:</b> (Including specialist first aid)	Rinse eyes or skin immediately if contact.						
<b>New Risk Rating</b>	High		Medium		Low x		
<b>Supporting Information Checklist</b> (Include details for each where relevant)							
<b>Waste Disposal</b>	Dyes and additives will be diluted and disposed in sink						
<b>Emergency Procedures</b> (including spill / leak control)	<ul style="list-style-type: none"> <li>- Any spills will be cleared away with blue roll</li> <li>- If a large spill, use spill kit in chem lab (310).</li> <li>- Broken glassware will be disposed of in the broken glassware container in chem lab.</li> <li>- In the event of a leaking wash bath, spillage will be cleared immediately and the bath will be switched off.</li> </ul>						
<b>Atmospheric Monitoring</b>	N/A						
<b>Health Surveillance</b>	N/A						
<b>Supporting Risk Assessments</b> (Please attach where relevant)	Biological N/A		DSEAR N/A		Radiation N/A		
<b>Assessment Details</b>							

Assessed By: Kathleen Martin	Date: 23/5/18
 Approved By: Sarah Foskett	Date: 31/5/18
Date of next review: May 2020	
<b>Description of Activity (Continuation sheet)</b>	
Continuation sheet number:	

### CoSHH Assessment Acknowledgement

By signing this document I acknowledge that I have read and understood the attached CoSHH assessment and have familiarised myself with the safety control measures and protective equipment necessary to carry out the task safely. I hereby agree to follow the safe system of work required and implement the required safety procedures fully.

Full Name	Signature	Date Completed
Kathleen Martin		23/5/2018










**Assessment Title: Oddy Testing Pēbēo Gutta on silk crepeline**

**Assessment Reference Number: R54 /C4**







**School / Service / Location: Centre for Textile Conservation, Robertson Building, level 3, room 314**

**Safety Coordinator: Sarah Foksett**

**Details of Hazardous Substances (Please attach safety datasheets where available)**

Name of Substance (Include all substances used or produced)	Quantity kg / g / ml	Physical Form	GHS Hazard Classification (Tick all that apply)										
													
Pēbēo water based gutta – Colourless 001	5ml	Liquid											
Decon 90 detergent	20ml	Liquid					X						
Acetone	20ml	Liquid		X			X						
Lead	4g	Metal					X				X	X	
Copper	4g	Metal											
Silver	4g	Metal											

**Special Hazards (\*Separate risk assessment may be required)**









 Carcinogenic Substance	<b>Details: Lead is harmful if swallowed. May cause cancer. Suspected of damaging fertility or the unborn child. May cause damage to organs through prolonged or repeated exposure.</b>	 Skin Sensitiser	<b>Details:</b>	 Respiratory Sensitiser	<b>Details:</b>
 Biological Material*	<b>Details: Lead is very toxic to aquatic life with long lasting effects.</b>	 Radioactive Substances*	<b>Details:</b>	 Explosive Atmosphere*	<b>Details:</b>


**Further Details / Other Special Hazards: Operations generating lead dust, fume or vapour can result in sufficient lead entering your body to be hazardous to your health.**

Exposure to Hazardous Substances						Workplace Exposure Limits	
Substance	Possible Exposure Route (Please tick)					8h TWA	15min STEL
	Inhalation	Ingestion	Skin	Injection	Other (State)		
Pēbēo water based gutta – Colourless 001			X			n/a	n/a
Decon 90	X	X	X	X		n/a	2 mg/m <sup>3</sup>
Acetone	X	X	X	X		500 ppm	1500 ppm
Lead			X	X		0.15 mg/m <sup>3</sup>	0.45 mg/m <sup>3</sup>
Copper			X	X		1 mg/m <sup>3</sup>	2 mg/m <sup>3</sup>
Silver			X	X		0.1 mg/m <sup>3</sup>	0.3 mg/m <sup>3</sup>

**Description of Activity (Continue on a separate sheet if required)**

- Applying the gutta to silk crepeline, steaming the fabric then washing out this product with water and allowing to dry.
- Cleaning test tubes with Decon 90 solution in water.
- Preparation of lead, copper and silver coupons using a glass brush and acetone, approx.. 20 ml.

- Handling of coupons for assessment following 28 days of testing at 60° C.				
<b>Persons at risk:</b> Students, staff, cleaners and visitors				
<b>Summary of Control Measures</b>				
<b>Assessment of risks and any existing control measures</b>	<ul style="list-style-type: none"> <li>-Contact with skin – lab coat, nitrile gloves, eyewear with side shields and closed toe shoes as outlined below.</li> <li>-Avoid ingestion and inhalation. Ensure adequate ventilation.</li> <li>-Acids are flammable - avoid heat, flame sparks and electrical equipment.</li> <li>-Use good workroom practice.</li> <li>-No eating or drinking in areas where mixture is used.</li> </ul>			
<b>Risk Rating (Before Control)</b>	High	Medium	Low x	
<b>Procedural Controls</b> (e.g. lone working, hygiene)	<ul style="list-style-type: none"> <li>- Cleaning away any spills immediately</li> <li>- Use of appropriate PPE</li> <li>- Handle in well-ventilated area.</li> </ul>			
<b>Engineering Controls</b> (e.g. fume cupboard)	Provide good ventilation of working area (local exhaust ventilation, if necessary).			
<b>PPE Requirements</b> (Please give details)  **Face fit testing required	 Dust Mask**		 Gloves	Nitrile gloves
	 Respirator**	Respirator recommended if work activity is likely to result in formation of lead fumes, vapours or dust.	 Footwear	Closed toe shoes/boots
	 Eye Protection	Safety goggles	 Protective Clothing	Lab coat
	 Face Shield	Face protection	 Other (Specify)	
<b>Instruction and Training</b>	Trained in lab procedures			
<b>Supervision Required?</b>	Yes, tutor must be present in the building			
<b>Other safety precautions:</b> (Including specialist first aid)	<p><b>Skin Contact:</b> In case of contact, immediately flush skin with plenty of water. Cover the irritated skin with an emollient. Remove contaminated clothing and shoes. Cold water may be used. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention.</p> <p><b>Serious Skin Contact:</b> Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek medical attention.</p> <p><b>Inhalation:</b> If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention if symptoms appear.</p> <p><b>Ingestion:</b> Do NOT induce vomiting unless directed to do so by medical personnel. Loosen tight clothing such as a collar, tie, belt or waistband. Seek medical attention.</p>			
<b>New Risk Rating</b>	High	Medium	Low x	
<b>Supporting Information Checklist</b> (Include details for each where relevant)				
<b>Waste Disposal</b>	<p><b>Acetone:</b> Do not dispose of in drains or waterways</p> <p><b>Lead:</b> Should be recycled or disposed as hazardous waste. Do not allow product to reach sewage system. Different Pb-bearing wastes resulting from the processes described above are generated in the form of dross, flue dust and slag. These waste products are mainly recycled in the production process or landfilled.</p>			
<b>Emergency Procedures</b> (including spill / leak control)	Any spills will be cleared away with blue roll			

<b>Atmospheric Monitoring</b>	<b>Avoid high and freezing temperatures</b>		
<b>Health Surveillance</b>	<b>Avoid inhalation of lead dust and monitor fragments of glass brush when cleaning coupons</b>		
<b>Supporting Risk Assessments</b> (Please attach where relevant)	<b>Biological</b> N/A	<b>DSEAR</b> N/A	<b>Radiation</b> N/A
<b>Assessment Details</b>			
<b>Assessed By: Kathleen Martin</b>		<b>Date: 18/6/18</b>	
 <b>Approved By: Sarah Foskett</b>		<b>Date: 18/6/18</b>	
<b>Date of next review: May 2020</b>			
<b>Description of Activity (Continuation sheet)</b>			
<b>Continuation sheet number:</b>			

### CoSHH Assessment Acknowledgement

By signing this document I acknowledge that I have read and understood the attached CoSHH assessment and have familiarised myself with the safety control measures and protective equipment necessary to carry out the task safely. I hereby agree to follow the safe system of work required and implement the required safety procedures fully.

<b>Full Name</b>	<b>Signature</b>	<b>Date Completed</b>
Kathleen Martin		10/6/2018



Assessment Title: **Azide Test of Pēbēo Gutta for sulfides**

Assessment Reference Number: **R54 /C5**

School / Service / Location: **Centre for Textile Conservation, Robertson Building, level 3, room 314**

Safety Coordinator: **Sarah Foskett**

**Details of Hazardous Substances (Please attach safety datasheets where available)**

Name of Substance (Include all substances used or produced)	Quantity kg / g / ml	Physical Form	GHS Hazard Classification (Tick all that apply)											
Pēbēo water based gutta – Colourless 001	<5ml	Liquid												
Decon 90 detergent	5% sol	Liquid						X						
Iodine	<1.27 g	Solid						X	X			X	X	X
Sodium azide	<3g	Crystalline									X	X	X	
Potassium iodide	<2g	solid						X						

**Special Hazards (\*Separate risk assessment may be required)**

	Details:		Details:		Details:
	Details:		Details:		Details:

**Further Details / Other Special Hazards:**

Cause eye and skin irritation (iodine, potassium azide), respiratory irritation (iodine)

Damage to organs (iodine and sodium azide) or brain (sodium azide) through prolonged/repeated exposure

Undiluted Decon 90 is corrosive

Fatal if swallowed or in contact with skin (sodium azide)

Harmful if swallowed or inhaled (iodine, potassium iodide)

**Exposure to Hazardous Substances**









**Workplace Exposure Limits**


Substance	Possible Exposure Route (Please tick)					8h TWA	15min STEL
	Inhalation	Ingestion	Skin	Injection	Other (State)		
Pēbēo water based gutta – Colourless 001			X			N/A	N/A
Decon 90	X	X	X			N/A	2 mg/m <sup>3</sup>
Iodine	X	X	X			0.01 ppm	0.01 ppm
Sodium azide	X	X	X			N/A	N/A
Potassium iodide	X	X	X			N/A	N/A

**Description of Activity (Continue on a separate sheet if required)**

- Solution of sodium azide and iodine did not need to be made up since it was prepared for a previous test at CTC.
- This solution will be used to test for sulphides in gutta resist using these steps:
  1. Clean microscope slide with Decon 90, rinse in distilled water and then acetone. Dry and polish with a lint free tissue. Place slide on microscope stage.
  2. Place a drop of gutta resist on microscope slide. Place cover slip on top.
  3. While examining sample down the microscope, place a drop of the sodium azide solution at the edge of coverslip using a disposable pipette. Capillary action draws the solution under it and spreads the solution around the sample.
  4. Observe for up to one minute, recording any gas bubbles formed and at what rate.



