# Effects of Concentration and Artificial Ageing on the Strength and Reversibility of Dynmaic<sup>®</sup> 208 Wallcover Adhesive

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# **Chapter 1 - Abstract**

Deteriorated marouflaged murals have been treated by conservators in a variety of ways but little scientific testing on the techniques, their reversibility, and ageing characteristics exists in current literature. Maintaining intended aesthetic qualities of marouflaged murals during conservation and reattachment requires techniques similar to the original marouflage technique. Thus, there is a need to find adhesives that will remain chemically stable and mechanically reversible when used for re-adhering conserved murals to their original substrates. It is not the intention of this research to find the perfect adhesive but to provide a preliminary investigation into one commercial wallpaper adhesive.

Wallpaper adhesives exhibit the characteristics of an effective marouflage adhesive: viscous pastes that apply evenly, have a slow setting time, and a high level of tack. They are accessible, economical and non-toxic, making them very appealing. However as a commercial product, the ingredients are not easily available and their use in conservation often raises a number of questions. What are the ingredients and their ageing characteristics? What are the long-term effects of the adhesive on canvas murals? Will the adhesive remain reversible over time?

To address these questions, reattachment of a marouflaged mural was simulated using a wallpaper adhesive, Dynamic<sup>®</sup> 208 Clear Wallcovering Adhesive, to attach primed linen to a sealed drywall substrate. The concentration of the adhesive, and the addition of interleaving, was varied with the objective of finding a technique that would achieve ease of reversibility and maintenance of bond strength. Samples were subjected to tensile peel strength testing, FT-IR analysis and qualitative reversibility testing. Testing was done before and after thermal accelerated ageing to analyze the material composition, assess mechanical properties, and reversibility. It was hypothesized that diluting the wallpaper adhesive with an aqueous solution of methylcellulose might accomplish sustained strength and mechanical reversibility without the need for an interleaving layer, under particular conditions. This could lead to more accurate reattachment techniques for marouflaged murals and help to maintain the intended aesthetic qualities.

### **Chapter 2 - Introduction**

### **2.1 General Introduction**

In Europe during the 17th century there was a movement away from painting on solid supports towards more portable painting techniques, giving rise to the easel painting tradition. Particularly in France, traditional fresco painting techniques were replaced by mural painting techniques similar to those used in easel painting. This new mural painting technique involved the use of textile supports that the artist could work on in their studio instead of on high scaffolding in large public places<sup>1</sup>. Marouflage is the term often used to describe this mural painting process, which involves attaching a large-scale painting on canvas to a solid substrate. The solid substrate is often architectural, such as a wall or ceiling<sup>2</sup>. The complete canvas murals were often hung by specialized mural hangers, not the artist themselves<sup>3</sup>. Experienced hangers developed techniques with adhesive properties that were able to bond, "two materials which have widely varying properties, such as degree of expansion and contraction, porosity, etc"<sup>4</sup>. Ideally a marouflage adhesive needs the following characteristics: a viscous paste that is easy to apply, a slow to medium setting time, high initial tack and bond strength. Using a thick paste prevents dripping and unwanted mess during application. A high initial tack prevents the mural from falling during installation and slower setting times allow substantial working time so that the large murals can be properly positioned<sup>5</sup>.

The traditional marouflage hanging technique involved coating both the wall and the canvas with an adhesive paste using large spatulas and then flattening the canvas onto the wall with rollers. At times, the edges were tacked to hold the mural in place while it dried. If there were air pockets, the canvas was sometimes slit and more paste was applied before it was pressed down flat<sup>6</sup>. Traditional mural adhesive recipes of the 17th and 18th centuries include Burgundian pitch, wax, resin and red ochre. In the 19th century lead white paste was most common; it was a combination of lead white (ceruse), linseed oil, dammar varnish, Venice turpentine, wax and bone glue<sup>7</sup>. Aqueous adhesives have also been used such as: starch pastes, casein, animal glues and gum arabic. Contemporary techniques include: casein-latex cement (an emulsion of casein glue with rubber latex), LePage's contact cement, various acrylic adhesives and commercial wallpaper pastes<sup>8</sup>. Ralph Mayer noted the following about traditional marouflage adhesives and some contemporary alternatives in the book *The Artist's Handbook of Materials and Techniques* published in the 1940s,

Although aqueous adhesives and some of the newer synthetic adhesives have been used satisfactorily for mural-hanging purposes, especially by those who have given the processes considerable trial and study, the traditional white lead method remains the standard and others are generally regarded as substitutes. The principal disadvantages of aqueous adhesives as compared with the white lead paste are that they dry or set too rapidly for ease in application, and that the moisture is liable to shrink the fabric, loosen the ground, or cause vapor blisters. Professional hangers, however, can successfully mount the average good quality oil-primed canvas with wallpaper paste. Semi-gesso and other canvases which contain much aqueous binder must be very cautiously handled<sup>9</sup>. As Mayer suggests, paintings marouflaged with lead white paste are known to be the most durable but this technique is also known to be highly toxic and the oil can sink into the painting and cause discolouration<sup>10</sup>. Unfortunately, no technique is perfect and over time canvas murals become damaged and detached from their substrates due to a variety of factors.

Mural paintings are subject to physical, chemical and biological agents of deterioration. Since murals are often part of a larger structure the murals must be removed from their original surroundings if that structure becomes damaged; for example cracking walls, a need for construction or demolition<sup>11</sup>. Along with the building, natural disasters, vandalism, changes in taste, poor application technique as well as general wear and tear are some of the physical factors that negatively effect canvas mural paintings<sup>12</sup>. Chemical agents such as: inherent vice causing weakening of the adhesive layer, moisture damage, pollution, salt infiltration and previous conservation treatments also aid in deterioration<sup>13</sup>. In addition, canvas mural paintings are often composed of organic materials that are susceptible to biological deterioration such as human contact and microorganisms<sup>14</sup>.

Once a marouflaged mural painting has become damaged and detached, partially or fully, there are a number of approaches that can be taken to repair and conserve the piece. The current conservation approaches include: local consolidation and repair *in situ*, full removal with the addition of lining or interleaf and reinstallation in the original location, or full removal and remounting on a new solid support<sup>15</sup>. The biggest issue with removal of canvas murals is that it can significantly alter the intended aesthetic qualities of the piece, especially if it is not reattached exactly as it was originally. The addition of linings and solid supports strengthens the mural and adds elements of protection from agents of deterioration and hanging adhesives, but can change the way the mural interacts with its surroundings. A wide variety of adhesives have been successfully used to hang canvas murals over the centuries and there is currently no standard adhesive used. From the conservation perspective, one of the most important characteristics of an adhesive is reversibility. In the case of canvas mural paintings using moisture, solvents or heat for adhesive reversal and removal can be difficult; thus an adhesive that remains mechanically removable is ideal<sup>16</sup>. There are a number of adhesives and hanging techniques considered suitable for use in the conservation and reattachment of canvas mural paintings however; the properties of the various techniques, their reversibility and ageing characteristics have not been thoroughly investigated.

### 2.2 Statement of the problem

To conserve and reinstall a marouflaged canvas mural, while maintaining the intended aesthetic qualities, a hanging technique similar to the original technique would be ideal. Thus, there is a need to find adhesives that could be used to reattach conserved murals to their original substrates without the need for additional lining, interleaving or supports. This adhesive would ideally remain chemically stable, mechanically reversible and maintain a strong bond that could hold up against gravity. It is not the intension of this research to find the perfect adhesive but, to provide a preliminary investigation into one commercial wallpaper adhesive, Dynamic<sup>®</sup> 208 Clear Wallcovering Adhesive, that is

currently being used in the mural conservation field.

This study follows a recent mural conservation project carried out by the Centre de conservation du Québec (CCQ), where a commercial wallpaper adhesive was used to re-adhere a series of four canvas murals that had been conserved and interleaved<sup>17</sup>. Consultation with local conservators who have worked with marouflaged murals as well as research of available treatment reports confirmed that the use of commercial wallpaper adhesive in the conservation of canvas murals is currently common in the field. Wallpaper adhesives exhibit the characteristics needed in a marouflage adhesive; they are viscous pastes that are easy to apply evenly, have a slow to medium setting time, and a high level of tack<sup>18</sup>. Additionally, they are easily accessible, economical and commonly nontoxic; which makes them very appealing. However as with any commercial product, the ingredients are not easily accessible. The use of any commercial product in conservation treatments often raises a number of questions. What does the commercial wallpaper adhesive contain? What are the aging characteristics of the products ingredients? What are the long-term affects of the adhesive on the mural? Will the adhesive remain reversible over time?

#### 2.3 Purpose and Significance

To address the questions mentioned above, reattachment of a marouflaged mural was simulated using the wallpaper adhesive, Dynamic<sup>®</sup> 208 Clear Wallcovering Adhesive, to attach primed linen to a sealed drywall substrate. The concentration of the

adhesive, and the addition of interleaving, was varied with the objective of finding a technique that would achieve ease of reversibility and maintenance of bond strength. Samples were subjected to tensile peel strength testing, FT-IR analysis and qualitative reversibility testing. Testing was done before and after thermal accelerated ageing to assess mechanical properties, material composition and reversibility. It was hypothesized that diluting the wallpaper adhesive with an aqueous solution of methylcellulose might accomplish sustained strength and mechanical reversibility without the need for an interleaving layer, under particular conditions. The effects of interleaving on bond strength and reversibility will also be assessed. Results could lead to more accurate reattachment techniques for marouflaged murals that help to maintain the intended aesthetic qualities.

### **Chapter 3 – Background**

### **3.1** Literature review

Conservation of marouflaged murals has been approached several ways with countless variations in techniques and materials. A brief review of available literature and correspondence with a number of North American conservators is summarized below, highlighting current approaches and some treatment examples. The least invasive approach involves local consolidation and repair *in situ*. Some adhesives that have been used for local consolidation include: conservation grade acrylic adhesives such as Jade 403 and *Rhoplex 234*, methylcellulose, various commercial and homemade wallpaper adhesives. The Black House mural by Canadian painter John Hamound was conserved using localized techniques; injection of *Rhoplex 234* thickened with an aqueous solution of methylcellulose was applied to areas that had detached from the wall. The adhesive was dispersed and set with local application of heat and pressure using the set-up seen in Appendix II (*Figure i*)<sup>19</sup>. Alternatively, external circumstances or more extensive damage and detachment may require that a mural be fully removed from its solid support for conservation treatment and then reinstalled in its original location when treatment is complete. Treatments of this type usually take one of two forms: those that are structurally repaired locally and those that are lined or interleaved overall. Lining is a common treatment done to support a highly weakened canvas painting but often, with canvas murals, lining and interleaving have been applied for a second purpose. Lining

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and interleaving can act as a sacrificial separation layer that is applied as an attempt to maintain reversibility of the bond to the solid support overtime. Many types of conservation grade adhesives have been used to reinstall fully removed murals such as: Jade 403, Beva gel and Beva film. Often, the adhesive systems are complex, containing two or more layers of adhesive and lining materials. Clare Meredith and Colleen Donaldson (2005) explain the conservation treatment of the Lonsdale Frieze at Mount Stuart, which was first lined with *Beva 371*, sailcloth and Mylar. When the lining was complete the Mylar was covering the complete verso surface as a protective interleaf. The Mylar interleaf was then attached to the original walls with  $3M^{\text{R}}$  Fastbond adhesive<sup>20</sup>. Another treatment method involves fully removing a canvas mural from its original solid support and remounting on a new support. This technique is by far the most invasive and can greatly alter the way a mural interacts with its surroundings and change its intended appearance. Aluminum honeycomb board and expandable stretchers are examples of remounting supports used. An example of this type of treatment is described by Tomas Lahoda et. al. (2002), an expandable aluminum spring stretcher was used to display a large-scale canvas mural as an alternative to reattaching it to the original wall<sup>21</sup>.

Each technique has positive aspects and draw back. For instance, adhesives such *Beva 371* are known to be highly stable and reversible but the techniques of layering *Beva 371* with other adhesives is not proven to be reversible. In addition, *Beva 371* application requires even heat over the murals huge surface area for attaching and removal, which can be very difficult to achieve. Synthetic adhesives, such as *Jade 403*, are easy to use

and known to be strong and flexible but they can be difficult to reverse without the introduction of solvents. It is not practical to be working with chemicals in the public spaces that commonly house canvas mural paintings. Adhesives, such as those mentioned above and many more, require the application of heat or weight to set; which are difficult tasks when working on a large vertical surface that is inaccessible from the verso. Various layering techniques may maintain reversibility to a degree, but they are highly time consuming; lining or laying interleaf for a huge surface area can be very impractical. Finally, all conservation treatment alters the original appearance of the murals, though maintaining the way the mural interacts with its surroundings is integral to its authenticity and should be preserved whenever possible. Accordingly, it would be ideal to treat canvas murals using the least invasive techniques, avoiding linings and new supports if able.

