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title: Analysis of Evolon CR as a Poultice for Wax-Resin Lining Adhesives

subtitle: Py-GCMS, BET, and SEM Analyses of Evolon CR Tissues after Use

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abstract: Evolon CR, an absorbent fabric composed of polyester and polyamide microfilaments, is currently used in conservation for the purpose of reducing and removing overpaint and varnish from paintings. This study explores its use as a poulticing agent for the reduction of wax from wax-resin lining treatments. Experiments involving different methods of using the tissue, as well as analyses of the tissue after use employing various analytical methods indicate that it is most effective when used as a single sheet of solvent-wetted tissue. While the tissue can be used more than once, its ability to poultice wax-resin significantly decreases after its first use. Finally, the most important role in influencing the efficacy of poulticing is the solvent choice.

short\_title: Evolon CR as a Poultice for Wax-Resin Lining Adhesives

# <A-head> Introduction

Despite the successful use of Evolon CR (a nonwoven tissue made of a polyester and polyamide blend[[1]](#endnote-1)) in removing overpaint and varnish ({{Ribits 2017}}), little research has been done on its ability to poultice wax-resin mixtures. Confronted with two wax-resin-lined paintings that required relining, the authors sought a method that would achieve a homogeneous surface topography and remove residual wax-resin mixtures on the verso. The successful reduction of wax-resin mixtures ([**fig. 49.1a**](fig-49-1a)) prompted further research into the efficacy of this method and into the constituent materials poulticed into and onto the Evolon CR tissue. The goals of this study were to better understand the chemical and physical properties of Evolon CR and to evaluate the tissue’s efficacy at reducing wax-resin mixtures in an effort to improve and expand its practical use in conservation.

# <A-head> Experimental Design

Fragments of twentieth-century study collection paintings, previously wax lined by students with a 2:1 microcrystalline wax: Piccolyte resin, were used for this study. Following the removal of the old lining with heat, the adhesive mixture on the verso of the canvases was reduced using the following procedure (unless otherwise noted): Evolon CR squares measuring 2 inches (all weighing 0.18 g ±0.001 g) were placed onto the verso of the painting and then wetted with 1 mL of petroleum benzine, delivered by Eppendorf 1000 uL pipette. They were then covered with Mylar and left for varying lengths of dwell time (**figs.** [**49.1b**](fig-49-1)**,** [**49.1c**](fig-49-1)).

Four experiments were conducted to evaluate solvent dwell time on the surface, the potential to reuse the tissue, different application methods, and the solvent delivered with Evolon CR.

All tissues were manipulated with gloves to prevent transfer of oils or dirt from hands in the event this would impact weight gain or pyrolysis gas chromatography–mass spectrometry (Py-GCMS) analysis, and all were allowed to off-gas overnight.

Quantitative analyses included calculating the weight and porosity changes between unused and used tissues. The weight of the tissue was measured before and after each use for all four experiments on a scale accurate to 0.0001g. Porosity change analyses were conducted using Brunauer-Emmett-Teller (BET) theory. Qualitative tests included scanning electron microscopy energy dispersive electron spectroscopy (SEM-EDS) with back scattered electron (BSE) imaging, and analysis of poulticed organic materials with Py-GCMS. All experiments and analyses were conducted in 2019 at Winterthur Museum Scientific Research and Analytical Laboratory (SRAL) and the University of Delaware Advanced Materials Characterization Laboratory (UDAMCL).

## <A-head> Dwell Time

Experiment 1, Dwell Time, assessed the efficacy of Evolon CR left on the surface and under Mylar for 1, 5,[[2]](#endnote-2) and 15 minutes. Time points were selected to explore practical and extreme scenarios.

## <A-head> Single- vs. Double-Layer Application

Experiment 2*,* Single- vs. Double-LayerApplication, assessed the difference between applying a single square of Evolon CR versus a two-layer system (a single wetted square under a dry square covered with Mylar). This method was investigated to determine whether layering a dry tissue over a solvent-soaked Evolon CR tissue could increase the poulticing ability of the material. Both the Single-Layer and Double-Layer experiments were run in triplicate with a five-minute dwell time.