5. Wash microscope slides and coverslips in Decon 90 and rinse in water. Rinse pipettes in water and dispose of in glass waste box.				
<b>Persons at risk:</b> Students, staff, cleaners and visitors				
<b>Summary of Control Measures</b>				
<b>Assessment of risks and any existing control measures</b>	-Exposure to chemicals via various routes including inhalation, skin contact and ingestion -Handling and use of chemicals involves only the use of small amounts. -Flammable solvents – no ignition sources will be present in the area			
<b>Risk Rating (Before Control)</b>	High	Medium x	Low	
<b>Procedural Controls</b> (e.g. lone working, hygiene)	- No food or drink to be consumed in the workrooms or labs. - Ensure good hygiene – wash hands before handling foodstuffs or doing other activities. - Tie back long hair - lab coat, eyewear with side shields and closed toe shoes as outlined below. - Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. - Iodine: use only under chemical fume cupboard. -Chemicals stored in cupboards. -Recording of sodium azide amount used in Poisons Log. -Maximum volume of iodine solution/azide solution made is 25 ml. -Cleaning away any spills immediately.			
<b>Engineering Controls</b> (e.g. fume cupboard)	Fume cupboard required when making up this solution			
<b>PPE Requirements</b> (Please give details)  **Face fit testing required	 Dust Mask**	Face mask	 Gloves	Nitrile gloves
	 Respirator**	Fume Hood	 Footwear	Closed toe shoes/boots
	 Eye Protection	Safety goggles	 Protective Clothing	Lab coat
	 Face Shield		 Other (Specify)	
<b>Instruction and Training</b>	Yes			
<b>Supervision Required?</b>	Yes, tutor must be present in the building			
<b>Other safety precautions:</b> (Including specialist first aid)	<b>First Aid-</b> <b>Inhalation-</b> supply fresh air <b>Skin contact-</b> wash instantly with soap and water and rinse thoroughly. <b>Eye contact -</b> rinse opened eye for several minutes under running water. <b>If Ingested -</b> Do not induce vomiting, give water to rinse out mouth if unconscious. Seek medical attention.			
<b>New Risk Rating</b>	High	Medium	Low x	
<b>Supporting Information Checklist</b> (Include details for each where relevant)				
<b>Waste Disposal</b>	Given the small quantities, wash waste sodium azide down fume cupboard sink with copious amounts of running water.			
<b>Emergency Procedures</b> (including spill / leak control)	All quantities are small, absorb spills with blue roll, bag in plastic bag and put in general waste.			
<b>Atmospheric Monitoring</b>				
<b>Health Surveillance</b>				
<b>Supporting Risk Assessments</b>	Biological	DSEAR	Radiation	

(Please attach where relevant)	N/A	N/A	N/A
<b>Assessment Details</b>			
Assessed By: Kathleen Martin		Date: 19/7/2018	
 Approved By: Sarah Foskett		Date: 19/7/2018	
Date of next review: May 2020			
<b>Description of Activity (Continuation sheet)</b>			
Continuation sheet number:			

### CoSHH Assessment Acknowledgement

By signing this document I acknowledge that I have read and understood the attached CoSHH assessment and have familiarised myself with the safety control measures and protective equipment necessary to carry out the task safely. I hereby agree to follow the safe system of work required and implement the required safety procedures fully.

Full Name	Signature	Date Completed
Kathleen Martin		19/7/2018

<b>Management Unit:</b>	Centre for Textile Conservation and Technical Art History	<b>Location: (Site/ Building/ Room)</b>	CTC 2 <sup>nd</sup> year workroom, Dye Lab and Chemistry Lab, Level 3, Robertson Building
<b>Assessment Date:</b>	23/05/2018	<b>Review Date:</b>	23/05/2019
<b>Assessors Name:</b>	Kathleen Martin	<b>Job Title:</b>	Student
<b>Task / Activity:</b>			
<p>1. Dyeing silk crepe line by immersion dyeing and painted application</p> <ul style="list-style-type: none"> <li>- Creating stencils from Melinex® polyester film and polythene</li> <li>- Cutting Correx® board into frames</li> <li>- Immersion dyeing silk crepe line fabric with Lanaset® dyes.</li> <li>- Using sodium carboxymethyl cellulose and sodium alginate to thicken the dyes</li> <li>- Painted application of dye on silk crepe line fabric</li> <li>- fixing by steaming over bain marie.</li> </ul> <p>2. Applying resists of cyclododecane (CDD) and gutta resists to silk before dyeing</p> <ul style="list-style-type: none"> <li>- Painting with gutta resist using the applicator tip on silk fabric.</li> <li>- Heating CDD to 60-90 degrees Celsius using heated metal tools and an electric double-boiler.</li> <li>- Applying CDD to silk fabric.</li> <li>- Allowing CDD to sublimate at room temperature in an open environment until it is no longer present.</li> <li>- Testing efficacy of CDD and gutta by painted application of dye and stereomicroscopic analysis.</li> </ul> <p>3. Testing materials and results</p> <ul style="list-style-type: none"> <li>- Taking colorimeter readings and photographs of dyed and painted samples.</li> <li>- Oddy testing of steamed fixed and unfixed gutta resist, which will require: cleaning test tubes with Decon 90 in water cleaning lead, copper and silver coupons using a glass brush and acetone.</li> <li>- Azide testing gutta resist, which will require: Using pre-made solution of sodium azide and iodine to test for sulphides in gutta resist. By placing a drop of the sodium azide solution at the edge of coverslip on microscope slide with a drop of gutta resist and observe for the formation of gas bubbles.</li> <li>- Use of the Colorimeter</li> <li>- Use of FTIR</li> </ul>			

What are the hazards? (See list of sample hazards)	What are the risks?	Who might be harmed? (eg Staff, students, visitors)	What control measures are required to eliminate or reduce the risks?	Risk Evaluation			Risk Rating
				Consequence (1 – 3)	Likelihood (1 – 3)	Overall risk (C x L)	Low, Medium or High
Sharp hand tools	Cuts from use of sharp hand tools to cut Melinex®, polythene, Correx®, silk crepeline	Students	<ul style="list-style-type: none"> <li>- Observance of appropriate work room behaviour</li> <li>- Use cutting board where appropriate</li> <li>- First aid kit in Room 315</li> <li>- Dispose dull blades into sharps box in chemistry lab under sink.</li> </ul>	2	1	2	Low
Use of electrical equipment (camera, dye bath, double boiler for heating CDD)	Electrical hazards Trip hazards	Staff, students and visitors	<ul style="list-style-type: none"> <li>- Unplug and switch off when not in use.</li> <li>- Ensure equipment is in good working order and has recently passed PAT testing.</li> <li>- Check all wires and plugs before use and avoid trailing wires.</li> </ul>	1	1	1	Low
Extreme heat Liquid and steam setting	Burns	Student	<ul style="list-style-type: none"> <li>- Wear heat resistant gloves during steaming process</li> <li>- Full PPE</li> <li>- Wait until water is cool to dispose of.</li> <li>- Well-ventilated room.</li> </ul>	1	2	2	Low
Handling and using CDD	<ul style="list-style-type: none"> <li>- Damage to health from inhalation, burns</li> <li>- Dust from CDD can form explosive mixtures with air</li> </ul>	Staff, student and visitors	<ul style="list-style-type: none"> <li>- See COSHH: R54/C1</li> <li>- Observance of appropriate PPE (rubber gloves, lab coat, closed toe shoes, respirator or dust mask). Long hair and jewellery secured.</li> <li>- Use of appropriate containers.</li> </ul>	3	1	3	Medium

			<ul style="list-style-type: none"> <li>- Provide adequate ventilation</li> <li>- Keep containers tightly closed in a dry, cool well-ventilated place.</li> <li>- If additional ventilation is required, local extraction will be applied.</li> <li>- Risk of static build up will be reduced by wearing non-synthetic clothes, cotton lab coat and leather soled shoes</li> </ul>				
Handling and using gutta resist	<ul style="list-style-type: none"> <li>- Spills</li> </ul>	Students	<ul style="list-style-type: none"> <li>- See COSHH: R54/C2</li> <li>- Observance of appropriate PPE (gloves, dust mask, lab coat and eye protection).</li> <li>- Use of appropriate and labelled glassware and containers.</li> <li>- Chemical spills to be cleaned using blue roll and appropriately disposed.</li> </ul>	1	1	1	Low
Handling and using dyes, thickeners, and dye additives	<ul style="list-style-type: none"> <li>- Damage to health</li> <li>- Spills</li> </ul>	Students	<ul style="list-style-type: none"> <li>- See COSHH: R54/C3</li> <li>- Observance of appropriate PPE (gloves, dust mask, lab coat and eye protection). Long hair secured.</li> <li>- Use of appropriate and labelled glassware and containers.</li> <li>- Chemical spills to be cleaned using blue roll and appropriately disposed.</li> </ul>	2	1	1	Low
Oddy Testing	<ul style="list-style-type: none"> <li>- Damage to health from lead coupons and glass brush</li> <li>- Spills</li> </ul>	Staff, students and visitors	<ul style="list-style-type: none"> <li>- See COSHH: R54/C4</li> <li>- Observance of appropriate PPE (gloves, dust mask, lab</li> </ul>	2	1	1	Low