It is often difficult to devise the least invasive as well as most successful longterm treatment plan for a damaged mural painting. The most important considerations are to give the mural the strength and support it needs, while maintaining reversibility and intended appearance. Michael O'Malley and his team at the Centre de conservation du Québec (CCQ) where faced with such a task in 2008 while devising a treatment plan for a series of canvas murals by Charles Huot (1855-1930). An example of this work, *La dernière Cène* (1892), can be seen before and after treatment in Appendix II (*Figure ii*)<sup>22</sup>. The Huot murals had developed areas of canvas that were detached and raised from the architectural support forming disturbing undulations in the painted image. Unfortunately, the detached areas could not be set flush with the wall without full removal of the canvas.

With limited time to complete the treatment, CCQ was not able to do much testing when deciding on the most appropriate reinstallation method. In consultation with the American Institute for Conservation (AIC), the commercial wallpaper adhesive Richard<sup>®</sup> R-070019 Heavy-duty Professional Grade Clear Wallcovering Adhesive was selected. The adhesive was briefly tested. A few primed cotton and linen canvas samples were prepared, some with an interleaving layer of *Reemay*, a non-woven polyester fabric, adhered with *Beva film*, a thermoplastic adhesive film<sup>23</sup>. These samples were glued to the original wall with Richard<sup>®</sup> adhesive and left for 24 hours. Even though the adhesives suggested setting time is one week, it was found that the samples adhered to the wall without an interleaving layer were nearly impossible to remove mechanically after 24 hours. As a result, the treated murals were interleaved with Reemay and Beva film prior to reinstallation because it was believed that the *Reemay* would provide sacrificial separation layer and this would maintain a level of reversibility in the future. Though a decision was reached and the treatment was complete there were still many questions left unanswered regarding the reattachment technique chosen and the use of commercial wallpaper adhesives in marouflaged mural conservation.

The results of researching and contacting conservators to find commercial wallpaper adhesive brands that had been used in the field of conservation were as follows. The brands referred to were; Roman Golden Harvest<sup>®</sup> GH-34<sup>24</sup>, Gardner-Gibson Dynamite<sup>®</sup> 780 clear strippable wallcovering adhesive<sup>25</sup>, Muralo<sup>®</sup> 8060 adhesive<sup>26</sup>, and Dynamic<sup>®</sup> Border Adhesive<sup>27</sup>. A few examples of their application are listed here. Golden

Harvest<sup>®</sup> was used, along with Shur-Stick<sup>®</sup> Wall Size<sup>28</sup>, for local reattachment of D.C. Lithgow's (1868–1958) canvas murals by the New York State Office of Parks, Recreation, and Historic Preservation Bureau of Historic Sites<sup>29</sup>. James Hamm (1988) discussed the successful use of Muralo<sup>®</sup> brand adhesives for the reinstallation of various lined and interleaved, hand painted wallpaper at the Martin Van Buren National Historic Site<sup>30</sup>. Unfortunately, only two of the brands mentioned in the literature and research were available for purchase in Canada, Richard<sup>®</sup> (the brand suggested by AIC and used by CCQ) and Dynamic<sup>®</sup>. After contacting the Head Office of Richard<sup>®</sup> Tools in Berthierville, Quebec it was found that the brand was no longer producing wallpaper pastes. Since, it would not be useful to run tests on a product that would soon be unavailable to conservators it was decided that further research would be done to find out if Dynamic<sup>®</sup> brand wallpaper adhesives would be an appropriate alternative; which would be accessible to conservators in the future.

# **Chapter 4 - Experimental Methodology**

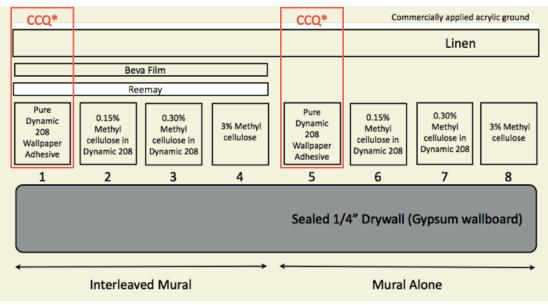
### 4.1 Materials

This research focused primarily on one commercially available wallpaper adhesive, Dynamic<sup>®</sup> 208 Clear Wallcovering Adhesive. Dynamic<sup>®</sup> was chosen over Richard<sup>®</sup> because, as mentioned in the previous section, the Richard<sup>®</sup> adhesive used by CCO with the Huot murals was not being produced anymore. The results of research and contacting conservators found that Dynamic<sup>®</sup> 208 had been used in conservation and was easily available. Additionally, FT-IR analysis confirmed that the Richard<sup>®</sup> adhesive and the Dynamic<sup>®</sup> adhesive were very similar in composition; thus the decision was made to focus on Dynamic<sup>®</sup> 208. Other commercial adhesives were examined but only as comparative samples. The comparative commercial wallpaper adhesives included were: Richard<sup>®</sup> Clear Wallcovering Adhesive R-070033, Richard<sup>®</sup> Heavy-Duty Adhesive R-070019 and Richard<sup>®</sup> Polyvinyl Boarder Adhesive R-070007. Additionally. *Methocel* A15C methylcellulose was used as a diluting agent with the Dynamic<sup>®</sup> adhesive and as a standard reference adhesive along with Paraloid B-72. A reference sample of B-72 was included because it is a conservation grade resin with known ageing properties<sup>31</sup>. The focus was on four types of adhesive solutions representing three concentrations of Dynamic<sup>®</sup> and a methylcellulose control adhesive which are illustrated in *Figure 1*. There were eight sample groups, and the adhesive concentration and interleaving were the only variation from sample to sample. The four adhesive solutions where paired with

interleaving (left side of *Figure 1*) and four without (right side of *Figure 1*). The Interleaving layer on half the samples was *Reemay* (0.008±0.002" thick) adhered with Beva film, following the interleaving technique recently used by CCQ on the Hout murals<sup>32</sup>. This interleaving technique was chosen by CCQ because *Reemay* and *Beva film* are both conservation grade materials with known long-term stability. The first adhesive solution was Dynamic<sup>®</sup> 208 adhesive used directly from the container for the pure aqueous solution of wallpaper adhesive in sample groups 1 and 5. These samples mimicked the tests done by CCQ with the Richard<sup>®</sup> adhesive. Dynamic<sup>®</sup> 208 was also prepared in two solutions of lower concentration: 0.15% methylcellulose in pure aqueous solution of Dynamic<sup>®</sup> 208 (groups 2 and 6)<sup>33</sup> and 0.30% methylcellulose in pure aqueous solution of Dynamic<sup>®</sup> 208 (groups 3 and 7)<sup>34</sup>. A viscous 3% w/v aqueous solution of methylcellulose (Methocel A15C) in distilled water acted as both the diluting agent for the adhesive solutions used in groups 2, 3, 6 and 7, and as the control adhesive solution used to adhere the canvas to the drywall in sample groups 4 and 8. Methylcellulose (*Methocel* A15C) was chosen as the diluting agent because it has the ability to carry a lot of water but maintain high viscosity and is known to have long-term stability.

The materials that remained consistent in the bonded samples were as follows. Gypsum <sup>1</sup>/<sub>4</sub>" drywall, meeting ASTM C1396/C1396M – 09 and C1597M – 04 standards, was selected to represent the wall substrate. Canvas murals are often found attached to either plaster or drywall, and both surfaces are often painted or sealed making them extremely similar<sup>35</sup>. Since drywall is inexpensive, readymade and easily accessible it was the best choice. The drywall was sealed with two coats of Behr<sup>®</sup> Premium Plus Ultra Paint &

Primer in One: Interior Eggshell Enamel Ultra Pure White No. 2750, before sample preparation. The painted mural was represented by linen canvas, commercially primed with acrylic gesso. Primed linen was chosen because linen is a support textile historically common in marouflaged murals and the primed surface mimics the painted surface of a canvas mural closely<sup>36</sup>. The weight of the primed linen was  $0.021\pm0.001$ ". When interleaved, the weight of the total textile was  $0.027\pm0.001$ ". Steel sheet metal ( $0.029\pm0.002$ " thick) was used as a support during mechanical testing and adhered to the verso of the drywall with epoxy. For the purpose of this study, it was assumed that the layered bonded sample of these materials was an adequate representation of a marouflaged mural attached to an architectural surface. Refer to *Figure 1* to see the eight sample groups and their layered structure.



\* Using Richards #R-070019 Clear Wallcovering adhesive

*Figure 1. Cross section of the layered structure of the eight types of samples investigated.* 

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#### **4.2 Sample Preparation**

Three types of samples were prepared for testing and they were prepared in two complete sets, one set underwent accelerated thermal ageing and one set did not. The unaged set will be called set 1 and the aged will be called set 2. The first type of sample prepared was for qualitative instrumental analysis with the FT-IR spectrometer. These were clean glass slides with each adhesive solution thinly applied to the surface and allowed to dry at room temp, refer to *Figure 2*. Two slides of the four main adhesives plus two slides of each comparative control adhesive were prepared, including two Paraloid B-72 film samples to act as standard samples (refer to Table 1). They all underwent FT-IR analysis, one slide before ageing and one slide after ageing.<sup>37</sup> For the second and third types of samples, reattachment of a marouflaged mural was simulated by using the four concentrations of adhesive to attach primed linen to a sealed drywall substrate. These samples were for tensile peel strength testing and qualitative reversibility testing. Both types were prepared as bonded samples with the varied layer structure mentioned above and seen in *Figure 1*. The basic structure of these samples is seen in Figure 3, a strip of drywall with a strip of linen (with or without interleaf) adhered using one of the adhesive solutions.

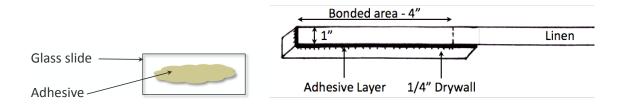
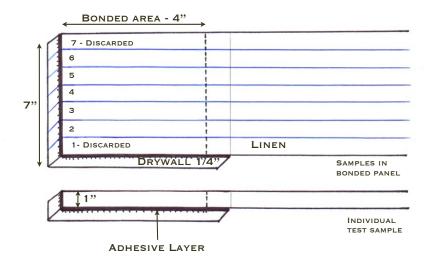


Figure 2. Adhesive sample slides for qualitative instrumental testing (FT-IR).

Figure 3. Structure of bonded samples used for peel tensile testing and qualitative reversibility testing.

According to recommendations made in ASTM D903-98, preparation of the bonded samples, for tensile peel strength testing and qualitative reversibility testing, was done in panels to reduce scatter<sup>38</sup>. Each of the eight types of samples were prepared as one bonded panel which was then cut down to form seven individual test specimens of each type and then the two outer samples (1 and 7) were discarded; this is illustrated in *Figure 4*. The panels were composed of a 7 x 7" square of sealed drywall with an adhesive layer applied evenly over 4 x 7" and then attached to linen or interleaved linen measuring 10-16" long and 7" wide. The adhered area was 4" instead of the 6" suggested by ASTM due to constraints imposed by the Instron Universal Vertical Tensile Tester used to assess the samples peel strengths<sup>39</sup>.



*Figure 4. Panel preparation of bonded samples for peel tensile testing and qualitative reversibility testing.* 

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For the bonded sample panels the individual materials were prepared and attached together as described below. The <sup>1</sup>/<sub>4</sub>" drywall was prepared by sealing it with two coats of primer and allowing the primer to dry for four hours between coats. Then the two finished paint layers were allowed to dry for 14 days as suggested by the manufacturer, before applying the adhesive layers. As mentioned in section 4.1, there were four types of adhesive layers tested in the bonded samples: pure Dynamic<sup>®</sup> 208 (full strength), 0.15% methylcellulose in pure aqueous solution of Dynamic<sup>®</sup> 208  $(5\% \text{ dilution})^{40}$ , 0.30% methylcellulose in pure aqueous solution of Dynamic<sup>®</sup> 208 (10% dilution)<sup>41</sup>, and 3% aqueous solution of methylcellulose alone (control). The aqueous methylcellulose was prepared as a weight/volume solution, 3g of methylcellulose in 100 mL of distilled water. The diluted Dynamic<sup>®</sup> adhesives were prepared as weight/weight (g of 3% methylcellulose solution/g of pure Dynamic<sup>®</sup> 208) solutions. For each of the sample groups 1-8, 40 samples (8 panels) were prepared: 20 for tensile peel strength testing and 20 for qualitative reversibility testing. A summary of the bonded samples prepared can be seen in *Tables 2* and *3*. Half of these, 10 samples, were prepared with linen interleaved with *Reemay* and *Beva film*. The interleaved textile was prepared using a hot suction table instead of a heated iron because it was more efficient and could provide more even heat application. The *Beva film* was first attached to the verso of the primed linen using heat and suction, and then the *Reemay* was attached on top of that in the same process. The hot-table was set at 70-75°C for both steps of the process and the Beva film and Reemay covered the full verso surface of the linen. Once the drywall substrate and the two textiles were prepared, they were adhered together. The adhesive layer was applied to the sealed

drywall and the un-primed side of the linen alone samples or the *Reemay* side of the interleaved samples was pressed against the adhesive layer to form a bond. Consistent adhesive application of  $6.4\pm0.03$ g per sample panel was achieved by weighing each drywall panel before and after the adhesive was applied evenly with a spatula. This ensured that all the panels had an equal amount of adhesive spread over the 4 x 7" bond area. The adhesive layer on the drywall was left for five minutes, as suggested by the manufacturer, before it was aligned with the linen component, pressed together and weighted overnight. The completed panels were then allowed to set for seven days at room temperature,  $20.7\pm0.7$ °C and  $34.5\pm3.5$ % RH, before being cut into seven 1" samples, as illustrated in *Figures 3* and *4*. The samples were separated by cutting through the canvas and scoring the drywall with a sharp utility knife; this was done before ageing for set 2 samples to prevent cutting defects. The two outer strips were discarded leaving five of the original seven samples ready for ageing and testing<sup>42</sup>.