## <A-head> Iterative Use

Experiment 3,Iterative Use, assessed the effects of reusing the same Evolon CR tissue up to three times. The experiment aimed to explore the capacity of the material for reuse and the subsequent changes in efficacy.

## <A-head> Solvent Studies

Experiment 4, Solvent Studies, assessed the effect of using different ratios of acetone and petroleum benzine and how the solvent selection influenced the materials poulticed and retained in the tissue. Ratios of 10% and 50% acetone in petroleum benzine were compared to neat acetone and petroleum benzine alone.[[3]](#endnote-3)

# <A-head> Analytical Methods

## <B-head> *SEM-EDS*

SEM-EDS was used to analyze the Iterative Use experiments to visualize morphology changes within the tissue structure after repeated uses. Samples were examined using a Zeiss EVO MA15 SEM with LaB6 source at an accelerating voltage of 20 kV for the electron beam. Each of the runs were imaged at 25X, 50X, 137X, and 302X. SEM-EDS was conducted by Dr. Judy Rudolph, a volunteer scientist at SRAL.

## <B-head> *BET*

Porosity measurements of the tissue were conducted for the Iterative Use experiments to assess the effects of iterative tissue use compared to a control. Porosity measurements were done on a Micromeritrics BET analyzer (Micromeritics ASAP 2020). The pore-size detection limit was 10 nanometers. BET was conducted by Dr. Jing Qu, research scientist, and Gerald Poirier, director of UDAMCL.

## <B-head> *Py-GCMS*

Py-GCMS was used to analyze the Solvent Studies experiment to characterize low-molecular-weight solvent-extractable materials poulticed from the surface. Samples were analyzed using a Frontier Lab Multi-Shot Pyrolyzer (EGA/PY-3030D), a double-shot pyrolysis system interfaced to an Agilent 7820A gas chromatograph equipped with a 5975 mass selective detector (MSD). GC-MS analysis was conducted by Dr. Chris Petersen, a volunteer scientist at SRAL.

# <A-head> Results and Discussion

The first three experiments aimed to understand the practical features of Evolon CR, while experiment 4, Solvent Studies, was designed to understand whether the amide and ester functional groups within the tissue influence the material poulticed.

## <A-head> Dwell Time

Differences in the amount of material poulticed nearly doubles when the tissue is left to dwell for five minutes compared to one minute ([**fig. 49.2**](fig-49-2)). However, an extra ten-minute dwell only yielded an additional 11% increase in poulticed material. These results support that poulticing capacity decreases as the tissue approaches its saturation point. The fifteen-minute time point, representing an extreme scenario, was tested to understand the length of time it would take for the tissue to approach saturation. In practice, the ideal dwell time should be tested for each particular case.

## <A-head> Single- vs. Double-Layer Application

The use of a two-layer application proved less effective at poulticing material into the tissue compared to using a single sheet of Evolon CR ([**table 49.1**](table-49-1)). Wetted single-layer tissues consistently poulticed more material compared to both wetted tissues only and the combined wet and dry tissues from the Double Layer experiment. A possible explanation is that the dry tissue wicks solvent out of the wet tissue, reducing the amount of solvent delivered to the wax-resin mixture.

## <A-head> Iterative Use

In addition to assessing the efficacy of wax-resin poulticing through weight gain, this series also used SEM-EDS and BET to gather information regarding physical changes. Each use of the Evolon CR tissue resulted in increased material sorption, albeit with decreased amounts with each iteration ([**fig. 49.3**](fig-49-3)). This trend can be explained using a similar rationale as the decreased weight gain when using excessively long dwell times: as the tissue approaches saturation, its poulticing capacity decreases.

SEM images (**figs.** [**49.4a**](fig-49-4)**,** [**b**](fig-49-4)**,** [**c**](fig-49-4)**, and** [**d**](fig-49-4)) visualized morphology changes on the surface. With each use, the individual fibers become increasingly less distinct, presumably as the wax resin coats and fills the pores. The loss of definition with each use could be attributed to redistribution of the wax resin already poulticed into the tissue upon re-exposure to solvent, the continued filling of tissue interstices with freshly solubilized/softened wax resin, or a combination of both.