			<ul style="list-style-type: none"> <li>coat and eye protection). Long hair secured.</li> <li>- Use of proper ventilation and exhaust system</li> <li>- Use of appropriate and labelled glassware and containers.</li> <li>- Chemical spills to be cleaned using blue roll and appropriately disposed.</li> </ul>				
Azide Testing	<ul style="list-style-type: none"> <li>- Damage to health from exposure to chemicals</li> <li>- Spills</li> </ul>	Staff, students and visitors	<ul style="list-style-type: none"> <li>- See COSHH: R54/C5</li> <li>- Observance of appropriate PPE (gloves, dust mask, lab coat and eye protection). Long hair secured.</li> <li>- Proper glove removal technique (without touching glove's outer surface)</li> <li>- Use of proper ventilation and exhaust system</li> <li>- Use of appropriate and labelled glassware and containers.</li> <li>- Dispose of contaminated gloves after use in accordance with laws and good laboratory practice.</li> <li>- Chemical spills to be cleaned using blue roll and appropriately disposed.</li> <li>- Recording of sodium azide amounts used in a Poisons log.</li> <li>-</li> </ul>				
Slips, trips and falls	<ul style="list-style-type: none"> <li>- Harm to health (physical and chemical)</li> </ul>	Staff, student and visitors	<ul style="list-style-type: none"> <li>- Observe good work room practice. Cover trip hazards.</li> <li>- Immediately clean spillages.</li> </ul>	2	1	2	Low

	<ul style="list-style-type: none"> <li>- Spillage of chemicals</li> <li>- Breakage of glassware</li> </ul>		<ul style="list-style-type: none"> <li>- Ensure work space and routes are clear.</li> </ul>				
Glassware breakage	Cuts which may lead to chemical exposure	Staff, students and visitors.	<ul style="list-style-type: none"> <li>- Handle glassware with care. Damaged glassware should be reported.</li> <li>- Broken glass should be communicated to surrounding people and cleared away.</li> <li>- Glassware should be disposed of in the glass disposal box in the chemistry lab (Room 310) and wet room (Room 315)</li> <li>-</li> </ul>	1	2	2	Low
Working in close proximity with others	Use of incompatible chemicals, poor workroom practices	Staff and students	<ul style="list-style-type: none"> <li>- Making sure that other students are aware of what chemicals are being used.</li> <li>- Avoid using CDD near strong oxidising agents</li> <li>- Don't operate electrical equipment near those working with chemicals.</li> <li>- Allow for enough space to safely complete tasks</li> </ul>	3	1	3	Medium

**Completed by (print name and position and sign):**

Kathleen Martin (student) 

**Date:** 2 June 2018

**Approved by (print name and position and sign):**

Sarah Foscett 

**Date:** 2 June 2018

### Appendix III Preliminary Experiments

#### Trial 1 - Washing with surfactant after steam fixing

- To test for dye bleed and colour loss, two samples of silk crepe line painted with Lanaset® dyes were steam fixed then cut in half.
- Each half contained both SCMC thickened and SA thickened dyes. One half was washed in (2) 5 minute 1 liter baths of Dehypon LS54 with a cmc of 0.6g/L x3 cmc.<sup>160</sup>
- After washing, the halves were compared using a spectrophotometer.
- For the locally applied sample with gutta and the sample with Melinex® stencils, there was only a negligible difference between colour before and after wet cleaning as seen in table 4. The  $\Delta E$  value is very low because the change in lightness, red-green colour axis, and blue-yellow colour axis were very slight.

Product	Time	L*	a*	b*	$\Delta E$
Gutta	Before	85.36	17.1	4.52	0.73
	After	84.918	17.65	4.71	
Melinex®	Before	85.83	15.93	4.42	0.28
	After	86.066	15.88	4.57	

Table 4: Comparison of locally applied dye samples, washed in surfactant vs. not washed in surfactant

- Conclusion: There was no visual dye bleed in either washed sample and very little change in the light/darkness or colour of the wet cleaned samples as documented by spectrophotometer readings. Therefore, there was no reason to warrant washing each of the samples in the main experiment after steam fixing. As each colour of Lanaset® has slightly different fastness properties, testing each colour separately would be essential future research.

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<sup>160</sup> Picard, 33.



### Trial 2 - Methods of tensioning

- No tension when painted / only tensioned when steamed resulted in blurry hexagons.
- Tensioned by stapling silk to frame resulted in uneven tension where there were no staples and stretching where there were staples. Many staples used were a waste of resources.
- Tensioned by method described in silk painting book using tape resulted in even tensioning and the tape stayed in place while steaming. Less silk crepeline was required for each sample.

### Trial 3 – Dyes: 1% dye stock vs 0.1% dye stock

- Observations: the higher depth of shade resulted in colour bleed especially in red SCMC with gutta and blue SCMC gutta (on 21 June trials). Staining due to colour bleed on white areas of silk occurred during steaming and was set in the steam fixing process.
- Conclusion: Dye bleed was more difficult to control with 1% dye stock than 0.1% dye stock.

### Trial 4 - Gutta vs CDD on one piece of silk crepeline

- Observations: The areas painted with gutta had a softer edge and a dull blueish tint where the gutta did not completely protect the silk from black dye and the areas with CDD had crisper areas and very little dye, almost no dye in some areas where the CDD protected both sides of the silk since it settled into the weave of the silk crepeline and covered the back of the silk as it rested on the Melinex®.
- Making a very thin line with the gutta was difficult even if a straight pin was dipped in the gutta and used to apply it as it was hard to control where the gutta would stick to the silk crepeline.
- Both gutta and CDD had a splotchy 'batik' appearance in some places where the resist had been and dye had been painted over the resist.
- CDD required 4 days to sublime on handkerchief design painted onto silk crepeline and 4 days to sublime on dye stock tests.

#### Trial 5 – polythene vs Melinex® vs silicone release paper as bed for silk crepeline

- Polythene was used as a bed for silk crepeline in the same method used when blocking for adhesive application, however it shifted and creases in the polythene transferred to the silk crepeline. This made painting the design difficult but may have worked without a paper template of the pattern.
- Process used:  
The table surface was dampened with a dahlia sprayer and a layer of polythene was placed on this surface. Using a window squeegee excess water was removed, creating a vacuum to adhere the polythene to the table. This was not done since I was using a paper copy of the hexagon design and it created too many layers with risk of shifting while painting.
- Melinex® was problematic because the dyes stuck to this surface when drying and in some areas painted dye adhered to the Melinex® when the silk was peeled away disrupting the pattern and leaving holes.
- Silicone release paper method used by Schmalz and Picard worked perfectly.<sup>161162</sup>  
The silk peeled cleanly from the surface when dry and left no damage to the painted dyes.

#### Appendix IV: Immersion Dyeing: The steps for dyeing the control samples of silk<sup>163</sup>

1. Dry non-scoured silk was weighed to assess the amount of dye needed.
2. The dye bath was heated to 50°C
3. The silk was added to the dye bath to wet out.
4. The dyeing auxiliary was added.
5. Additives recommended by manufacturer was added.
6. The dye was added.
7. The bath temperature was raised slowly from 50°C - 84°C.
8. The temperature was held at 75°C for 20 minutes.

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<sup>161</sup> Susan Schmalz, "When Patching is Impractical: Nontraditional Compensation for Loss in a Quilt," *Journal of the American Institute for Conservation*, vol. 38, no. 3 (Autumn -Winter, 1999): 390.







<sup>162</sup> Picard, 31.

<sup>163</sup> Sarah Foskett and Katharina Mackert, "Dye Techniques Manual," (Glasgow: Centre for Textile Conservation, 2016), 41.

9. The temperature was raised and held at 84°C for 30 minutes to fix the dye and trying not to exceed this temperature was not exceeded since the dyestuff is silk.<sup>164</sup>
10. The fabric was cooled in the dye bath and rinsed thoroughly.<sup>165</sup>

During the dyeing process, the bath was stirred throughout, and the fabric unfolded to make sure the dye penetrated all the silk. The fabric was removed when additives and dye were added to make sure they were not applied directly to the fabric.

### Appendix V: Immersion Dyeing Sheet

DYE SHEET		Name of Conservator: <i>K. MARTIN</i>				Item & No: <i>DISSERTATION</i>				Date: <i>12/6/18</i>					
		1		2		3		4		5		6		DYESTUFF <i>LANASET</i>	
% (C)	Depth of Shade →P (total % Individual dye)	<i>0.25</i>		<i>0.5</i>		<i>1.0</i>		<i>1.5</i>		<i>2.0</i>		<i>2.5</i>		Material	
<b>Total Mls Dye</b>		<i>0.5</i>		<i>1</i>		<i>2</i>		<i>3</i>		<i>4</i>		<i>5</i>		<i>CREPELINE</i>	
DYE COLOUR & REF NO		%	ml	%	ml	%	ml	%	ml	%	ml	%	ml	Wt of sample (W)	<i>.4 g</i>
<i>0.2</i>	<i>BLUE 2R</i>	<i>100</i>	<i>.5</i>	<i>100</i>	<i>1</i>	<i>100</i>	<i>2</i>	<i>100</i>	<i>3</i>	<i>100</i>	<i>4</i>	<i>100</i>	<i>5</i>	Liquor Ratio	<i>50:1</i>
														Total Liquor (TL)	<i>20 ml</i>
														Notes:	
ADDITIVES (%P OR g/l)		(P) %/g/l	ml	(P) %/g/l	ml	(P) %/g/l	ml	(P) %/g/l	ml	(P) %/g/l	ml	(P) %/g/l	ml		
<i>10%</i>	<i>ALBA FLOW FFC</i>	<i>0.5</i>	<i>0.1</i>	→											
<i>1%</i>	<i>ACETIC ACID</i>	<i>1%</i>	<i>0.4</i>	→											
<i>1%</i>	<i>ALBEGAL SET</i>	<i>1%</i>	<i>0.4</i>	→											
<i>10%</i>	<i>SODIUM ACETATE</i>	<i>1</i>	<i>0.2</i>	→											
<i>20%</i>	<i>SODIUM SULPHATE</i>	<i>5%</i>	<i>0.1</i>	→											
CHECK & ADJUST pH															
TOTAL MLS DYE & ADDITIVES		<i>1.7</i>		<i>2.2</i>		<i>3.2</i>		<i>4.2</i>		<i>5.2</i>		<i>6.2</i>			
MLS WATER		<i>48.3</i>		<i>47.8</i>		<i>46.8</i>		<i>45.8</i>		<i>44.8</i>		<i>43.8</i>			
AFTER TREATMENT		%(P)	ml	%(P)	ml	%(P)	ml	%(P)	ml	%(P)	ml	%(P)	ml	%(P)	ml
		<i>1</i>	<i>0.4</i>	<i>1</i>	<i>0.4</i>	<i>1</i>	<i>0.4</i>	<i>1</i>	<i>0.4</i>	<i>1</i>	<i>0.4</i>	<i>1</i>	<i>0.4</i>	<i>1</i>	<i>0.4</i>
SAMPLE															

<sup>164</sup> Ann Milner, *The Ashford Book of Dyeing* (Christchurch: Shoal Bay Press, 1998), 54.