Once cut, set 1 samples to be included in tensile peel strength testing were epoxied to metal supports, one on each side of the steel strip, as seen in *Figure 5*. Set 2 samples on the other hand, were aged before being epoxied to metal supports in the same format as set 1. The compositions of the completed samples for tensile peel strength testing were as follows and can be seen in *Figure 5*. The steel metal supports were 8" long by 1" wide and  $0.74\pm0.05$  mm thick. The drywall measuring 7" long by 1" wide by 1/4" thick was epoxied to either side of the steel. This part of each composite sample represented the architectural substrate. One of the four types of adhesive layers covered 4" of the sealed side of the drywall and was bonded to a 10-16" x 1" strip of

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commercially primed linen, with or without interleaving, which represented the painted canvas mural<sup>43</sup>. Also, each set included textile control strips measuring 14-16" by 1": ten primed linen samples, ten *Reemay* samples, and ten samples of primed linen interleaved with *Reemay* and *Beva film*.

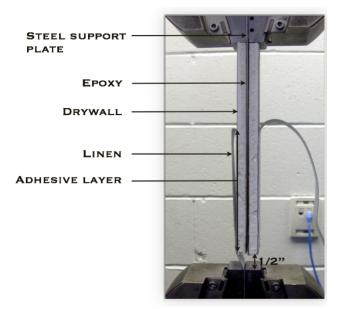


Figure 5. Orientation of the bonded samples during tensile peel testing.

The samples used for qualitative reversibility testing had almost the same dimensions but they only contained a sealed drywall substrate, an adhesive layer and primed linen or interleaved primed linen textile; no metal support was needed, refer to *Figures 3* and *4*. Furthermore, the primed linen strips with and without interleaving were shorter then in the tensile peel strength test samples at no more then 8" long.

A total of 241 individual samples were prepared for the two sample sets. A summary of the samples prepared for each type of testing can be seen in *Tables 1, 2 and 3*: 18 adhesive samples for FT-IR analysis (1 dry powder and 17 dried films), 80 bonded samples for reversibility testing, and 150 samples for peel testing (92 bonded samples and 58 textile control strips).

Qualitative Instrumental Analysis – FT-IR ATR						
Un-aged (set 1) Powder (no water)	Richards <sup>®</sup> R-070033	1				
	Pure Dynamic <sup>®</sup> 208 (groups 1 & 5)	1				
Un-aged (set 1) Dry Adhesive Film	0.15% methylcellulose in Dynamic <sup>®</sup> 208 (groups 2 & 6)	1				
	0.30% methylcellulose in Dynamic <sup>®</sup> 208 (groups 3 & 7)	1				
	3% methylcellulose (groups 4 & 8)	1				
	Approx. 0.80% methylcellulose in Dynamic <sup>®</sup> 208	1				
	Pure Richards <sup>®</sup> R-070033	1				
	0.30% methylcellulose in Richards <sup>®</sup> R-070033	1				
	2% methylcellulose	1				
	Pure Richards <sup>®</sup> R-070007 (Border adhesive)	1				
	Pure Dynamic <sup>®</sup> 208 (groups 1 & 5)	1				
	0.15% methylcellulose in Dynamic <sup>®</sup> 208 (groups 2 & 6)	1				
Aged (set 2)	0.30% methylcellulose in Dynamic <sup>®</sup> 208 (groups 3 & 7)	1				
Dry Adhesive Film	3% methylcellulose (groups 4 & 8)	1				
000C (50/ DIL 20 1.	Approx. 0.80% methylcellulose in Dynamic <sup>®</sup> 208	1				
80°C, 65% RH, 20 days	Pure Richards <sup>®</sup> R-070033	1				
	0.30% methylcellulose in Richards <sup>®</sup> R-070033	1				
	Pure Richards <sup>®</sup> R-070007 (Border adhesive)	1				
Total						

Table 1. Summary of adhesive films prepared for qualitative instrumental analysis.

	Mechanical Strength Testing	Group #	Interleaved Mural	Group #	Mural Alone	Interleaf Alone
Un-aged (set 1)	Pure Dynamic <sup>®</sup> 208	1	5	5	4 (5)	
	0.15% methylcellulose in Dynamic <sup>®</sup> 208	2	5	6	5	
	0.30% methylcellulose in Dynamic <sup>®</sup> 208	3	5	7	6	
	3% methylcellulose	4	5	8	5	
	Canvas control		10		10	
	Reemay Control					10
Aged (set 2) 80°C 65% RH 20 Days	Pure Dynamic <sup>®</sup> 208	1	6	5	5 (6)	
	0.15% methylcellulose in Dynamic <sup>®</sup> 208	2	7	6	7	
	0.30% methylcellulose in Dynamic <sup>®</sup> 208	3	6	7	7	
	3% methylcellulose	4	5 (6)	8	6	
	Canvas control		7 (8)		10	
	Reemay control					9 (10)
	Total		46 (146 have u	sable data	a out of 15	0)

Table 2. Summary of samples prepared for tensile peel strength (mechanical strength) testing.

Table 3. Summary of samples prepared for qualitative reversibility testing.

	Qualitative Reversibility Test	Group #	Interleaved Mural	Group #	Mural Alone
Un-aged (set 1)	Pure Dynamic <sup>®</sup> 208	1	5	5	5
	0.15% methylcellulose in Dynamic <sup>®</sup> 208	2	5	6	5
	0.30% methylcellulose in Dynamic <sup>®</sup> 208	3	5	7	5
	3% methylcellulose	4	5	8	5
Aged (set 2) 80°C 65% RH 20 days	Pure Dynamic <sup>®</sup> 208	1	5	5	5
	0.15% methylcellulose in Dynamic <sup>®</sup> 208	2	5	6	5
	0.30% methylcellulose in Dynamic <sup>®</sup> 208	3	5	7	5
	3% methylcellulose	4	5	8	5
	Total		8	0	

### 4.3 Instrumentation

### 4.3.1 Despatch LEA series Chamber 1-69 - Thermal accelerated ageing

As mentioned in section 4.2, two complete sets of samples were prepared, set 2 underwent accelerated thermal ageing (aged samples) and set 1 did not (un-aged samples). Thermal accelerated ageing of all set 2 samples for peel strength testing, reversibility testing and compositional analysis was carried out in a Despatch LEA series Chamber 1-69 at 80°C and 65% RH for 20 days<sup>44</sup>. The ageing parameters were chosen in consultation with literature and studies related to accelerated ageing of cellulosic materials and adhesives, particularly the work of Zappala-Plossi (1976/77), Feller (1990), and Dupont  $(2002)^{45}$ . Accelerated ageing was chosen since time for this study was limited and there was a need to see how the materials would change when the energy of the system was sped up, simulating the effects of time. It was understood that natural ageing would be ideal and preferred but unfortunately it was not possible in this situation<sup>46</sup>. Thermal ageing was chosen over light ageing because light ageing did not seem appropriate for an adhesive which would be behind a painted canvas mural and not exposed to much light in reality. Additionally, the thermal ageing would better penetrate all layers of the bonded samples and would be much faster<sup>47</sup>. It was assumed that all samples were exposed to the same conditions in all areas of the chamber and that 80°C was below the glass transition temperature (Tg) of all the materials involved.

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# 4.3.2 Nicolet Avatar 320 Fourier Transform Infra Red (FT-IR) Spectrometer – Qualitative Instrumental Analysis

The Nicolet Avatar 320 FT-IR Spectrometer with attenuated total reflectance (ATR) accessory was used to assess the composition of the four main adhesive solutions and the comparative control adhesives, before and after ageing, giving qualitative results. An image of the testing apparatus with attachment can be seen in Appendix II (*Figure iii*). The four main adhesives and the comparative control adhesives were tested with the FT-IR ATR set at 32 scans at 4-cm<sup>-1</sup> resolution. *EZ OMNIC* software collected the results and the spectra were compared to each other and with reference spectra from databases of infrared spectra using the *GRAMS/32* program supplied by Galactic Industries Corp. Since the Dynamic<sup>®</sup> 208 adhesive is a commercial product it was important to find out what its main ingredients were and compare this to the control adhesives and standard spectra in the database. In addition, the spectrometer was used to observe if any changes occurred in the composition of the adhesives during the accelerated ageing process.

### 4.3.3 Instron Universal TTDL Vertical Tensile Tester - Tensile Peel Strength Testing

Mechanical strength testing of the adhesive bonds was done in the form of tensile peel tests and was carried out using an Instron Universal TTDL Vertical Tensile Tester with a 1000 pound load cell and a cross head speed 1" per minute. Data was collected at one point per second for the first five seconds and then one point per 0.1 seconds onward. The samples were not pre-loaded and the machine was not in the pull-to-break mode. The computer program collecting the data was a home-built system and the data collected was input into *Microsoft Excel* software for further analysis. The peel test was chosen because it is a common testing method used to assess the strength of an adhesive bonding a flexible substrate to a rigid substrate and it is the method most often used when testing the strength of adhesive bonds between paintings and lining materials<sup>48</sup>. Moreover, this test has a very low peel angle, which mimics the mural removal method of prying the canvas away from the drywall substrate. It was assumed that the Instron pulled at the same angle for all samples and that the crosshead speed remained constant for all samples.

### 4.3.4 Leica M 651 Optical Microscope – Visual Examination

Optical microscopic analysis was used in conjunction with visual examination on each sample that underwent peel testing and reversibility testing at 10x, 25x and 40x magnification. The location of bond failure was closely observed in order to assess the mode of fracture and collect other information as to the nature of the bond and influences of the fracture location on the results.

### 4.4 Methods of Investigation

The two sets of samples were investigated using the previous instruments to assess the mechanical properties and mode of fracture of the adhesive bonds and analyze the composition of the adhesive. In addition, one more qualitative technique was used to assess the reversibility of the various concentrations of adhesive bonding linen alone and interleaved linen to a sealed drywall substrate. Set 2 samples underwent thermal accelerated ageing before any testing. After seven days of setting under the conditions stated in section 4.2, the set 2 panels were cut and seven samples from each of the eight test groups were placed into the Despatch LEA series Chamber 1-69. Along with the bonded samples, the textile control strips and the set 2 dry adhesive films for FTIR analysis were prepared as stated in section 4.2 and placed inside the ageing chamber. When all of the set 2 (aged) samples were complete and inside the Despatch ageing chamber it was set to 80°C and 65% RH and left for 20 days<sup>49</sup>. The set 1 (un-aged) samples were all allowed to set for 14 days in the controlled settings stated in section 4.2 and then testing of those samples began.

### 4.4.1 Qualitative Instrumental Analysis of the Adhesives

The thin adhesive films on glass slides were allowed to dry in the controlled conditions stated in section 4.2. After seven days the set 1 films were sampled by scraping a small amount of dry adhesive off the slide with a clean scalpel blade and placing it directly onto the ATR Golden Gate diamond. The set 2 adhesive films were sampled in the same way after accelerated ageing. It was assumed that the fraction of the sample placed on the diamond was pure and free from contamination of any kind. The collected spectra were compared to each other and with reference spectra in the database.

#### 4.4.2 Mechanical Strength Testing - Tensile Peel Testing and Visual Examination

Initially mock tests were run on the Instron to confirm that the mechanical testing procedure would be successful. Three bonded samples of pure Dynamic<sup>®</sup> 208 adhesive with the interleaving layer and three without, plus three 3% methylcellulose adhesive with interleaving and three without, were made according to the sample preparation proposed in section 4.2. To allow for immediate testing the samples were force dried in a low heat oven, set at 70°C, for 27 hours and then cut and epoxied to steel supports as described in section 4.2. Textile control strips (three primed linen samples, six *Reemay* samples, and three interleaved primed linen samples) were also prepared as explained in section 4.2 for mock tests. A summary of the samples involved in the mock testing can be seen in Appendix I (*Table i*). Mock testing confirmed that the tensile peel testing procedure on the Instron would work and mechanical strength testing of set 1 and then set 2 began.

Each bonded sample was oriented in the clamps of the Instron with the steel metal support in the top grip and the mural canvas in the bottom grip. The bottom grip was positioned  $\frac{1}{2}$ " from the drywall component of the sample and the canvas component was folded over and placed in the bottom grip leaving some slack, as seen in *Figure 5* showing the sample on the left is oriented for peel testing. The samples were run under controlled condition, 21.4±1.9°C and 22.9±0.9% RH, until 2" of the 4" bond area had been separated. The last 2" of bond area were left for other possible testing methods in the future<sup>50</sup>. This was achieved by marking the 2" point on the sample and manually stopping the crosshead and the data collection when the sample had been peeled to this

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exact point. It was assumed that all samples were pulled exactly 2". The data obtained from each sample was used to calculate the samples peel strength (N/m), which is equal to the average load (N) divided by the width of the sample (m); then the peel strength for each set of five similar samples was averaged and the standard deviation was calculated using *Microsoft Excel* software. After the tensile peel testing on the Instron was complete each sample was examined visually and under the optical microscope to observe and record the mode of fracture: cohesive, interfacial or material. Additionally, the location of bond failure was recorded, whether adhesive failure or interleaving failure, and other information that may effect the results<sup>51</sup>.