BET analysis yielded a surprising result ([**fig. 49.5**](fig-49-5)). An increase in measured surface area was observed after the first use, from 0.0982 m2/g (unused control) to 0.4315 m2/g (after single use). Contrary to the weight gain data and SEM images, both of which confirmed that the tissue had picked up wax resin, BET suggested the porosity increased. However, after the first iteration, BET data trended as expected: with each iterative use, the measured surface area decreased as more wax resin was poulticed into the tissue.

While this publication was in preparation, further analysis of the BET data revealed an anomaly. After further discussion with external scientific colleagues, our preliminary data suggests that BET might not be the ideal instrument for analyzing porosity in Evolon CR due to the relatively low porosity of the fibrous tissue compared with materials BET is typically used to analyze. Further research is needed to develop a quantitative method for measuring porosity changes in Evolon CR before and after use.

## <A-head> Solvent Studies

The 1:9 mixture of acetone and petroleum benzine gave the largest weight increase, followed by neat petroleum benzine ([**fig. 49.6a**](fig-49-6)). These results demonstrate a clear relationship between the choice of solvent and amount of poulticed material. The 1:9 mixture likely performed best due to acetone’s ability to solubilize resinous components in the wax-resin mixture. As expected, neat acetone resulted in negligible weight gain given its chemical incompatibility with nonpolar, aliphatic compounds. Importantly, this latter result also suggests that the chemical groups making up the Evolon CR (the polyamide and polyester fibers) have minimal effects in poulticing wax resin. The efficacy of poulticing appears to come primarily from solvent choice. Py-GCMS analysis ([**fig. 49.6b**](fig-49-6)) of the poulticed material showed the characteristic Gaussian distribution of hydrocarbon peaks from the wax component. As expected, the wax-based hydrocarbon peaks increased in intensity as the ratio of petroleum benzine to acetone increased.

In summary, the results from all experiments indicate that Evolon CR is an effective material for reducing wax resin; however, the tissue alone never resulted in complete removal of the adhesive.

# <A-head> Evolon CR in Practice

Much still remains to be understood about practical uses of Evolon CR, yet results from these experiments indicate that, under our testing conditions, it was less effective to use two layers compared to a single sheet of solvent-wetted tissue. Additionally, while Evolon CR can be used more than once, its ability to poultice wax-resin significantly decreases after its first use. Finally, our results indicate solvent choice plays an important role in poulticing, and that this role, at least for the removal of wax-resin, is stronger than that resulting from the chemical composition of the Evolon CR fibers. Our studies did not use controlled loading of Evolon CR, a recently developed parameter of application where the tissue is loaded with only a fraction of its maximum solvent capacity instead of full saturation ({{Tauber et al. 2019}}).

# <A-head> Future Directions

While most research on Evolon CR has centered around its use in varnish and overpaint reduction and removal, more study is needed to maximize the efficacy of Evolon CR at poulticing wax-resin lining mixtures. This can include testing aromatic/aliphatic solvent mixtures in differing ratios to most effectively target the wax-resin mixture.

To better understand whether solvents play a role in increasing the porous network of Evolon CR through a rearrangement of the microfilament bundles or if another physical or chemical interaction is at work, continued analysis on solvent-exposed Evolon CR is needed. These experiments should also be conducted on other common poulticing materials used in the field, including Tek Wipe and cotton blotting paper, for comparison.

# <A-head> Notes

1. See [https://evolon.freudenberg-pm.com/evolon\_technology/technology](about:blank). [↑](#endnote-ref-1)
2. Data for the five-minute time point is the average of the triplicate run from the Single-Layer experiment in the Single- vs. Double-Layer Application portion of the study. Note that the data for the one-minute and fifteen-minute time points are single runs (not an average). [↑](#endnote-ref-2)
3. Data for neat petroleum benzine solvent is the average of the triplicate run from the Single-Layer experiment in the Single- vs. Double-Layer Application portion of the study. Note that the data for the neat acetone, 1:9 acetone: petroleum benzine, and 1:1 acetone–petroleum benzine solvents are single runs (not an average of a triplicate run). [↑](#endnote-ref-3)