<sup>165</sup> Foskett and Mackert, 41.

Appendix VI: MSDS of pēbēo water-based colourless gutta



## SAFETY DATA SHEET

### 1 - IDENTIFICATION OF THE SUBSTANCE/PREPARATION AND OF THE COMPANY/UNDERTAKING

**Identification of the substance or preparation:**

Name: Gutta à l'eau toutes couleurs / water based gutta all colours.  
Product code: 147002.

**Company/undertaking identification:**

Registered company name: PEBEO INDUSTRIES.  
Address: 305 AVENUE DU PIC DE BERTAGNE - BP106 -.13881.GEMENOS CEDEX.FRANCE.  
Telephone: 33 (0) 4.42.32.08.08. Fax: 33 (0) 4.42.32.01.70.  
cedeyne@pebeo.com  
www.pebeo.com

**Emergency telephone: 33 (0) 1.45.42.59.59.**

Association/Organisation: INRS / ORFILA <http://www.centres-antipoison.net>.

**Use of the substance/preparation:**

Paint & Varnish

### 2 - HAZARDS IDENTIFICATION

This product is not classed as flammable. Refer to the recommendations regarding the other products present on the site  
This preparation is not classed as hazardous to health by directive 1999/45/EC.

### 3 - COMPOSITION/INFORMATION ON INGREDIENTS

**Hazardous substances present on their own:**

(present in the preparation at a sufficient concentration to give it the toxicological characteristics it would have in a 100% pure state)

This preparation contains no hazardous substance in this category.

**Other substances representing a hazard:**

No known substance in this category present.

**Substances present at a concentration below the minimum danger threshold:**

No known substance in this category present.

**Other substances with occupational exposure limits:**

No known substance in this category present.

### 4 - FIRST AID MEASURES

As a general rule, in case of doubt or if symptoms persist, always call a doctor.  
NEVER induce swallowing in an unconscious person.

### 5 - FIRE-FIGHTING MEASURES

Not relevant.

### 6 - ACCIDENTAL RELEASE MEASURES

**Personal precautions:**

Consult the safety measures listed under headings 7 and 8.

**Environmental precautions:**

Contain and control the leaks or spills with non-combustible absorbent materials such as sand, earth, vermiculite, diatomaceous earth in drums for waste disposal.  
Prevent any material from entering drains or waterways.

**Methods for cleaning up:**

Clean preferably with a detergent, do not use solvents.

**7 - HANDLING AND STORAGE**

The regulations relating to storage premises apply to workshops where the product is handled.

**Fire prevention:**

Prevent access by unauthorised personnel.

**8 - EXPOSURE CONTROLS/PERSONAL PROTECTION**

Use personal protection equipment as per Directive 89/686/EEC.

**Technical measures:**

Personnel shall wear regularly laundered overalls.

**9 - PHYSICAL AND CHEMICAL PROPERTIES**

**General information:**

Physical state: viscous liquid

**Important health, safety and environmental information:**

pH of the substance or preparation: neutral.

When a pH measure is possible, it has a value of: 7.50

Flash point interval: not relevant.

Vapour pressure: not relevant.

Density: > 1

Water solubility: Dilutable.

**Other information:**

VOC (g/l): 6.82

**10 - STABILITY AND REACTIVITY**

The preparation is stable at the handling and storage conditions recommended per § 7 of the safety data sheet.

**11 - TOXICOLOGICAL INFORMATION**

The preparation contains no substance classed as hazardous per directive 67/548/EEC.

No data is available regarding the preparation itself.

**12 - ECOLOGICAL INFORMATION**

No ecological data on the product itself is available.

**13 - DISPOSAL CONSIDERATIONS**

Do not pour into drains or waterways.

**Waste:**

Recycle or dispose of waste in compliance with current legislation, preferably via a certified collector or company.

Do not contaminate the ground or water with waste, do not dispose of waste into the environment.

**Soiled packaging:**

Empty container completely. Keep label(s) on container.

Give to a certified disposal contractor.

**Codes of wastes (Decision 2001/573/EC, Directive 2006/12/EEC, Directive 94/31/EEC on hazardous waste) :**

08 01 11 \* waste paint and varnish containing organic solvents or other dangerous substances

**14 - TRANSPORT INFORMATION**

Exempt from transport classification and labelling.

Transport product in compliance with provisions of the ADR for road, RID for rail, IMDG for sea and ICAO/IATA for air transport (ADR 2007 - IMDG 2006 - ICAO/IATA 2007).

#### 15 - REGULATORY INFORMATION

This preparation was classified in compliance with the directive known as <All preparations> 1999/45/EC and its adaptations. In addition directive 2008/58/EC with the 30° adaptation of directive 67/548/EEC (Hazardous substances) have been taken into account.

In addition directive 2009/2/EC with the 31° adaptation of directive 67/548/EEC (Hazardous substances) have been taken into account.

In addition Regulation (EC) No 1272/2008 have been taken into account.

This preparation is not classed as hazardous to health by directive 1999/45/EC.

This product is not classed as flammable.

#### Particular provisions:

NFPA 704 Label: Health=0 Flammability=1 Instability=1 Special Hazards=none



#### 16 - OTHER INFORMATION

Since the user's working conditions are not known by us, the information supplied on this safety data sheet is based on our current level of knowledge and on national and community regulations.

The product must not be used for any purposes other than those specified under heading 1 without first obtaining written handling instructions.

It is at all times the responsibility of the user to take all necessary measures to comply with legal requirements and local regulations.

The information given on this safety data sheet must be regarded as a description of the safety requirements relating to our product and not a guarantee of its properties

#### Full text of risk phrases appearing in section 3:

### Difference Report

Revision: N°5 (30/04/2009) / Version: N°1 (30/04/2009)

Revision: N°2 (10/06/2004) / Version: N°2 (08/06/2005)

#### 1 - IDENTIFICATION OF THE SUBSTANCE/PREPARATION AND OF THE COMPANY/UNDERTAKING

##### Use of the substance/preparation:

textile paint auxiliaries

Paint & Varnish

#### 8 - EXPOSURE CONTROLS/PERSONAL PROTECTION

Use personal protection equipment as per Directive 89/686/EEC.

#### 9 - PHYSICAL AND CHEMICAL PROPERTIES

##### Important health, safety and environmental information:

pH of the substance or preparation:

slightly basic.

When a pH measure is possible, it has a value of:

not stated.

pH of the substance or preparation:

neutral.

When a pH measure is possible, it has a value of:

7.50 .

**Other information:**

~~Decomposition point/decomposition range:~~

~~not specified:~~

VOC (g/l):

6.82

**11 - TOXICOLOGICAL INFORMATION**

No data is available regarding the preparation itself.

**14 - TRANSPORT INFORMATION**

~~Transport product in compliance with provisions of the ADR for road, RID for rail, IMDG for sea and ICAO/IATA for air transport (ADR 2005 - IMDG 2004 - ICAO/IATA 2005).~~

Transport product in compliance with provisions of the ADR for road, RID for rail, IMDG for sea and ICAO/IATA for air transport (ADR 2007 - IMDG 2006 - ICAO/IATA 2007).

**15 - REGULATORY INFORMATION**

~~In addition directive 2001/59/EC with the 28° adaptation of directive 67/548/EEC (Hazardous substances) have been taken into account.~~

In addition directive 2008/58/EC with the 30° adaptation of directive 67/548/EEC (Hazardous substances) have been taken into account.

In addition directive 2009/2/EC with the 31° adaptation of directive 67/548/EEC (Hazardous substances) have been taken into account.

In addition Regulation (EC) No 1272/2008 have been taken into account.

**Particular provisions:**

NFPA 704 Label: Health=0 Flammability=1 Instability=1 Special Hazards=none





## Appendix VII: Materials testing of pēbēo

### 1 Materials Testing: pH

The gutta resist was pH tested because degraded textiles can be sensitive to acidic or alkaline environments. In the case that the gutta is not completely removed from the silk support fabric and contained mild acidic or alkaline substances, it could weaken textiles or exacerbate an already chemically unstable environment.<sup>166</sup> Pēbēo gutta tested slightly alkaline when applied wet to pH testing strips (pH 7.5-8, as neutral is 7) in figure 34. As Thickett and Lee at the British Museum considered materials between 5.5-8.5 safe for use as storage or display materials, this material does not appear to pose a concern from the pH reading.<sup>167</sup>

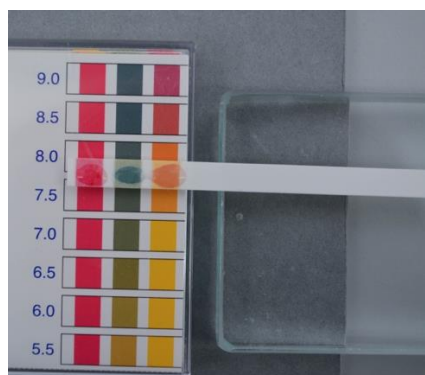


Figure 34: pH testing pēbēo gutta resist

### 2 Oddy testing

The Oddy test is an accelerated aging test created at the British Museum to detect the presence of volatile components (sulphides, chlorides, and organic acids) emitted from an organic material as it naturally ages, i.e. in storage or after treatment. Gutta was evaluated by Oddy testing before and after the fixing process by placing samples of silk crepe line with gutta resist in sealed glass test tubes with cleaned strips of copper, lead, and silver. Copper responds to sulfides, chlorides, and organic acids, lead to organic acids, and silver to

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<sup>166</sup> Tímár-Balázsy and Eastop, 36.