The textile control samples were run on the Instron until they failed completely or until a peak load was surpassed<sup>52</sup>. Each sample was placed into the clamps with the grips 7" apart and with as little slack as possible; the samples were not pre-loaded. The data obtained from each sample was used to calculate the ultimate tensile strength (UTS) (N/mm<sup>2</sup>), which is equal to the maximum load prior to failure (N) divided by the average cross sectional area of the sample (mm<sup>2</sup>); then the UTS for each set of similar samples was averaged and the standard deviation was calculated using *Microsoft Excel* software.

### 4.4.3 Qualitative Reversibility Testing

Qualitative Reversibility Testing was run on 80 bonded samples prepared as is stated in section 4.2 and seen in *Table 3*. This testing method was modeled after an experimental method used by Jane Down, at the Canadian Conservation Institute (CCI),

for assessing the reversibility of adhesive tapes<sup>53</sup>. For this test set, the samples adhered with 3% aqueous solution of methylcellulose were considered the control samples. The procedure was as follows; five conservation students separated the adhesive bond between the painted mural component and the drywall component of 16 samples each, eight un-aged and eight aged. They then rated the samples based on ease of separation. It was assumed that there was no time constraint on removal. The removal technique was careful separation with a microspatula and removal of any remaining adhesive from the verso of the canvas with a scalpel with the addition of moisture if necessary. Each sample was rated as Very Easy given a rating of 100%, Easy (75%), Difficult (50%), Very Difficult (25%), or Impossible given a rating of 0%.

The ratings were defined as follows:

VE = very easy to separate and given a score of 100% (i.e., canvas comes off with only slight or no adhesive to remove from the verso, is quick to do, a large area can be removed easily with a good result and with no damage to the "paint film") E = easy to separate and given a score of 75% (i.e., canvas comes off with more then 50% adhesive to remove from the verso, it is not as quick as above, but a large area can be removed easily with a good result and with no damage to the "paint film")<math>D = difficult to separate and given a score of 50% (i.e., separation may require some moisture, it is slow to do and there is less success separating mechanically, adhesive on the verso is hard to remove mechanically, can only remove small areas, and there is possible damage to the "paint film")

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VD = very difficult to remove and given a score of 25% (i.e., separation requires moisture, it is slow to do and there is minimal success separating mechanically, adhesive on the verso is hard to remove mechanically, can only remove small areas, and there is possible damage to the "paint film")

I = impossible to remove and given a score of 0% (i.e., not removable by mechanical technique even with moisture)

Each student filled out a form to collect the results by filling in the rating for each of the 16 samples along with notes. The format of the data collection sheet and the testing set-up can be seen in Appendix I (*Table ii*) and Appendix II (*Figures iv and v*) respectively. The rating system was explained in detail to participants before the testing began and written versions of the definitions were provided for reference during testing. Once the rating system was explained it was assumed that all participants understood the rating system and answered the survey truthfully.

For each of the conservation student's ratings, if staining occurred 3% was subtracted from the score, 2% if slightly stained, and 1% if very slightly stained. Once the results were collected, an overall average for each sample group (1-8) was calculated by averaging all the results together, un-aged and aged, of a single sample group. For example, all the aged and un-aged samples of group 1 (*Figure. 1*) were averaged together. This gave an indication of the overall ease of removability by mechanical means for that one type of bond and then these results were compared with the other seven sample

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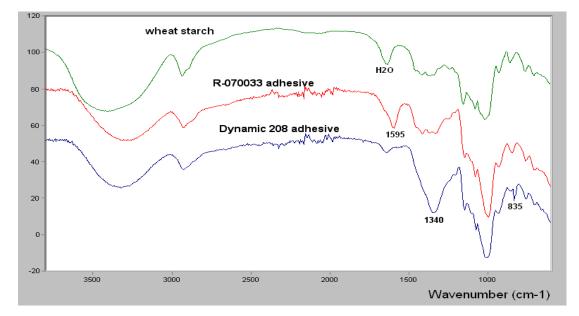
groups reversibility averages<sup>54</sup>. All the data was organized and analyzed using *Microsoft Excel* software to calculate average reversibility ratings and standard deviation.

## **Chapter 5 - Results and Discussion**

### 5.1 Qualitative Instrumental Analysis

When it was discovered that the brand name Richard<sup>®</sup> was no longer producing wallpaper paste, the decision was made to find a similar alternative. The results of research and contacting conservators to find wallpaper adhesives that had been used in the field of conservation led to a list of brands that were stated in section 3.1. All the brands referred to were selling a similar product described as a "clear strippable wallcovering" adhesive" but the ingredients of each was not available. Some products MSDS sheets mentioned starch but very few other ingredients, thus they were difficult to compare. The local hardware stores had three brands of wallpaper adhesive available Richard<sup>®</sup>, Zinsser<sup>®</sup> and Dynamic<sup>®</sup>. Since Richard<sup>®</sup> and Dynamic<sup>®</sup> were the only brands that had been referred to by a conservation source and Richard<sup>®</sup> was no longer being produced, the Dynamic<sup>®</sup> brand adhesive was purchased. It was then tested with FT-IR ATR (32 scans at 4 cm<sup>-1</sup> resolution) to see how closely it resembled the Richard<sup>®</sup> adhesive originally suggested by AIC and used by CCQ with the Huot murals. As seen in *Figure 6*, the spectrum of the Richard<sup>®</sup> R-070033 adhesive (the dehydrated powder version of R-070019) and the Dynamic<sup>®</sup> 208 adhesive both resemble the standard spectrum of wheat starch indicating that they are similar cellulose based adhesives. They have minor differences which are seen on the spectra at points 1595 cm<sup>-1</sup> for the Richard<sup>®</sup> adhesive and points 1340 cm<sup>-1</sup> and 835 cm<sup>-1</sup> for the Dynamic<sup>®</sup> adhesive. These peaks are likely due to different additives

or fillers. The availability of Dynamic<sup>®</sup> products in Canada and the similarities to the Richard<sup>®</sup> paste were the deciding factors in choosing Dynamic<sup>®</sup> 208 Clear Wallcovering Adhesive as an alternative to Richard<sup>®</sup> R-070033 adhesive and appropriate for further testing.



*Figure 6. Infrared spectra of two wallpaper adhesives compared to a standard spectrum of wheat starch* 

Initially, it was thought that the strong peaks at 1340 cm<sup>-1</sup> and 835 cm<sup>-1</sup> in the Dynamic<sup>®</sup> adhesive showed the presence of a filler, possibly an inorganic carbonate, but when compared with standard samples there was no inorganic carbonate spectrum that correlated with the peaks. Correspondence with Dynamic<sup>®</sup>'s customer service department reveled that the ingredient list for this product was protected and unavailable but they were able to provide an MSDS sheet for the product. The MSDS sheet stated that the paste was 24% solids and contained 3-7% sodium nitrate, a compound often used as a preservative.

When the Dynamic<sup>®</sup> adhesive spectrum was compared to a standard spectrum of sodium nitrate both unknown peaks correlated and a third smaller peak at 1790 cm<sup>-1</sup> also matched up perfectly, as seen in *Figure 7*. The strong peak at 1340 cm<sup>-1</sup> showed up as more of a broad band in the standard sodium nitrate sample but this could be due to physical properties of the compound, for example a more powdered then crystalline sample might cause such an effect<sup>55</sup>. Since all peaks in the spectra were accounted for by wheat starch and sodium nitrate it could be concluded that these are the main ingredients of the adhesive and that other additives and fillers are likely in the mixture in very small amounts (less then 1%).

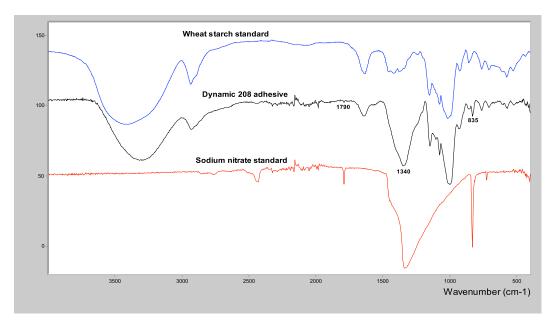


Figure 7. Infrared spectrum of  $Dynamic^{(R)} 208$  wallpaper adhesives compared to the standard spectra of wheat starch and sodium nitrate

Along with running FT-IR ATR spectra for the Richard<sup>®</sup> R-070033 adhesive and the Dynamic<sup>®</sup> 208 adhesive, other wallpaper paste and methylcellulose adhesive solutions,

seen in section 4.2 (*Table 1*), were scanned and compared. The 0.30%<sup>56</sup> methylcellulose in pure aqueous solution of Dynamic 208 did not show any evidence of methylcellulose in the spectra. Thus, a solution with even more methylcellulose, a 0.9%<sup>57</sup> solution of methylcellulose in pure Dynamic<sup>®</sup> 208 was tested, but again there was very little evidence of the methylcellulose in the spectra and this can be seen in *Figure 8*. Since the Dynamic<sup>®</sup> adhesive and methylcellulose are both cellulose based materials it could be possible that the spectra are too similar to show obvious differences. It may have been possible to identify the methylcellulose based on peaks related to the preservative in the methylcellulose but, the preservative was sodium chloride (NaCl); which has no chemical bonds and thus does not show up in FT-IR analysis. It was also possible that there was such a small amount of methylcellulose solids in the solutions that it could not be picked up by the FT-IR<sup>58</sup>.

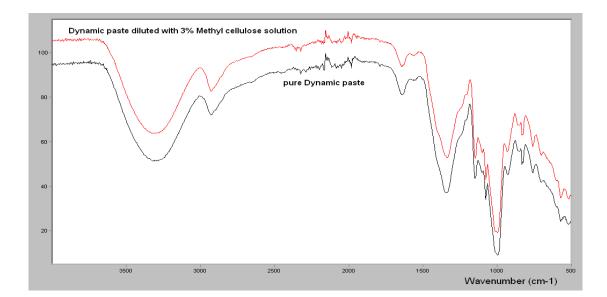


Figure 8. Infrared spectrum of pure Dynamic<sup>®</sup> 208 adhesive compared to a spectrum of a 30% dilution of the pure Dynamic<sup>®</sup> 208 adhesive with methylcellulose (0.9% solution of methylcellulose in pure Dynamic<sup>®</sup> 208)

Richard<sup>®</sup> Universal Border Paste R-070007 was also tested for comparison with the other wallpaper pastes. The results showed that the border adhesive was not similar to either Richard<sup>®</sup> R-070033 or Dynamic<sup>®</sup> 208 but, comparable to a Polyvinyl alcohol standard spectrum with indications of other ingredients such as filler, seen in Appendix II (*Figure vi*). The Dynamic<sup>®</sup> brand also carries a boarder paste product that could be similar to the Richard<sup>®</sup> boarder adhesive in composition thus, it is important to note that not all Dynamic<sup>®</sup> brand wallpaper adhesive products are similar in composition. Before using any Dynamic<sup>®</sup> brand wallpaper adhesive products in conservation testing the composition is suggested.

#### 5.1.1 Effect of accelerated ageing on adhesive composition

The four main adhesives and all the comparative control adhesives were tested with FT-IR before and after ageing, but little variation was seen in the spectra. *Figure 9* compares the spectra of pure Dynamic<sup>®</sup> 208 adhesive, before and after ageing, and clearly demonstrates that there was almost no change in the spectra after ageing, implying that the composition did not change. The comparison of the FT-IR spectra, before and after ageing, for all the other adhesives tested can be seen in Appendix II (*Figures vii - xii*). The un-aged slide of 0.15%<sup>59</sup> methylcellulose in pure aqueous solution of Dynamic<sup>®</sup> 208, gave a spectra that appeared to be contaminated and a new slide was made and retested giving a clearer result, this can be seen in Appendix II (*Figure vii*).

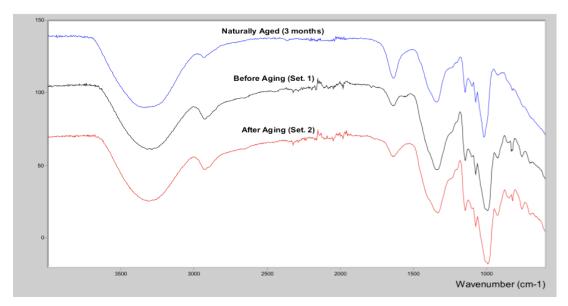


Figure 9. A comparison of the infrared spectra of pure Dynamic<sup>®</sup> 208 wallpaper adhesive before ageing, after three months of natural ageing and after accelerated ageing.

Though the FT-IR spectra showed no notable differences in the various concentrations and combinations of adhesives, before and after accelerated ageing, it was visually evident that all of the adhesives containing Dynamic<sup>®</sup> 208 or Richard<sup>®</sup> R-070033 had become discoloured. The discolouration ranged from shades of light yellow-brown to darker rusty brown as seen in *Figure 10*. As expected, the *B-72* standard control samples showed no visual change and no change in FT-IR spectra after ageing, as seen in the Appendix II (*Figure xii*)<sup>60</sup>.



Figure 10. Samples of various concentrations of Dynamic<sup>®</sup> adhesive and one pure Richard<sup>®</sup> adhesive sample (bottom left) as seen before ageing (BA) and after ageing (AA).

Under short-term natural ageing conditions, an interesting discovery was made about the various concentrations of Dynamic<sup>®</sup> 208 diluted with aqueous methylcellulose. The sealed containers were left in  $20.7\pm0.7$ °C and  $34.5\pm3.5\%$  RH conditions for approximately five weeks and all adhesive mixtures containing Dynamic<sup>®</sup> 208 and methylcellulose began to exhibit white mold growth. In contrast, the Dynamic<sup>®</sup> 208 alone and the 3% aqueous methylcellulose solution alone, under the same conditions, did not grow mold. As can be seen in *Figure 11*, the amount of mold growth was relative to the amount of methylcellulose in the solution. The solution that was 10% methylcellulose ( $0.30\%^{61}$  methylcellulose in pure aqueous solution of Dynamic<sup>®</sup> 208), seen on the left in *Figure 11*, had less mold growth then the solution that was 30% methylcellulose ( $0.9\%^{62}$  solution of methylcellulose in pure aqueous solution of Dynamic<sup>®</sup> 208), seen on the right. Once the mold layer was removed and the sealed jars were placed in a refrigerator, no further mold growth was observed. Through correspondence with customer service agents at both, Dynamic<sup>®</sup> and Dow Chemical (producer of Methocel A15C methylcellulose), it was revealed that both products, if left in water under these conditions, would eventually mold even though they contain preservatives such as sodium nitrate and sodium chloride as well as biocides. Since both cellulose-based products contained a large amount of water originally and methylcellulose is known to be highly effective at holding water in solution, the following was concluded. The methylcellulose likely held water around the starch based Dynamic<sup>®</sup> adhesive for an extended amount of time and this caused the starch to mold. This explains why the mixtures with more methylcellulose exhibited an increased amount of mold growth; more methylcellulose means more water held in solution and thus more mold growth occurs. Dow Chemicals suggested that to prevent this, one might be able to add more biocide to the mixture but that it should be the same biocide already in the Dynamic<sup>®</sup> adhesive. It is a good suggestion but unfortunately, as already mentioned, information about the biocide used in Dynamic<sup>®</sup> products is protected and therefore it would be impossible to add more of the identical biocide used in the original mixtures.



Figure 11. Comparison of mold growth seen on 10% dilution of Dynamic<sup>®</sup> 208 (left) and 30% dilution of Dynamic<sup>®</sup> 208 (right) after 5 weeks of natural aging in sealed jars.

#### 5.2 Mechanical Testing - Tensile Peel Strength

5.2.1 Effect of accelerated ageing and adhesive concentration on mechanical properties

The peel strength results for all sample groups are given in *Tables 4* (before ageing) and 5 (after ageing). The peel strength values vary from 70.0 to 323.5 N/m and the values in the tables are arranged from highest peel strength to lowest. An overall summary of the variation in tensile peel strength test results can be seen in Appendix I (*Table iii*). As seen in *Tables 4* and 5, the peel strengths did not follow the expected trend before or after ageing. It was expected that pure Dynamic<sup>®</sup> 208 would have the strongest bond, followed consecutively by the two adhesive solutions of lower concentration and then by the 3% methylcellulose controls (which is a concentration considered inadequate to be used as an adhesive)<sup>63</sup>. Additionally, it was expected that each interleaved sample would have slightly weaker bond strength than the corresponding sample without. Thus, it was predicted that the samples would be numerically ordered as follows: sample groups

5, 6, 7, and 8 (without interleaving) would be the strongest and in that order followed by sample groups 1, 2, 3 and 4 (with interleaving). It was also expected that the sample groups may follow this order: 5, 1, 6, 2, 7, 3, 8, 4, with each interleaved sample group falling right below the corresponding group without interleaving. Before ageing, the samples nearly follow the expected trends with some exceptions, as seen in *Table 4*. The lowest concentration (0.30%) of the Dynamic<sup>®</sup> adhesive with interleaving was expected to have a lower peel strength then it did and the pure Dynamic<sup>®</sup> adhesive with interleaving was expected to have a higher peel strength then it did.

There was some variation in the testing method of the un-aged samples; which is noted here. Though both the "mural alone" and the "interleaved mural" samples adhered with pure Dynamic<sup>®</sup> (seen in *Tables 4* and 5 marked with an asterisk\*) were pulled with only one sample on the metal support instead of the two sample arrangement used for all the other samples, this does not appear to have had an effect on the peel strength in any way. Additionally, in some cases the average peel strength was calculated from four samples instead of five. This occurred when one samples data was far outside the data of the other four samples and thus was excluded from the results (seen in *Tables 4* and 5 marked with a double asterisk\*\*).

Group #	Sample	Peel Strength (N/m) Before Aging	±SD
5	Pure Dynamic 208: Linen	303.8* **	42.4
6	0.15% methyl cellulose in Dynamic 208 (5% dilution): Linen	292.3	42.7
7	0.30% methyl cellulose in Dynamic 208 (10% dilution): Linen	282.1	36.5
3	0.30% methyl cellulose in Dynamic 208 (10% dilution): Interleaved linen	266.6	62.4
2	0.15% methyl cellulose in Dynamic 208 (5% dilution): Interleaved linen	257.3	27.1
1	Pure Dynamic 208: Interleaved linen	239.4*	66.5
8	3% methyl cellulose (control): Linen	128.6	23.8
4	3% methyl cellulose (control): Interleaved linen	107.6	14.3

Table 4. Peel strength (N/m) before ageing (set 1) in descending order.

Legend:

\* Were tested with only one sample attached to the metal support instead of 2

\*\* Average of only 4 samples instead of 5

The aged samples show less correlation with the expected trends, as seen in *Table 5*. After ageing, the lowest peel strength was given by the lowest concentration (0.30%) solution of Dynamic. This was expected since it was the most dilute solution, but the peel strength was significantly lower then any of the other sample types. This could mean that a dilution this low may be too weak for use with marouflaged murals but further testing would be required to confirm such a conclusion.

Group #	Sample	Peel Strength (N/m) After Aging	±SD	<b>%</b> - = Gain + = Loss
8	3% methyl cellulose (control): Linen	323.5 (D)(1S)	60.3	-151.6
1	Pure Dynamic 208: Interleaved linen	245.9 (D)(1E)(1S)	35.3	-2.7
5	Pure Dynamic 208: Linen	183.1(D)	41.8	39.7
4	3% methyl cellulose (control): Interleaved linen	145.6 (1E)(2S)	18.3	-35.3
3	0.30% methyl cellulose in Dynamic 208 (10% dilution): Interleaved linen	140.1 (1E)(2S)	26.8	47.4
2	0.15% methyl cellulose in Dynamic 208 (5% dilution): Interleaved linen	127.8 (2E)(1S)	64.6	50.3
6	0.15% methyl cellulose in Dynamic 208 (5% dilution): Linen	103.7(1E)	29.2	64.5
7	0.30% methyl cellulose in Dynamic 208 (10% dilution): Linen	70.0*** (1E)	33.1	75.2

Table 5. Peel strength (N/m) after ageing (set 2) in descending order.

Legend:

\*\*\* Below acceptable peel strength for a lining adhesive

(D) A damaged sample is part of this average

(IE) One edge sample (sample #1 or 7 from the composite panel) included in average

(2E) Two edge samples included in average

(IS or 2) One or two of the samples in the average separated at the wrong point during peel testing.

The ageing process caused two types of damage that were not conducive to maintaining useable samples; the first was overall moisture damage and the second was heat damage to the interleaving *Beva film* bond. The moisture, likely due to dripping water in the ageing chamber, caused lifting in various layers of the bonded samples due to weakening of the adhesives, the drywall paper and plaster. The damage and separation can be seen in *Figure 12* and a summary of the damage observed on the bonded samples after ageing is described in Appendix I (*Table iv*). In some cases the drywall plaster was

significantly dissolved where it came into contact with moisture. Additionally, a large number of the samples were stained on the primed surface of the linen and the adhesive layer in the composite samples had become discoloured. The staining is evident when the samples are compared before and after ageing as seen in *Figure 13*. The moisture damage likely occurred because the samples were too close to the walls of the internal chamber where moisture had collected. This may have been avoided if fewer samples were placed in the chamber at one time but due to time constraints this was not possible. Fortunately, less then 10% of the samples were badly damaged. Only three samples of 127 were withdrawn from further testing. Five samples, which had some damage, were still tested and were either included in the peel strength average and marked with a (D) in *Table 5* or edge samples from the sample panels (#1 or 7 of bonded panel seen in *Figure 4*) were substituted into the total set to replace the damaged samples. These are marked as (1E) or (2E) in *Table 5* depending on whether one or two edge samples are included in the peel strength average respectively.



Figure 12. Moisture damage on bonded samples after ageing

Effects of Concentration and Artificial Ageing on the Strength and Reversibility of Dynamic® 208 Wallcovering Adhesive - April 2010

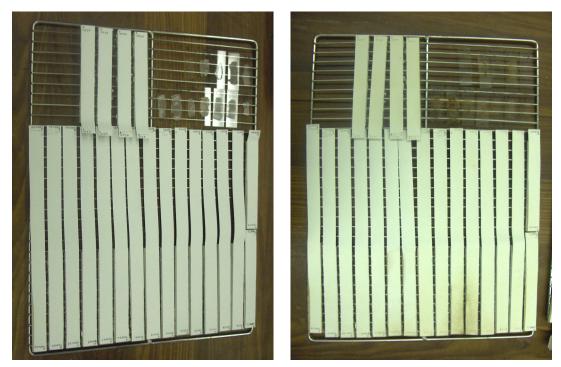


Figure 13. Comparison of samples before (left) and after (right) ageing. Staining of the primed linen and discolouration of the adhesives can be seen in the aged samples (right)

The second type of damage was more related to heat then moisture. Some of the aged samples (about 12%) exhibited separation of the *Reemay* interleaf from the "mural" linen after ageing and during peel testing. As seen in *Figure 14*, the *Reemay* interleaf was being peeled from the solid substrate but the "mural" linen was no longer attached to the *Reemay*. The accelerated ageing conditions, 80°C and 65%RH, were chosen based on conditions previously used for testing cellulosic adhesives and materials, but the high temperature may have caused the *Beva film* bond holding the interleaf to the linen to fail in some cases. Since *Beva film* s melting point is between 65-75°C it is possible that the 80°C was too high for the *Beva film* component of the bonded samples to maintain their bond. The heat could have caused the adhesive to melt, weaken and then more easily

separate under the stress of peel testing. The effect separation issues may have had on the peel strengths results will be discussed in section 5.2.2.

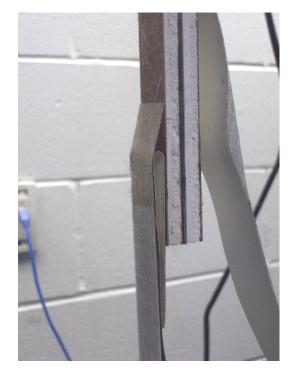
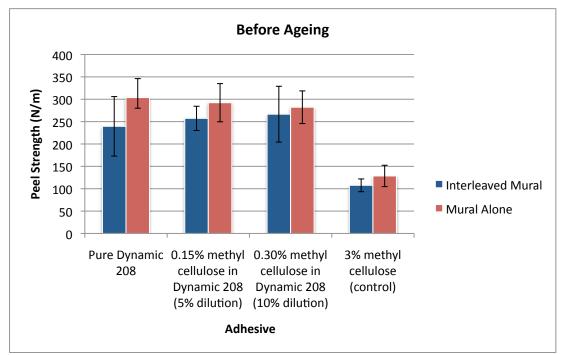
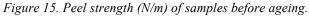


Figure 14. Example of interleaf separation seen during peel testing of a set 2 (aged) sample.

Overall, before ageing the "mural alone" samples formed a stronger bond then the interleaved samples with all concentrations of adhesive, as seen in *Figure 15*. After ageing, this flipped and the interleaved samples formed stronger bonds, with the exception of the methylcellulose control, as seen in *Figure 16*. The fact that the control samples, adhered with 3% aqueous methylcellulose, not only maintained the highest peel strengths after ageing but also gained strength is of particular interest. Methylcellulose in such a low concentration is not known to have good adhesive properties but overall

methylcellulose is known to remain strong and stable after  $ageing^{64}$ . Shashoua (1995) found that methylcellulose bonds adhering paper lap joints exhibited a 13% loss in tensile strength after ageing (70±1°C, 28 days), while gluten-free starch showed a greater loss in strength after ageing, at 25%. This same pattern of results could be the case with the *Methocel A15C* and Dynamic<sup>®</sup> 208, which is a starch based adhesive. This does not, however, explain why the methylcellulose not only maintained strength but gained strength after ageing, seen in Figure 15 and 16, and Table 5. Shashoua's study also found that methylcellulose formed strong bonds with cellulosic substrates and was able to maintain strong bonds with cellulosic substrates better than other natural adhesives in the study<sup>65</sup>. This could be a possible explanation for the high bond strength seen in the samples with methylcellulose in direct contact with the cellulosic linen and not the same strength with the samples bonded to the *Reemay* interleaved linen. Another possible explanation is that the 3% methylcellulose adhesive was applied thicker then the other three adhesive solutions improving the bond. Since the adhesives were applied based on weight the 3% methylcellulose may have had a slightly lighter mass then the solutions containing Dynamic<sup>®</sup> 208 and it is possible that a higher quantity of adhesive was involved in the methylcellulose bonds.