<sup>167</sup> D. Thickett and L. R. Lee, "Selection of Materials for the Storage or Display of Museum Objects," *British Museum Occasional Paper* no. 111 (London: the British Museum, 2004), 346.

sulfides that may be present in the tested material. As a control, one tube was sealed with silk crepeline without gutta resist and metal coupons, and one contained only coupons.

Eight Test tubes prepared (in figure 35):

- 1 blank - just coupons
- 1 control - just silk crepeline
- 2 gutta on glass slides
- 2 from the tube on silk not steamed
- 2 on silk after steaming, running under hot water until gutta dissolved and running under cold water



Figure 35: the sealed test tubes after ‘accelerated ageing’

The test tubes were placed in a Heraeus accelerating ageing oven for 28 days at 60° C (140° F) in figure 36. Afterwards, the samples were evaluated for corrosion on each metal coupon to determine if they released harmful gases. Although this test is widely used in conservation, it can be inconclusive and the tested materials may cause corrosion to alloys of copper, zinc, and silver in future use although they displayed no corrosion during the Oddy test.<sup>168</sup> As this test has been successful at reducing corrosion in museum collections, it is a useful tool when introducing a new material into a museum or conservation studio to minimise the risk of damage to conserved objects.<sup>169</sup>

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<sup>168</sup> Thicket and Lee, 346.

<sup>169</sup> Capucine Korenberg, et al., “Refinements Introduced in the Oddy Test Methodology,” *Studies in Conservation*, 63:1 (2018): 2.



Figure 36: Samples for Oddy test in Heraeus accelerating ageing oven

Table 5: Oddy test results

Test Tube	Material Tested	Test Result	Observation Ag / Pb / Cu
1	no materials	T-1	Ag – no tarnish
			Pb – no corrosion
			Cu – no corrosion
2	unscoured silk crepeline	T-2	Ag – no tarnish
			Pb – no corrosion
			Cu – slight areas of bluish corrosion and loss of shine
3	gutta on glass slide	T-3	Ag – no tarnish
			Pb – no corrosion
			Cu – bluish corrosion on ends and loss of shine
4	gutta on glass slide	T-4	Ag – no tarnish
			Pb – no corrosion
			Cu – no corrosion
5	gutta on unscoured silk crepeline – not steamed	T-5	Ag – black/blue tarnish around area closest to sample (at top of coupon in photograph)
			Pb – small white corrosion products overall

			Cu – brown and yellowish corrosion in area closest to sample with loss of shine in the metal
<b>6</b>	gutta on unscoured silk crepeline – not steamed	T-6	Ag – no tarnish
			Pb – no corrosion
			Cu- no corrosion
<b>7</b>	gutta on unscoured silk crepeline – steamed/rinsed	T-7	Ag – no tarnish
			Pb – no corrosion
			Cu – small amount of dark bluish corrosion around flaw in the metal coupon
<b>8</b>	gutta on unscoured silk crepeline – steamed/rinsed	T-8	Ag – no tarnish
			Pb – no corrosion
			Cu – no corrosion

The outcomes observed were:

- Only the blank coupons were completely unchanged.
- There were slight dark red and slight dull blueish areas on some copper coupons including one of gutta on silk, one of gutta washed from silk, one gutta on slide, and one silk without gutta.
- One sample of gutta on silk unwashed and unsteamed showed the most tarnish/corrosion of any of the test tubes. On the silver coupon in the area closest to the sample there was bluish-grey tarnish, small white corrosion products on the lead and yellowish-brown corrosion in the copper with loss of polish as seen in figures 37 and 38.

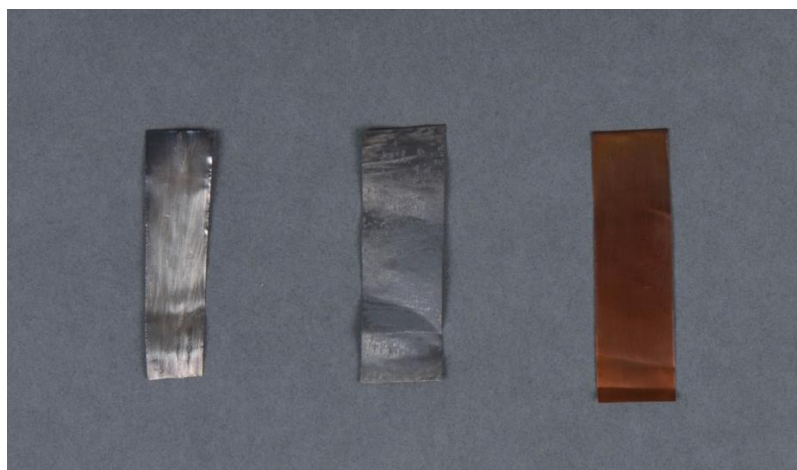


Figure 37: Coupons from silk with gutta - Front

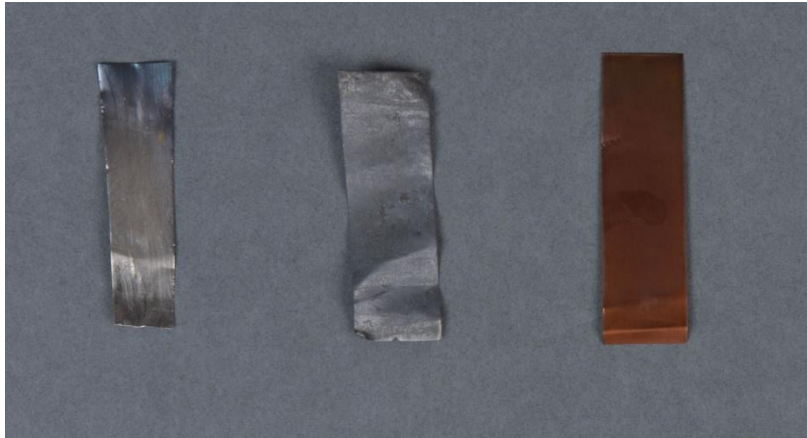


Figure 38: Coupons from silk with gutta - Back



Figure 39: Details of corrosion on lead coupon

### Conclusion

Based on these results, gutta could potentially emit organic acids with age: lead acetate, lead carbonate or plumbonacrite could be indicated by the white corrosion on the lead<sup>170</sup> (figure 39), and sulfides and chlorides as indicated by the corrosion on one unwashed sample.<sup>171</sup> Analysis with a technique like x-ray diffraction would be necessary to identify the specific corrosion products. Sulfides may also be released by the silk crepe line, since only the coupons in the blank test had no tarnish or corrosion. There was only a minimal reaction from the coupons where the gutta had been rinsed out indicating that the risk of deterioration may be reduced by thorough rinsing of gutta from the substrate. Photographs of all coupons are located in Appendix X. This method is generally quite subjective and the results were not consistent, therefore further testing is required to draw a firm conclusion regarding the ageing of gutta.

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<sup>170</sup> Korenberg et al.,3.

<sup>171</sup> Thicket and Lee.

### 3 Azide Test

An azide test was used to positively detect sulphur compounds in gutta resist. Pēbēo gutta was placed on a glass slide covering an area of 3 mm in diameter. A cover slide was placed on top and a drop of azide solution of sodium azide and iodine was placed at the edge of the cover slip and was drawn underneath by capillary action. The test revealed the presence of sulphides through the production of nitrogen bubbles when the azide solution reacted to the gutta resist.

The azide test was performed on three replicates each of wet and dry gutta. All wet gutta samples resulted in a positive result. However, the dry gutta samples produced inconsistent results – one test showed a positive result after a few minutes, another started to react after a few minutes and the last gave a negative result. This indicates sulfides are likely present in the gutta resist. Once the azide solution worked on the gutta for a few minutes, a chemical reaction occurred as the bubbles of nitrogen developed and grew larger on the outer edge of the gutta sample.

As gutta is used on support fabric and rinsed from the fabric before use, it is difficult to know how much of the sulphur compounds remain in the fabric. This highlights the importance of washing the gutta completely from the silk or support fabric since it may contribute harmful sulfides to the object if used in conservation.