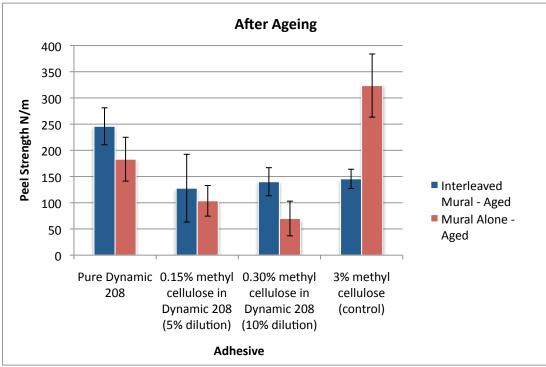


Figure 16. Peel strength (N/m) of samples after ageing.

Before ageing, the addition of methylcellulose to Dynamic<sup>®</sup> to form an adhesive of lower concentration did not have a large effect on the peel strength of either the "mural alone" or the "interleaved mural" samples. As seen in *Figures 15 and 16*, all the bars are within 30 N/m of each other. However, after ageing, there is much greater variation in peel strength; exceeding 100 N/m. Thus, the concentration of Dynamic<sup>®</sup> 208 may not have a marked initial effect on the bond strength but over time an adhesive with a lower concentration will exhibit much lower bond strength then the pure adhesive. This is not a fully consistent trend with the aged "interleaved mural" samples but, the aged "mural alone" samples show that as the concentration lowered the bond strength lowered respectively, though not within the same proportions. If we ignore the methylcellulose controls the results show that ageing had a more negative effect on the bond strength of the samples without interleaving; which all lost more strength after the aging process then their respective interleaved samples. As mentioned above, there was some outlying data. After ageing the lowest concentration of Dynamic<sup>®</sup> 208 without interleaving had a peel strength that was significantly low, below 100 N/m, which is often considered too weak for a lining adhesive. Even more curious is that pure Dynamic<sup>®</sup> adhesive with interleaving increased in peel strength after ageing. This result is likely due to the effects of the interleaving on the samples bond strength.

#### 5.2.2 Effect of interleaving and adhesive concentration on mechanical properties

Overall, it was found that interleaving had a greater effect on the bond strengths

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after ageing, maintaining more strength with all adhesive concentrations then the samples without interleaving. There are a number of factors that could be influencing these results. Porosity of the fabrics may have played a role. The linen on its own was very porous and as the adhesive dried out during accelerated ageing it is possible that less surface area remained bonded to the drywall. In contrast, the *Reemay* had a smoother surface that may have maintained greater surface contact and improved the bond. Secondly, the mode of fracture likely contributed. There were three types of bond failure observed, cohesive failure within the adhesive layer and interfacial failure, between the adhesive and the adherent. The interleaved samples were found to have cohesive failure at the *Reemay* (interleaf) layer 40 - 50% of the time. This type of fracture would have caused the *Reemay* fibers to be pulled and stretched before breaking, possibly increasing the perceived bond strength. This effect can be as seen in Figure 17. Additionally, unintended fracture occurred in about 20% of the interleaved samples after ageing. Some samples separated between the linen and the interleaf during peel testing while others separated within the drywall paper interface and a few samples exhibited both issues. *Figure 14*, seen in the previous section, and *Figure 18* both show separation issues observed during peel testing of the interleaved samples after ageing. These samples have the linen with the *Reemay* interleaf adhered with *Beva film* and then the *Reemay* interleaf is adhered to the drywall with the Dynamic<sup>®</sup> 208 adhesive solution. The linen and *Reemay* should peel away from the drywall in one piece but, in these cases, the *Reemay* interleaf was peeled from the solid substrate but the linen was no longer attached to the Reemay. Another form of unintended fracture occurred when the top paper layer of the drywall fractured and was

peeled back with the Reemay interleaf, as seen in *Figure 18*. The samples that peeled in this way may not show peel strength values that are solely based on the strength of the adhesive bond in question (Dynamic<sup>®</sup> 208) and thus effects the overall perceived peel strength of the interleaved samples.



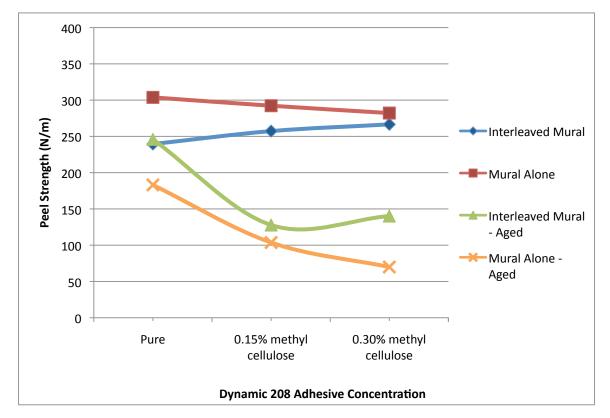
Figure 17. Example of cohesive bond failure at the interleaf, showing stretching Reemay fibers.



Figure 18. Example of separation issues observed during peel testing.

If the methylcellulose control samples are exempt, and the peel strength of each concentration of Dynamic<sup>®</sup> 208 is connected with a line, some trends are apparent. The samples without interleaving, before ageing and after ageing, show that as the concentration of the adhesive is lowered, the peel strength also lowers, but the interleaved samples, before ageing and after ageing, do not follow this trend. This is exemplified in

*Figure 19*. Generally, this implies that the addition of methylcellulose to Dynamic<sup>®</sup> 208 can lower the adhesive's bond strength; especially after accelerated ageing where the decreasing trend was more extreme. More importantly, the inconsistency in the interleaved samples correlation to concentration variations further implies that the interleaving played a more prominent role in the bond strength then the concentration of the adhesive itself.



*Figure 19. Effect of concentration on peel strength (N/m)* 

#### 5.2.3 Textile control samples

The average ultimate tensile strength (UTS) of the three types of control samples tested is summarized in *Table 6*. In all control sample sets the UTS decreased, though not by much, after accelerated ageing as expected. It is interesting to note that the addition of *Reemay* interleaf appears to have slightly weakened the tensile strength of the linen. The interleaved samples had lower UTS values then the linen alone and perhaps the process of attaching the interleaf weakened the linen. The tensile strength curves, seen in Appendix II (*Figures xiv, xv and xvi*), show that the basic properties of each material remained similar before and after ageing. The primed linen ("mural alone") control samples exhibited the curve of a strong material that underwent slight plastic deformation before breaking. The *Reemay* (interleaf) control samples were more ductile, showing a weak material with extended plasticity (necking) as seen in *Figure 20*. The control samples of primed linen interleaved with *Reemay* ("interleaved mural") exhibited slightly different curves before and after ageing; after ageing the material appears to be less strong then before ageing but has extended plasticity.

Sample	Ultimate Tensile Strength (N/mm <sup>2</sup> ) Before Ageing	±SD	Ultimate Tensile Strength (N/mm <sup>2</sup> ) After Ageing	±SD
Primed Linen: "Mural Alone" Control	37.8	2.4	37.8	2.5
Primed Linen Interleaved with <i>Reemay</i> : "Interleaved Mural" Control	35.2	2.0	32.5	1.7
Reemay: Interleaf Control	4.3	0.5	4.0	0.7

Table 6. Summary of ultimate tensile strength of the textile control samples

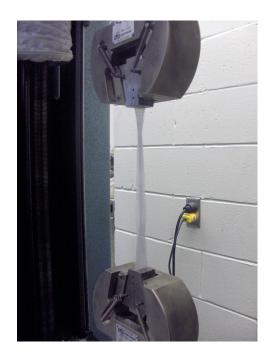
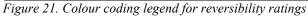


Figure 20. Necking and extended plasticity of the Reemay interleaf control samples during tensile testing.

## 5.3 Qualitative Reversibility Testing

The results of the qualitative reversibility tests were summarized in *Table 7*. Following the experimental method used by Down (2009), the average reversibility rating (%) results were colour coded, as seen in *Figure 21*; which was calculated as explained in section 4.4.3<sup>66</sup>.

Poor	Caution	Fair	Good
<25%	26-49%	50-75%	>76%



Sample	Reversibility Rating (%) Before Aging	Reversibility Rating (%) After Aging	Average Reversibility Rating (%)	±SD
Pure Dynamic 208: Linen	69.4%	54.4%	61.9%	32.5%
Pure Dynamic 208: Interleaved linen	70.0%	40.0%	55.0%	30.7%
0.15% methyl cellulose in Dynamic 208 (5% dilution): Linen	50.0%	78.4%	64.2%	20.7%
0.15% methyl cellulose in Dynamic 208 (5% dilution): Interleaved linen	65.0%	48.8%	56.9%	20.5%
0.30% methyl cellulose in Dynamic 208 (10% dilution): Linen	85.0%	92.8%	88.9%	17.3%
0.30% methyl cellulose in Dynamic 208 (10% dilution): Interleaved linen	80.0%	50.1%	65.1%	27.1%
3% methyl cellulose (control): Linen	60.0%	75.0%	67.5%	26.5%
3% methyl cellulose (control): Interleaved linen	75.0%	100.0%	87.5%	21.2%

Table 7. Overall Variation in Reversibility (%)

### 5.3.1 Effect of accelerated ageing and interleaving on reversibility

All the sample groups showed good (>76%) to fair (50-75%) average reversibility ratings implying that even after accelerated ageing the bonds remained mechanically reversible. Based on the brief study done at CCQ, it was expected that the pure Dynamic<sup>®</sup> 208 samples would rate low in reversibility. These samples do rate lowest overall but they remained fairly reversible mechanically. As with the peel test samples, the qualitative reversibility samples after ageing, showed various signs of moisture and heat damage as well as discoloration of the adhesive layer and the primed linen. Fortunately, none were so damaged that they could not be included in testing. As stated in section 4.4.3 of this

report, any sample that exhibited visible staining on the primed surface of the linen after aging lost 1-3 % points from their reversibility rating depending on the degree of the staining. Over 50% of the aged samples lost at least 1 % point due to staining. Additionally, the sample groups with 3% aqueous methylcellulose as the adhesive showed a darkening of the linen canvas before and after ageing. Since the darkening was not evident on the upper "paint layer" of the sample, no points were deducted from the reversibility ratings.

Accelerated ageing affected the interface at which the bond separated most easily. The testers all reported that the aged samples often broke at the interleaf cohesively, with much of the interleaf fibers left strongly adhered to the drywall surface; the methylcellulose control samples were an exception. Even though all samples without interleaving had slightly higher reversibility ratings, all test participants reported that interleaving facilitated safer bond separation. They found there were more canvas deformation and more damage to the "architectural substrate" or drywall when the samples without interleaving were mechanically separated.

# **Chapter 6 – Conclusions**

Overall, it was concluded that Dynamic<sup>®</sup> 208 remains mechanically reversible to a fair degree, before and after ageing, whether with or without an interleaving layer. Unfortunately, Dynamic<sup>®</sup> 208 discoloured significantly after ageing and this is a major concern. This could potentially cause color changes in treated murals, which is not acceptable. Without further testing, using Dynamic<sup>®</sup> 208 as a long-term adhesive for conservation treatments is not advised. Furthermore, results indicate that the practice of interleaving is beneficial, providing higher bond strength and maintenance of safe mechanical reversibility after ageing. As a side, if preparing mixtures of Dynamic<sup>®</sup> 208 containing methylcellulose, it is suggested that mixing is done as close to the time of use as possible. If adhesive dilutions must be made in advance they should be stored in the refrigerator to avoid any mold growth.

Further research is warranted since little testing has been done in this area and there is a need to develop appropriate testing methods and standards. Clip peel testing and 90<sup>0</sup> peel testing could also be useful techniques for assessing the bond strength of wallpaper adhesives<sup>67</sup>. Qualitative reversibility samples that do not have a detached tail end but instead are fully adhered to the drywall surface may be a more realistic representation of the difficulties involved with mechanical removal of marouflaged murals. There is also a need to find adhesives with appropriate working properties for use reattaching marouflaged murals that remain stable and reversible over time. Possible causes for the discolouration seen after ageing in the Dynamic<sup>®</sup> 208 adhesive could be

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further investigated. Identification of fillers or additives, research into the ageing characteristics of sodium nitrate, and pH testing of the samples could assist in understanding the processes that may be occurring in the material causing discolouration. A study comparing multiple commercial products and homemade wallpaper paste recipes is recommended. Finally, since no definite conclusions were reached about the effects of adhesive concentration variation on wallpaper adhesive bonds, further investigation comparing a larger number of adhesive concentrations is recommended.

# Chapter 7 – APPENDIX I

Pee	l Test - Trial run (Mock tests)	Interleaved Mural	Mural Alone	Interleaf Alone
Mock Test Un-aged Force dried	Pure Dynamic <sup>®</sup> 208 (bonded)	3	3	
	3% methylcellulose control (bonded)	3		
70°C 27 hrs	Canvas control (Fabric only)	3	3	
	Reemay control (Fabric only)			6
	Total		57	

Table i. Summary of samples prepared for mock tensile peel testing.

Qualitative Reversibility Test		Comments	#	# Mural Alone			Comments #		Interleaved Mural						
				VE	Е	D	VD	Ι			VE	Е	D	VD	Ι
Un-aged (set 1)	Pure Dynamic <sup>®</sup> 208		5							1					
	3 % methyl cellulose (control)		6							2					
	0.15% methylcellulose in Dynamic <sup>®</sup> 208		7							3					
	0.30% methylcellulose in Dynamic <sup>®</sup> 208		8							4					
Aged	Pure Dynamic <sup>®</sup> 208		5							1					
(set 2) 80°C, 65% RH	3% methyl cellulose (control)		6							2					
	0.15% methylcellulose in Dynamic <sup>®</sup> 208		7							3					
	0.30% methylcellulose in Dynamic <sup>®</sup> 208		8							4					

 Table ii. Data collection form for Qualitative Reversibility Testing

Group #	Sample	Peel Strength (N/m) Before Ageing	±SD	Peel Strength (N/m) After Ageing	±SD
1	Pure Dynamic <sup>®</sup> 208: Linen	303.76*	42.37	183.08	41.84
5	Pure Dynamic <sup>®</sup> 208: Interleaved linen	239.45	66.50	245.86	35.27
2	0.15% methylcellulose in Dynamic <sup>®</sup> 208: Linen	292.27	42.68	103.70	29.25
6	0.15% methylcellulose in Dynamic <sup>®</sup> 208: Interleaved linen	257.29	27.06	127.79	64.57
3	0.30% methylcellulose in Dynamic <sup>®</sup> 208: Linen	282.15	36.51	69.96	33.09
7	0.30% methylcellulose in Dynamic <sup>®</sup> 208: Interleaved linen	266.56	62.44	140.15	26.78
4	3% methylcellulose (control): Linen	128.57	23.84	323.54	60.26
8	3% methylcellulose (control): Interleaved linen	107.64	14.26	145.63	18.31

Table iii. Overall Variation in Peel Strength (N/m).

\*Only four samples instead of five

*Table iv. Condition of qualitative reversibility and peel tensile test samples after ageing.* 

Sample #	G = ready for testing B = too damaged	Condition of the Canvas	Condition of the Adhesive	Condition of the Drywall
A1DYI = Aged Pu	ire Dynamic <sup>®</sup>	208 with interleaf (Qualitative Reven	rsibility Sample S	et)
A1DYI1	G	Staining	Discolored	
A1DYI2	G	Staining	Discolored	
A1DYI3	G	Staining	Discolored	
A1DYI4	G	Staining	Discolored	
A1DYI5	G	Staining	Discolored	
A1DYI6	G	Staining	Discolored	
A1DYI7	G	Staining and drips	Discolored	
A2DYNI = Aged	Pure Dynami	ic <sup>®</sup> 208 w/o interleaf (Qualitative Rev	ersibility Sample	Set)
A2DYNI1	G		Discolored	
A2DYNI2	G		Discolored	
A2DYNI3	G		Discolored	

A2DYNI4	G	Slightly convex	Discolored	
A2DYNI5	G	More convex, staining and drips	Discolored	Discoloured in upper section
A2DYNI6	G	More convex, staining and drips	Discolored	Section
A2DYNI7	G		Discolored	
A3DYI = Aged I	Pure Dynamic	<sup>®</sup> 208 with interleaf (Peel Tensile Test	ting Set)	
A3DYI1	G	Interleaf detached near end of drywall, badly stained, drip marks		
A3DYI2	G	Interleaf detached near end of drywall, stained	Discoloured	
A3DYI3	G	Interleaf detached near end of drywall, top edge and along top right and left edge of adhered area, badly stained, drip marks	Discoloured	
A3DYI4	В	Interleaf detached near end of drywall, badly on top edge and along left and right edge of adhered area, badly stained, drip marks	Discoloured	Paper lifted from top 3 <sup>1</sup> / <sub>4</sub> <sup>2</sup> , and plaster dissolved along right side 1 <sup>3</sup> / <sub>4</sub> <sup>2</sup> .
A3DYI5	B, will peel anyway	Interleaf detached near end of drywall, badly on top edge and along top left edge of adhered area, badly stained, drip marks	Discoloured	Paper lifted from top 2
A3DYI6	G	Interleaf detached near end of drywall, top edge and along top right edge of adhered area, badly stained, drip marks	Discoloured	
A3DYI7	G	Interleaf detached near end of drywall	Discoloured	
	Pure Dynami	c <sup>®</sup> 208 w/o interleaf (Peel Tensile Test		1
A4DYNI1	В	Badly stained	Discoloured	Paper lifted from top 2 $\frac{3}{4}$ "
A4DYNI2	B, will peel anyway	Badly stained	Discoloured	Paper lifted from top 2 $\frac{1}{4}$
A4DYNI3	G		Discoloured	
A4DYNI4	G		Discoloured	
A4DYNI5	G		Discoloured	
A4DYNI6	G		Discoloured	
A4DYNI7	G		Discoloured	
0		llulose with interleaf (Qualitative Rev	ersibility Sample	e Set)
A5MCI1	G	Minor staining		
A5MCI2	G	Minor staining		
A5MCI3	G	Minor staining		
A5MCI4	G	Minor staining		
A5MCI5	G	Minor staining		
A5MCI6	G	Minor staining, crinkling of interleaf/canvas		
A5MCI7	G	Minor staining, crinkling of interleaf/canvas		
A6MCNI = Aged	3% Methylce	ellulose w/o interleaf (Qualitative Reve	ersibility Sample	Set)

A CMCNT1	C	Distantion of an all 1		
A6MCNI1	G	Distortion of un-adhered area		
A6MCNI2	G			
A6MCNI3	G			
A6MCNI4	G			
A6MCNI5	G			
A6MCNI6	G			
A6MCNI7	G			
A7MCI = Aged 3%	% Methylcell	ulose with interleaf (Peel Tensile Test	ing Set)	
A7MCI1	G	Interleaf detached near end of drywall, stained, drip marks but near middle instead of top like others		Plaster discoloured brownish in middle- bottom area
A7MCI2	G	Interleaf detached near end of drywall, stained		
A7MCI3	G	Interleaf detached near end of drywall, stained		
A7MCI4	B worst	Interleaf detached near end of drywall, badly on top edge and along left and right edge of adhered area, badly stained, drip marks		Paper lifted from top 3 3/4" and bottom 1 1/2", plaster dissolved along top 1 1/2", bad discolouration bottom 1-2".
A7MCI5	G	Interleaf detached near end of drywall, badly stained		Plaster discoloured brownish on top edge
A7MCI6	G	Interleaf detached near end of drywall, top edge of adhered area, stained		
A7MCI7	G	Interleaf detached near end of drywall, top edge of adhered area, badly stained, drip marks		
A8MCNI = Aged 3	% Methylcel	lulose w/o interleaf (Peel Tensile Test	ing Set)	
A8MCNI1	В	Badly stained, drip marks	8 /	Paper lifted from top 3"
A8MCNI2	B, will peel anyway	Stained, drip marks		Paper lifted from top 1 1/8"
A8MCNI3	G			
A8MCNI4	G			
A8MCNI5	G			
A8MCNI6	G			
A8MCNI7	B, will pull anyway	Slice in canvas		
		ethylcellulose in Dynamic <sup>®</sup> 208 with in	nterleaf	
(Qualitative Rever A95DYMCI1	B	Highly stained and drips, distortion and interleaf detached top and bottom	Discoloured	Highly discoloured in top area
A95DYMCI2	G	Staining and drips, interleaf detached at bottom	Discoloured	
A95DYMCI3	G	Staining and drips, interleaf detached at bottom	Discoloured	

A95DYMCI4	В	Stained and drips, distortion and	Discoloured	Highly dissoloured in
A95D Y MIC14	в	interleaf detached top and bottom	Discoloured	Highly discoloured in top and bottom area,
A95DYMCI5	В	Stained and dring distartion and	Discoloured	paper detached in top Highly discoloured in
A95DYMC15	D	Stained and drips, distortion and interleaf detached top and bottom	Discoloured	top and bottom area, paper detached in top
A95DYMCI6	G	Staining and drips	Discoloured	
A95DYMCI7	G	Staining and drips, interleaf detached at bottom	Discoloured	
A105DYMCNI = (Qualitative Reven		Methylcellulose in Dynamic <sup>®</sup> 208 w/o ple Set)	interleaf	
A105DYMCNI1	G	Minor staining	Discoloured	
A105DYMCNI2	G	Minor staining	Discoloured	
A105DYMCNI3	G	Minor staining and drips	Discoloured	
A105DYMCNI4	G	Staining and drips	Discoloured	
A105DYMCNI5	G	More staining and drips	Discoloured	
A105DYMCNI6	В	Highly stained and drips and distortion of canvas	Discoloured	Highly discoloured in top area, paper detached in top
A105DYMCNI7	G	Minor staining	Discoloured	
A115DYMCI = A	ged 0.15% N	1ethylcellulose in Dynamic <sup>®</sup> 208 with <b>1</b>	interleaf (Peel T	ensile Testing Set)
A115DYMCI1	В	Slice in canvas, interleaf detached near end of drywall	Discoloured	
A115DYMCI2	G	Interleaf detached near end of drywall, top edge and along top right edge of adhered area	Discoloured	
A115DYMCI3	G	Interleaf detached near end of drywall, top edge and detachment from adhesive for 1.5 cm	Discoloured	
A115DYMCI4	G	Interleaf detached near end of drywall, top edge and detachment from adhesive for 1 cm	Discoloured	
A115DYMCI5	G	Interleaf detached near end of drywall	Discoloured	
A115DYMCI6	G	Interleaf detached near end of drywall	Discoloured	
A115DYMCI7	G	Interleaf detached near end of drywall	Discoloured	
A125DYMCNI =	Aged 0.15%	Methylcellulose in Dynamic 208 w/o i	interleaf (Peel Te	ensile Testing Set)
A125DYMCNI1	G		Discoloured, brown	
A125DYMCNI2	G		Discoloured, brown	
A125DYMCNI3	G		Discoloured, brown	
A125DYMCNI4	G		Discoloured, brown	
A125DYMCNI5	G		Discoloured, brown	
A125DYMCNI6	G		Discoloured, brown	
			010 111	

			brown	
		Methylcellulose in Dynamic <sup>®</sup> 208 with	interleaf	
(Qualitative Rever	sibility Sam		r	1
A1310DYMCI1	G	Staining and drips, interleaf detached at bottom and top	Discoloured	Minor staining in top area
A1310DYMCI2	G	Staining and drips, interleaf detached at bottom	Discoloured	
A1310DYMCI3	G	Staining and drips, interleaf detached at bottom	Discoloured	
A1310DYMCI4	В	Stained and drips, distortion and interleaf detached top and bottom	Discoloured	Highly discoloured in top area, paper detached in top
A1310DYMCI5	B?	stained and drips, distortion and interleaf detached top and bottom	discoloured	Highly discoloured in top area, paper minor detached in top
A1310DYMCI6	G	Staining and drips, interleaf detached at bottom	Discoloured	
A1310DYMCI7	G	Staining and drips, interleaf detached at bottom	Discoloured	
A1410DYMCNI = (Qualitative Rever		Methylcellulose in Dynamic <sup>®</sup> 208 w/o	o interleaf	
A1410DYMCNI1	G	Staining and drips	Discoloured	
A1410DYMCNI2	В	Highly stained and drips and distortion of canvas	Discoloured	Highly discoloured in top area, paper minor detached in top
A1410DYMCNI3	G	Highly stained and drips	Discoloured	
A1410DYMCNI4	G	Staining and drips	Discoloured	
A1410DYMCNI5	G	Minor staining	Discoloured	
A1410DYMCNI6	G	Minor staining	Discoloured	
A1410DYMCNI7	G	Minor staining	Discoloured	
A1510DYMCI = A	ged 0.30% I	Methylcellulose in Dynamic <sup>®</sup> 208 with	interleaf (Peel T	ensile Testing Set)
A1510DYMCI1	G	Interleaf detached near end of drywall	Discoloured	
A1510DYMCI2	G	Interleaf detached near end of drywall and along most of the surface	Discoloured	
A1510DYMCI3	G	Interleaf detached near end of drywall	Discoloured	
A1510DYMCI4	G	Interleaf detached near end of drywall	Discoloured	
A1510DYMCI5	G	Interleaf detached near end of drywall	Discoloured	
A1510DYMCI6	G	Interleaf detached near end of drywall	Discoloured	
A1510DYMCI7	G	Interleaf detached near end of drywall	Discoloured	
A1610DYMCNI =	Aged 0.30%	• Methylcellulose in Dynamic <sup>®</sup> 208 w/e	o interleaf (Peel '	<b>Fensile Testing Set</b> )
A1610DYMCNI1	G		Discoloured, brown	
A1610DYMCNI2	G	Canvas strip is convexly curved, possible water damage (shows more	Discoloured, brown	

A1610DYMCNI3	G	Canvas strip is convexly curved, possible water damage	Discoloured, brown
A1610DYMCNI4	G	Canvas strip is convexly curved, possible water damage	Discoloured, brown
A1610DYMCNI5	G		Discoloured, brown
A1610DYMCNI6	G		Discoloured, brown
A1610DYMCNI7	G		Discoloured, brown

Table v. Total Reversibility Rating (%) in descending order.