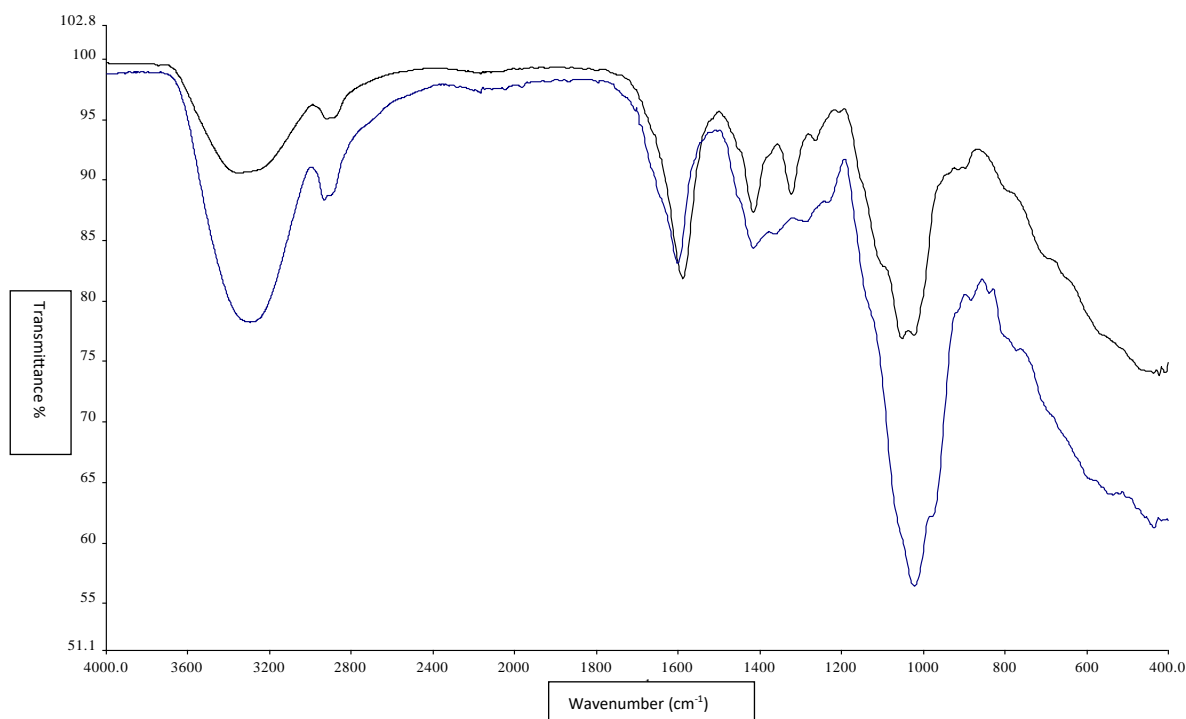
### 4 FTIR

Fourier-Transform Infrared Spectroscopy (FTIR) uses infrared energy to identify chemical groups within organic materials – specific bonds will respond to specific infrared frequencies by stretching, bending, or wagging.<sup>172</sup> This technique was used to determine what chemical bonds are present in pēbēo brand gutta resist. The FTIR spectrum resulting from a film of pēbēo water-based gutta has a similar to natural gums, especially acacia, a gum used in industrial printing of silk as dye thickener.<sup>173</sup>

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<sup>172</sup> Michele R. Derrick, Dusan Stulik and James M. Landry, "Infrared Spectroscopy in Conservation Science," *Scientific Tools for Conservation* (Los Angeles: The Getty Conservation Institute, 1999).

<sup>173</sup> Storey, 93.



Graph 4: pēbēo gutta (in blue) compared to acacia gum (in black).

The spectra of natural gums demonstrate a peak at  $1620\text{cm}^{-1}$  (partially associated with bound water within the molecules and the presence of a carboxyl group) and a broad peak around  $1080\text{cm}^{-1}$  due to the C-O bond.<sup>174</sup> These are also present in the gutta spectra. However, this is not fully conclusive evidence that gutta is a natural gum as there are non-corresponding peaks between the  $1500\text{-}1200\text{cm}^{-1}$  area.

### Conclusion

The FTIR analysis suggests that the gutta resist is a natural gum, although further analysis is needed. Gum arabic, a polysaccharide from the *Acacia* species, can degrade by hydrolysis and photo-oxidation which can cause discolouration, and the molecules can be cross-linked, making it hard and brittle.<sup>175</sup> However, further research will be required to determine how readily these deterioration mechanisms are catalysed. This could be undertaken by taking FTIR spectra of aged gutta films and comparing them to the spectra of the unaged films to attempt to identify any chemical changes.

<sup>174</sup> Michele R Derrick, Dusan Stulik, and James M. Landry, "Infrared Spectroscopy in Conservation Science," *Scientific Tools for Conservation* (Los Angeles: Getty Conservation Institute, 1999), 108.

<sup>175</sup> Velson Horie, *Materials for Conservation: Organic Consolidants, Adhesives and Coatings*, 2<sup>nd</sup> ed. (Abingdon: Routledge, 2010), 226.

## Appendix VIII: MSDS for cyclododecane

Material Safety Data Sheet  
According to regulation (EC) No. 1907/2006 (REACH)

### 87100 - Cyclododecane



PIGMENTE  
Printed: 11.06.2010

Revised edition: 07.03.2005

#### 1. Identification of the Substance/Preparation and of the Company/Undertaking

##### Identification of the Product

Product Name: Cyclododecane  
Article No.: 87100  
Use of the Substance/Preparation: Artists' and Restoration Material

##### Company

Company: Kremer Pigmente GmbH & Co. KG  
Address: Hauptstrasse 41-47, D 88317 Aichstetten  
Tel/Fax: Tel +49 7565 91120, Fax +49 7565 1606  
Internet: www.kremer-pigmente.de, kremer-pigmente@t-online.de  
Emergency No.: +49 7565 91120, Mon-Fri 8:00 - 17:00

#### 2. Hazard Identification

Hazard designation: Dust can form explosive mixtures with air.  
When heated the vapors can form explosive mixtures with air.

#### 3. Composition/Information on Ingredients

Chemical Characterization: Cyclododecane 100 %; CAS No. 294-62-2; EINECS 206-033-9

#### 4. First Aid Measures

After inhalation: Remove person to fresh air. In case of complaints consult a physician.  
After skin contact: Wash with soap and rinse with plenty of water.  
After eye contact: After contact with molten material cool affected skin with water. Rinse open eyes with plenty of water for at least 15 minutes. Consult physician.  
After ingestion: Rinse mouth with water and drink plenty of water. If symptoms persist consult physician.

#### 5. Fire-Fighting Measures

Suitable extinguishing media: Foam, carbon dioxide (CO<sub>2</sub>), extinguishing powder, water spray.  
Protective equipment: Wear self-containing breathing apparatus and protective clothing.  
Special hazards: When heated an explosive mixture with air can be formed.  
Dust can form explosive mixtures with air.  
In case of fire: formation of carbon monoxide and dioxide.  
Further information: Contaminated extinguishing water and debris should be disposed of according to local regulations.  
Contaminated extinguishing water and debris should be collected separately; avoid contamination of sewage system.

#### 6. Accidental Release Measures

Personal precautions: Keep away from sources of ignition. No smoking.  
Ensure adequate ventilation.  
Avoid dust formation, wear protective clothing.





Revised edition: 07.03.2005

Environmental precautions: Do not discharge into drains, surface or ground water.  
Methods of cleaning/absorption: Take up mechanically and collect in suitable container for disposal.  
Avoid dust formation.

## 7. Handling and Storage

### Handling

Instructions on safe handling: Provide adequate ventilation.  
Avoid contact with eyes, skin and clothing.  
Wear adequate protective clothing (see para. 8).  
Do not swallow or inhale.

Information on fire and explosion protection: Dust in combination with air can form an explosive compound.

Keep away from sources of ignition - do not smoke. Take precautionary measures against static discharges.

### Storage

Storage conditions: Store in tightly sealed containers in a cool and well ventilated location.

Storage compatibility: Do not store together with: food, drink and animal feeding stuffs.

## 8. Exposure Controls/Personal Protection

Additional information about design of technical systems: Provide adequate ventilation in case of dust formation.

### Personal protective equipment

General protective measures: The usual precautionary measures are to be adhered to when handling chemicals.

Do not inhale dust. Do not eat, drink or smoke during work. Wash hands before breaks and at the end of the shift.

Respiratory protection: None required under normal operating conditions.

If exposure limit is exceeded: Mask P3 (for solid and liquid particles DIN 3181).

Hand protection: Protective gloves

Do not wear cotton or leather gloves.

Protective glove material: Nitrile rubber (480 min, 0.35 mm)

Eye protection: Safety glasses with protective shields (EN 166).

Body protection: Wear heat resistant clothing and shoes.

Protective suit with long sleeves.

## 9. Physical and Chemical Properties

Form: amorphous  
Color: white  
Odor: light  
Melting temperature: 60.7°C  
Boiling temperature: 247°C  
Flash point: 98°C  
Ignition temperature: 175°C  
Lower explosion limit: 0.7 Vol%

Revised edition: 07.03.2005

Upper explosion limit:	7.5 Vol%
Vapor pressure:	1.33 kPa (100°C)
Density:	0.82 g/cm <sup>3</sup> (80°C)
Solubility in water:	0.01 g/l (20°C)
Viscosity dynamic:	2.21 mPa.s (65°C)
Coefficient of variation (n-Octanol/Water):	6.71 (logPow)

#### 10. Stability and Reactivity

Thermal decomposition/Conditions to be avoided:	Do not overheat.
Hazardous reactions:	Unknown.

#### 11. Toxicological Information

##### *Acute toxicity*

LD50, oral:	> 10000 mg/kg (rat; OECD TG401)
LD50, dermal:	> 34 mg/l
LD50, inhalation:	> 3160 mg/kg

##### *Primary effects*

Irritant effect on skin:	Non irritating (rabbit)
Irritant effect on eyes:	Non-irritating to eyes (rabbit)
Sensitization:	No sensitizing effect (guinea pig).
Mutagenicity:	Not mutagenic (Ames-Test)
Further toxicological effects:	Application: oral, NOEL: ca. 250 mg/kg (14d, rat; OECD TG 407).

#### 12. Ecological Information

Elimination (Persistency and Degradability):	3 % (28d); not readily biodegradable
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##### *Further information*

Water hazard class:	0
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#### 13. Disposal Considerations

Product:	Must be treated as toxic waste according to local laws and regulations.
Uncleaned packaging:	Recycling is possible when packaging is clean. Completely empty packaging can be disposed of with the regular waste.

#### 14. Transport Information

Further information:	Not classified as a dangerous good under transport regulations.
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#### 15. Regulatory Information

Water hazard class:	0, not hazardous
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Material Safety Data Sheet

According to regulation (EC) No. 1907/2006 (REACH)

**87100 - Cyclododecane**



**PIGMENTE**  
Printed: 11.06.2010

Revised edition: 07.03.2005

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**16. Other Information**

This product should be stored, handled and used in accordance with good hygiene practices and in conformity with any legal regulations.

This information contained herein is based on the present state of knowledge and is intended to describe our product from the point of view of safety requirements. It should be therefore not be construed as guaranteeing specific properties.