Group #	Sample	Total Reversibility Rating (%)	±SD
7	10% dilution (0.30% methylcellulose in Dynamic 208): Linen	88%	17%
4	3% methyl cellulose (control): Interleaved linen	88%	21%
8	3% methyl cellulose (control): Linen	68%	26%
6	5% dilution (0.15% methylcellulose in Dynamic 208): Linen	65%	21%
3	10% dilution (0.30% methylcellulose in Dynamic 208): Interleaved linen	65%	29%
5	Pure Dynamic 208: Linen	63%	32%
2	5% dilution (0.15% methylcellulose in Dynamic 208): Interleaved linen	58%	21%
1	Pure Dynamic 208: Interleaved Linen	55%	31%

# Chapter 8 – APPENDIX II



Figure i. Canvas mural by John Hamound in the Black house at Mount Allison University, New Brunswick, Canada (Karpowicz, Adam and Jane Tisdale, 2009)

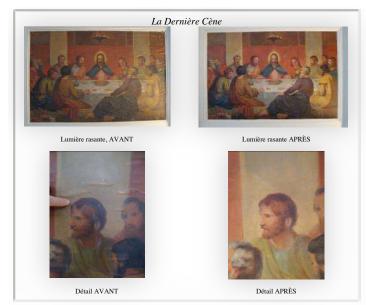


Figure ii. Charles Huot (1855-1930), <u>L'Adoration des mages</u>, <u>L'Adoration des bergers</u>, <u>Les noces de Cana</u> <u>et La dernière Cène</u>, 1892 (O'Malley, 2009).



Figure iii. The Nicolet Avatar 320 FTIR Spectrometer with ATR Golden Gate attachment.

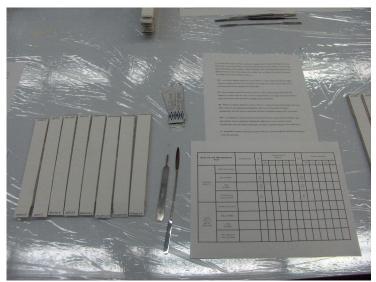
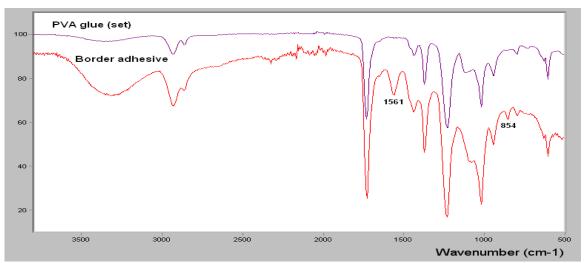


Figure iv. Qualitative reversibility testing set-up showing 8 samples, scalpel, microspatula, hard copy of rating definitions and rating collection form (from left to right).



Figure v. Conservation students partaking in qualitative reversibility testing.



*Figure vi. Infrared spectra of Border adhesive R-070007 (dried) and PVA glue (set)* **Comments:** 

- The border adhesive is clearly PVA glue.
- The weak absorptions at 1561 and 854 cm<sup>-1</sup> indicate the presence of a minor amount of another unidentified ingredient.

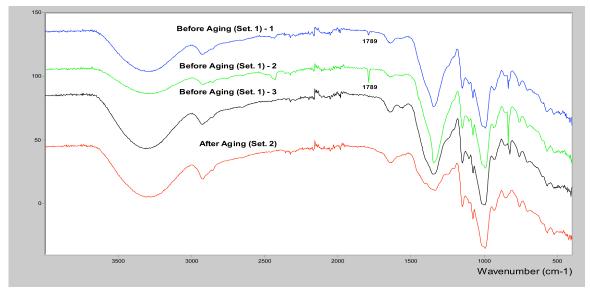
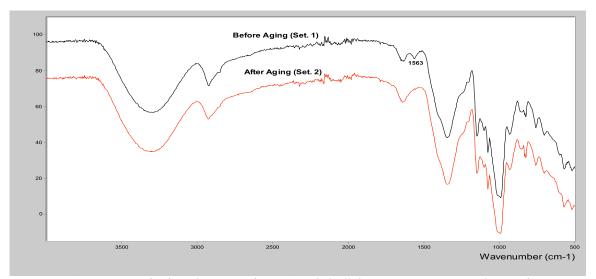


Figure vii. Comparison of Infrared spectra of 0.15% methylcellulose in pure aqueous solution of Dynamic 208 (5% dilution), before and after ageing showing little to no difference.



*Figure viii.* Comparison of Infrared spectra of 0.30% methylcellulose in pure aqueous solution of Dynamic 208 (10% dilution), before and after ageing showing little to no difference.

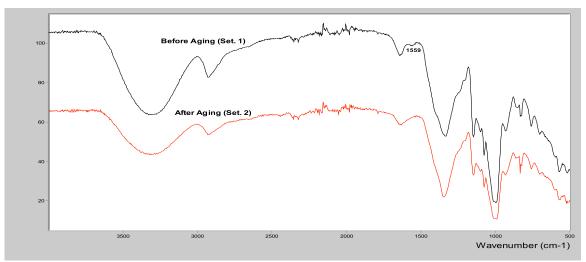
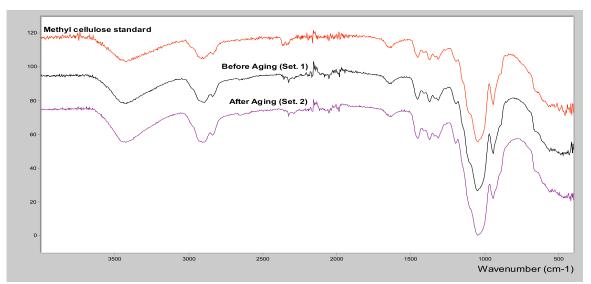


Figure ix. Comparison of Infrared spectra of 0.90% methylcellulose in pure aqueous solution of Dynamic 208 (30% dilution), before and after ageing showing little to no difference



*Figure x. Comparison of Infrared spectra of 3% methylcellulose (control), before and after ageing showing little to no difference* 

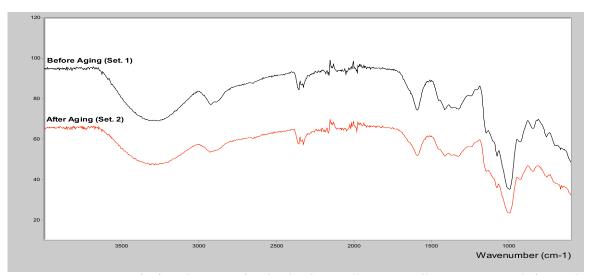
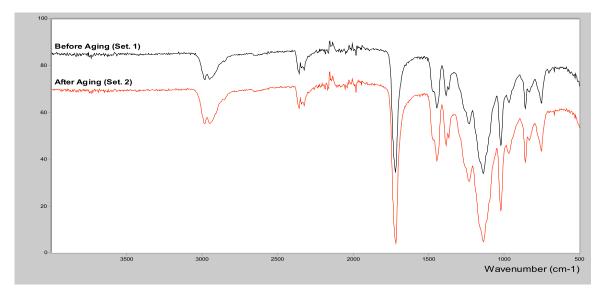


Figure xi. Comparison of Infrared spectra of Richards Clear Wallcovering Adhesive R-070007, before and after ageing showing little to no difference



*Figure xii.* Comparison of Infrared spectra of Paraloid -72, before and after ageing showing little to no difference

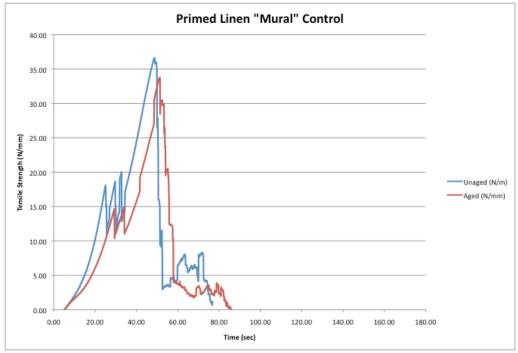


Figure xiv. Tensile strength curves for "mural alone" textile control strips

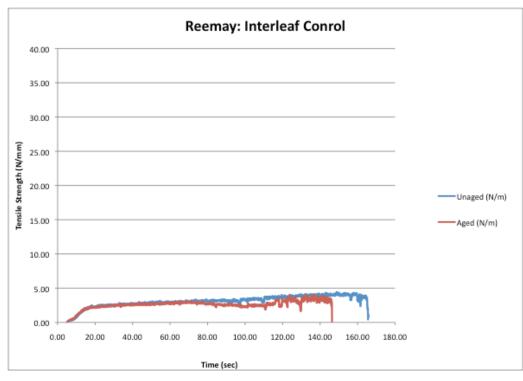


Figure xv. Tensile strength curves for "interleaf" textile control strips

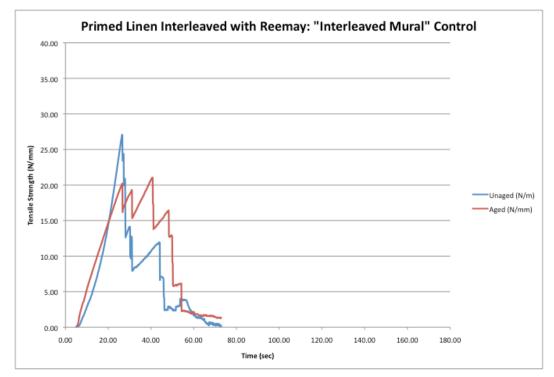


Figure xvi. Tensile strength curves for "interleaved mural" textile control strips

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<sup>26</sup> Muralo wallpaper adhesives: wheat starch, dextrin, methylcellulose, and fungicide. <a href="http://cool.conservation-us.org/coolaic/sg/bpg/annual/v07/bp07-02.html">http://cool.conservation-us.org/coolaic/sg/bpg/annual/v07/bp07-02.html</a>. Accessed October 2009.

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<sup>28</sup> Shur-Stick Wall Size: proprietary methylcellulose in distilled water, Gibson-Homans, Twinsburg, OH.
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<sup>33</sup> Dynamic 208 diluted by 5%, measured w/w 5g of 3% methylcellulose aqueous solution in 95.5g of pure aqueous solution of Dynamic 208 wallpaper adhesive.

<sup>34</sup> Dynamic 208 diluted by 10%, measured w/w 10.4g of 3% methylcellulose aqueous solution in 89.9g of pure aqueous solution of Dynamic 208 wallpaper adhesive.

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<sup>38</sup> Comyn, John, *Adhesive Science* (Cambridge: The Royal Society of Chemistry, 1997), p. 125.

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<sup>40</sup> Dynamic 208 diluted by 5%, measured w/w 5g of 3% methylcellulose aqueous solution in 95.5g of pure aqueous solution of Dynamic 208 wallpaper adhesive.

<sup>41</sup> Dynamic 208 diluted by 10%, measured w/w 10.4g of 3% methylcellulose aqueous solution in 89.9g of pure aqueous solution of Dynamic 208 wallpaper adhesive.

<sup>42</sup> 'Standard Test Method for Peel or Stripping Strength of Adhesive Bonds', D 903-98,' *Annual Book of ASTM Standards (2008) web version*. Accessed October 2009.

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<sup>54</sup> Ibid.

<sup>55</sup> Shurvell, H. F. (Gus) (Emeritus Professor, Department of Chemistry and Adjunct Professor, Queen's University, Kingston, ON), personal communication, November 2009.

<sup>56</sup> Dynamic 208 diluted by 10%, measured w/w 10.4g of 3% methylcellulose aqueous solution in 89.9g of pure aqueous solution of Dynamic 208 wallpaper adhesive.

<sup>57</sup> Dynamic 208 diluted by 30%, measured w/w 30.2g of 3% methylcellulose aqueous solution in 70.1g of pure aqueous solution of Dynamic 208 wallpaper adhesive.

<sup>58</sup> Shurvell, H. F. (Gus) (Emeritus Professor, Department of Chemistry and Adjunct Professor, Queen's University, Kingston, ON), personal communication, November 2009.

<sup>59</sup> Dynamic 208 diluted by 5%, measured w/w 5g of 3% methylcellulose aqueous solution in 95.5g of pure aqueous solution of Dynamic 208 wallpaper adhesive.

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<sup>62</sup> Dynamic 208 diluted by 30%, measured w/w 30.2g of 3% methylcellulose aqueous solution in 70.1g of pure aqueous solution of Dynamic 208 wallpaper adhesive.

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