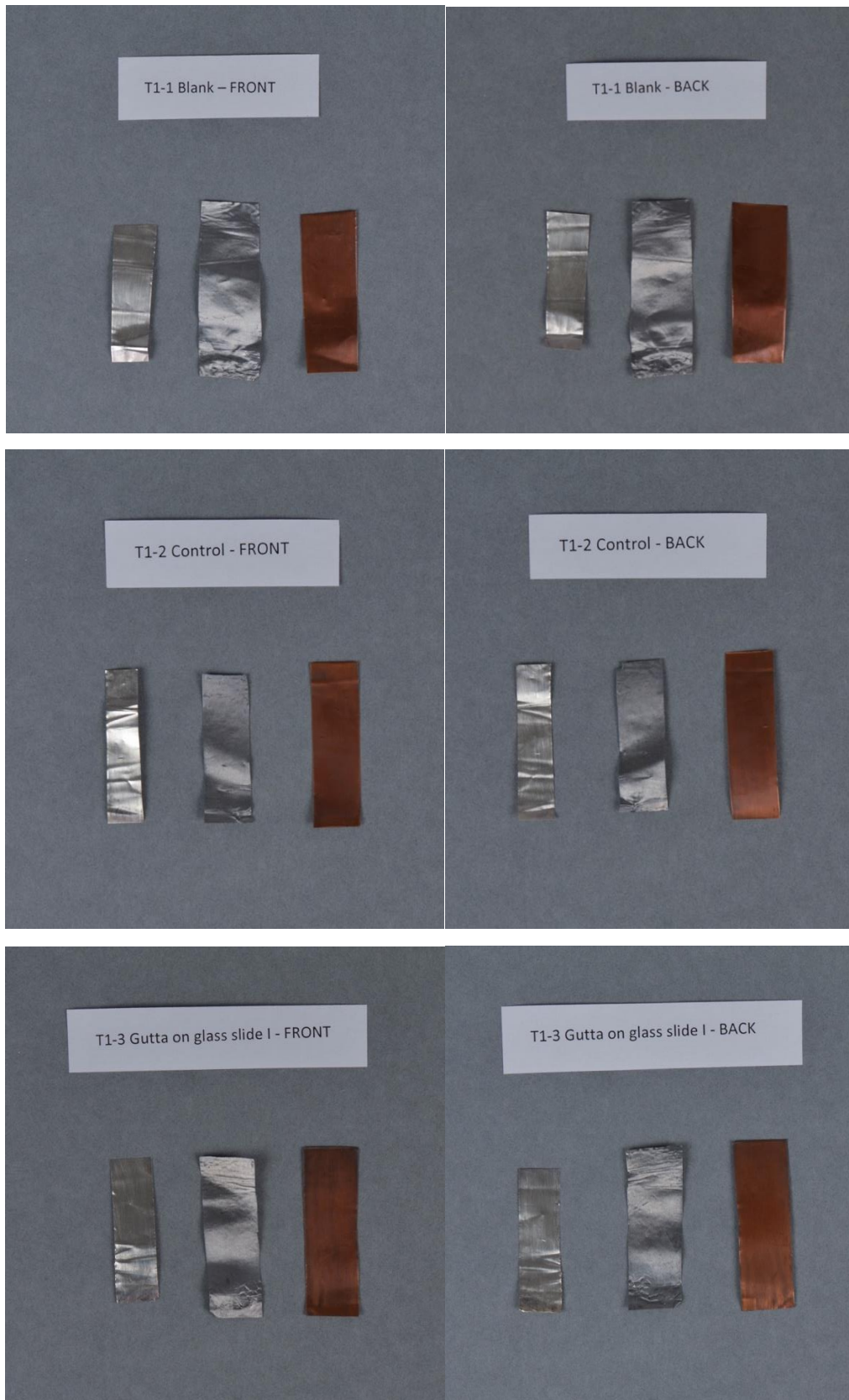
## **Appendix IX: Oddy testing procedures**

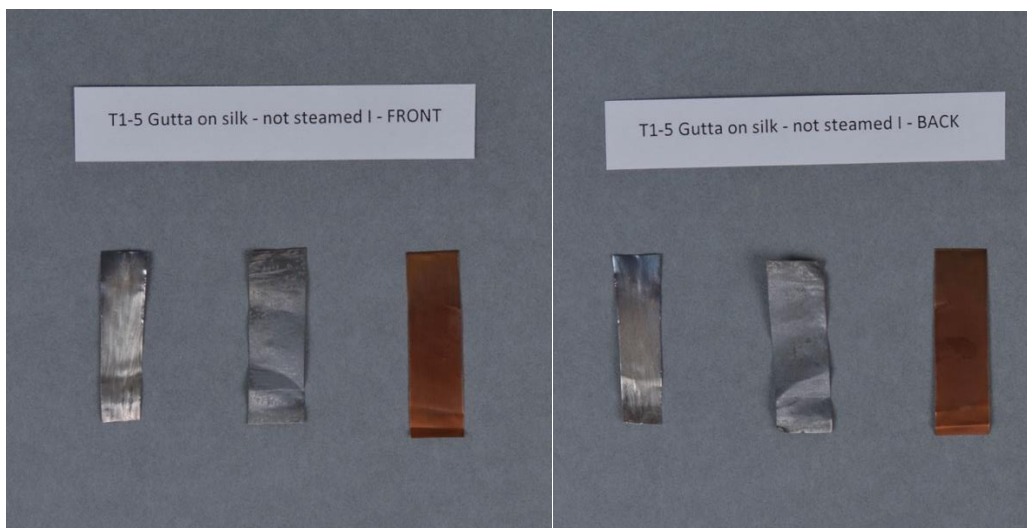
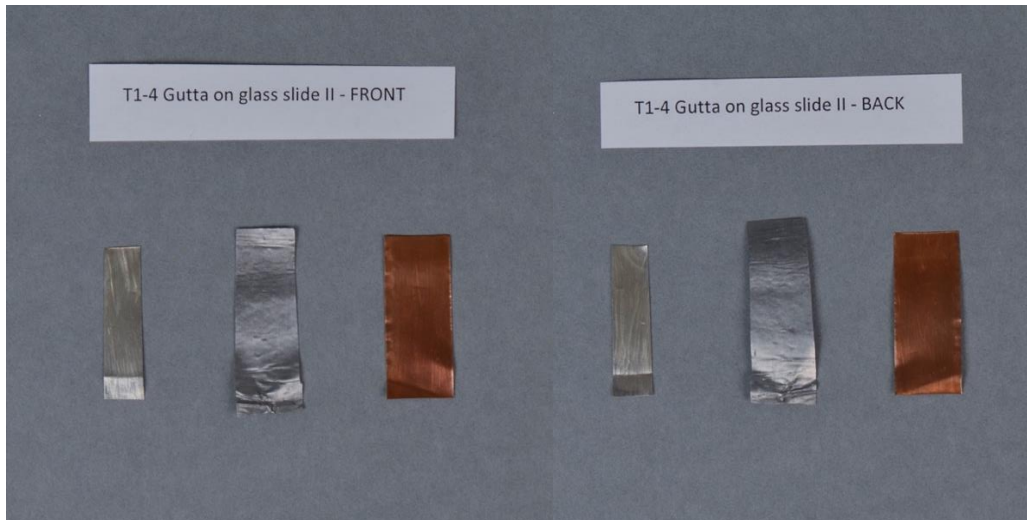
1. Eight test tubes, eight micro test tubes and eight silicone stoppers were soaked overnight in Decon 90 alkaline detergent at 2 % v/v and rinsed with deionised water.<sup>176</sup> They were dried in a Heraeus accelerating ageing oven at 60°C (140° F).
2. All eight test tubes were labelled with the contents above.
3. Eight silver, eight copper and eight lead coupons were abraded with a glass bristle brush on both sides in the fume cupboard until all fingerprints and tarnish was removed resulting in each coupon shiny and clean.
4. The coupons were soaked in three petri dishes half filled with acetone.
5. As each coupon was removed with tweezers from the acetone, it was blotted immediately with lint free tissues to prevent air drying.
6. One of each of the metal coupons were placed into three pre-cut grooves in the bottom of the silicone stoppers with lead in the centre position and copper and silver in the outer positions. None of the metal coupons were touching each other or the glass.
7. Each test tube was filled with as close to 2 g as possible of each the materials to be tested along with a micro test tube half filled with deionised water and stoppered with a small ball of cotton wool.
8. The silicone stoppers were inserted into each of the test tubes and the labelled test tubes were put in a tray in the oven at 60°C (140° F) for 28 days (figure 36).

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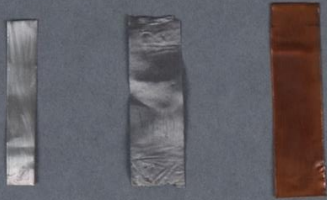
<sup>176</sup> Korenberg et al., 5.

**Appendix X: Photographs of metal coupons used in Oddy Test**





T1-7 Gutta on silk - steamed I - FRONT



T1-7 Gutta on silk - steamed I - BACK



T1-8 Gutta on silk - steamed II - FRONT



T1-8 Gutta on silk -- steamed II - BACK



**Appendix XI: Photographs of Replicates in the study after steam setting**



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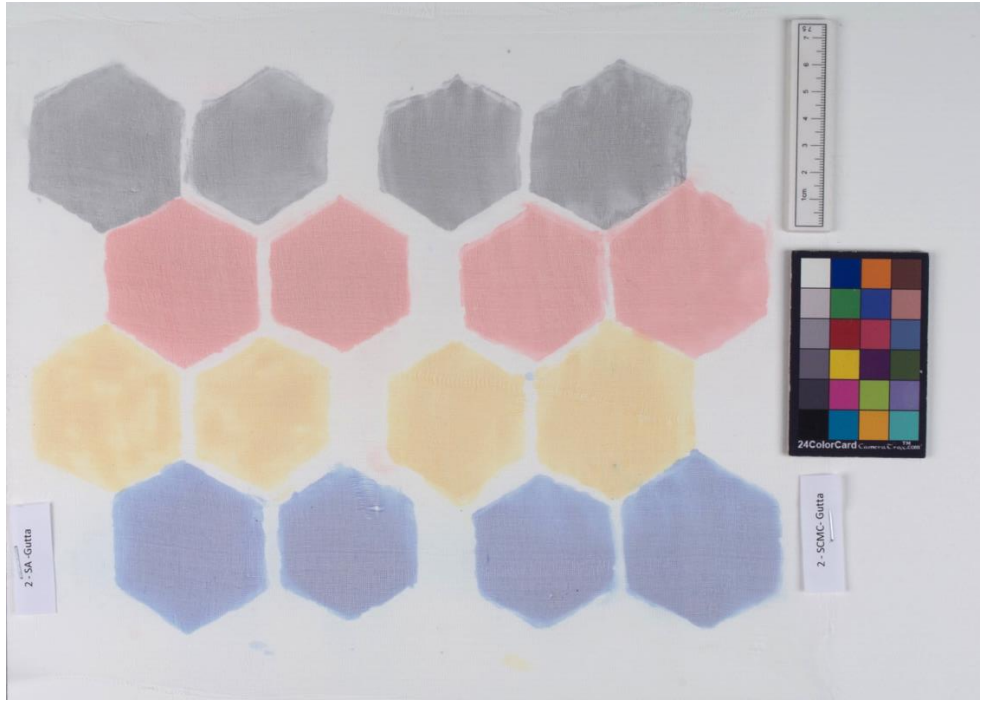
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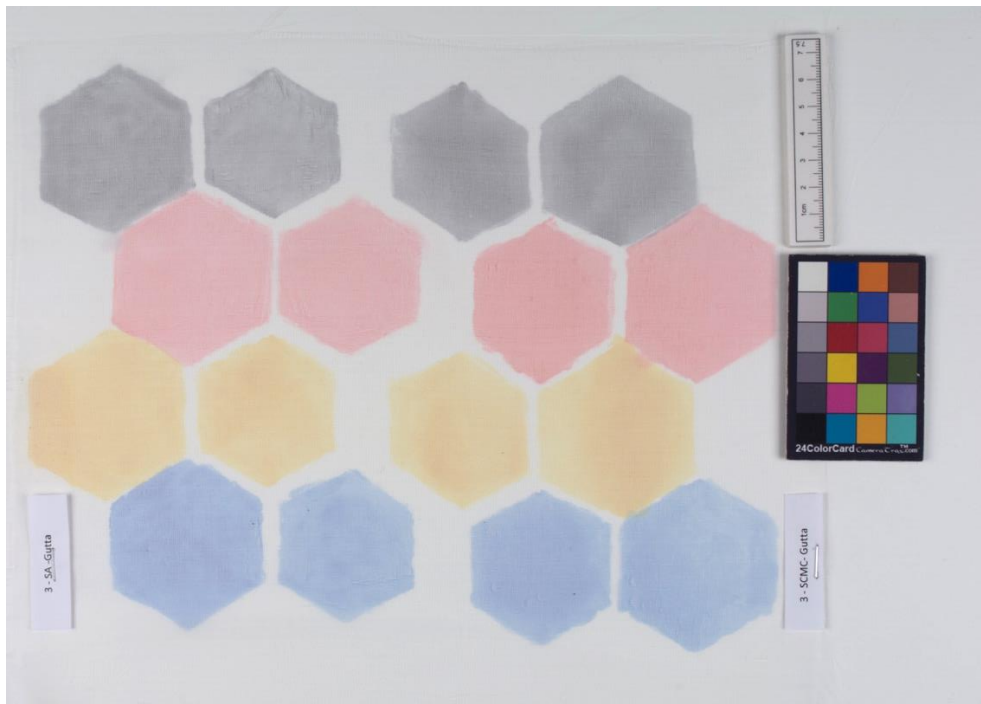
Melinex®



Gutta



Gutta



Gutta



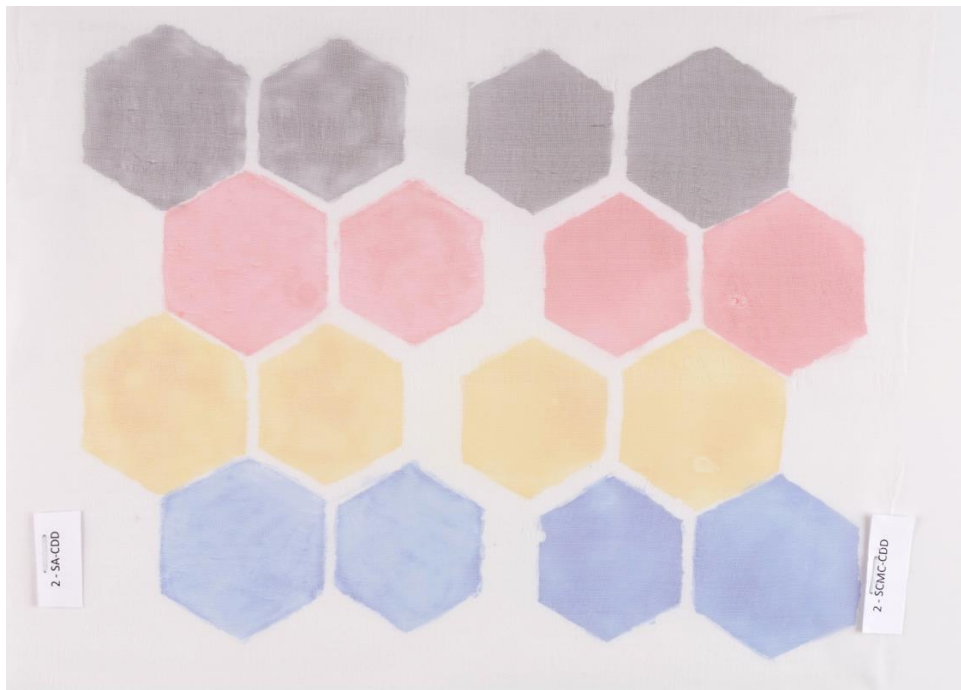
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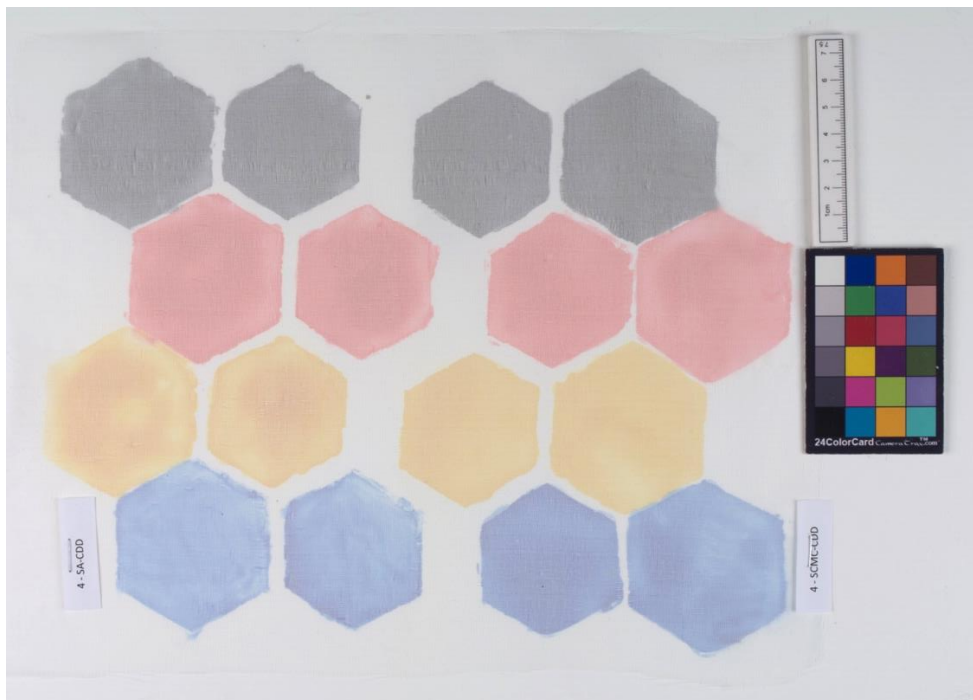
Cyclododecane



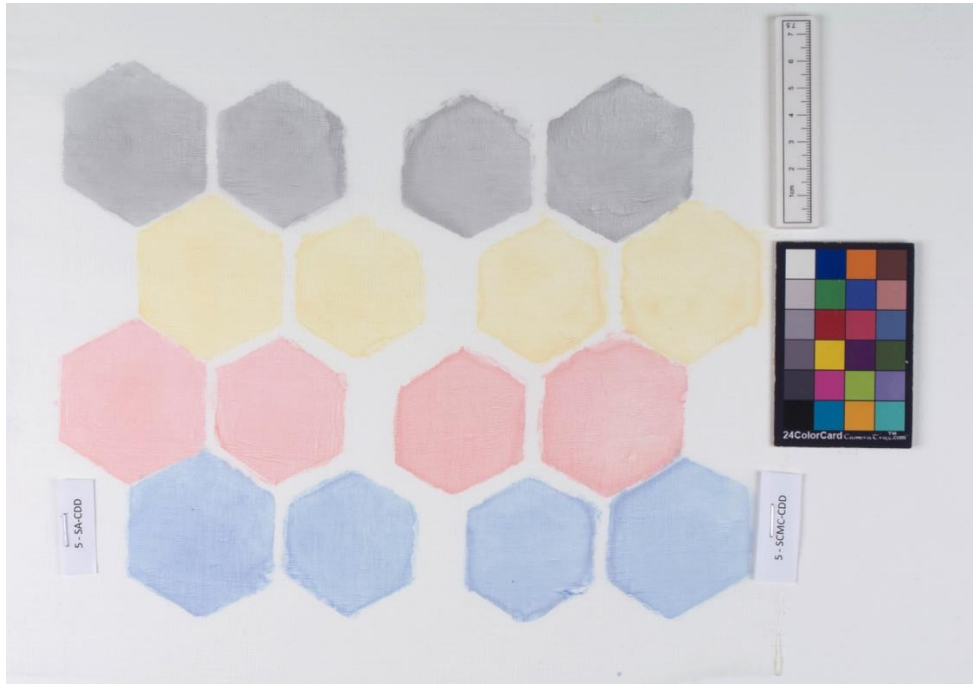
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