Summary

Crosslinked synthetic latex
was identified as one of
the adhesives used in
adventitious repair to
oriental carpets and other
textiles. A method to
remove this latex was
developed. The experimental
treatment method uses an
aqueous solution of
ammonium acetate, then air
drying, followed by
drycleaning with 1,1,1
trichloroethane.

THE REMOVAL OF CROSSLINKED SYNTHETIC LATEX FROM CARPETS:
PRELIMINARY RESULTS

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History.

Several decades ago, collectors, dealers, artists, and oriental
rug restorers--without any review of product literature,
scientific study, or accelerated ageing data--casually treated
their oriental rugs and fiber art with water emulsible adhesives
("latex"). Today these oriental rugs, hooked rugs, and needlepoint
carpets, as well as modern fiber artworks, are beginning to appear
in textile conservation laboratories with post-production latex
backings on the verso of the object.

The latex backing was used to secure repairs to the pile of the
rug, to hold new repair knots in place, and to prevent carpets
from sliding on floors. Nineteenth century hooked rugs and
needlepoint rugs can also be found which have foundation fabrics
(substrates) smeared with latex to hold tufts of yarn or rag pile
in place. The fiber artist Sheila Hicks has used a layer of latex
to "hold in the canvas the bits of wool that were not knotted"
[15]. In this instance, the latex "saved time" as well.

A latex--a polymeric emulsion adhesive--provided a quick and
simple method to repair tears or splits; it temporarily stabilized
the weave/pile matrix; and it reduced the slipperiness of the
carpet on a hardwood floor. When the latex application was
confined to the outside perimeter of the carpet, it could be
assumed that the aim or goal of the application was to prevent
slippage on a hardwood floor. Wool felt underlayments do not
prevent small carpets from sliding; the frictional adhesion
between the felt and the floor is not nearly as great as that
between the carpet and the felt.

At least one carpet dealer/restorer is known to have treated every
small carpet entering his establishment with a latex adhesive
backing so that the small area rugs could not slip on the waxed
wood floors of his customers [7]. This basis for treatment would
not be necessary today as other types of underlayment are
available for small carpets. It is likely that this treatment
occurred ten or twenty years ago. In almost all the cases, the
treatments occurred at least ten years ago; the actual products
used were not recorded; and the presence of the polymeric compound
has been found to accelerate the deterioration of the rug.

For the textile conservator, the use of a latex backing introduces
a material unsympathetic to the natural flexibility of a fabric or
carpet. The increased stiffness in the previous portion of the
carpet will actually induce damage to the object: the carpet
foundation begins to crack. Fracture can also occur in storage
from the stress on the foundation while it is rolled, even if the
storage tube is of generous diameter. Furthermore, the absorption
of the polymeric emulsion into the foundation structure prevents
additional repairs at a later time: the foundation weave becomes
too rigid to support the manipulation that conventional repair
with a needle and thread would require. The fracture itself begins
on the verso at the substrate, so that damage is not apparent
until the carpet has been examined or until the carpet has already
developed major splits.

Examination and Analysis.

Visual Examination. Four carpets belonging to American historical
societies and museums had similar patterns of deterioration: a
Sultanabade carpet (1850-1875); a Karabagh carpet (mid 19th
century), an Iranian carpet (ca. 1875-1900); and a "Brussels"
(machine woven) carpet (ca. 1985). On all four, an adhesive
backing had been applied only to the back or verso of the object.
All four showed increased stiffness in areas of treatment with the
polymeric emulsion. Where the latex backing was evident, the
carpets had greater damage than where no backing had been applied.
The fractures to the weave structure varied from horizontal splits
across the warp to wholesale breakage to vertical splits parallel
to the warp.

The adhesive backings varied from opaque to translucent; they generally added a yellow appearance to the surfaces of the weave structures to which they had been applied. Each adhesive backing formed a cohesive mass sitting in large measure above the yarn but filling the interstices of the weave structure, as well as those spaces between the fibers on the outside surface of each yarn. The thickness of the polymeric film varied from carpet to carpet and among areas of a given carpet. It may have been applied by brushes or, as reported to the textile conservator examining the Brussels carpet, with painting rollers [7]. Because of the liquid nature of polymeric emulsions, the <u>verso</u> surface of the latexed carpet was usually fairly uniform: the polymer flowed before drying.

Most of the latex backings retained some flexibility and tenacity to the fibers and mechanical removal would have only damaged the foundation structure of the carpet. A scalpel could not have safely separated the foundation of the rug from the latex. One carpet fragment did have a more brittle latex: here the polymer could be scratched from the <u>verso</u> with an ivory bone folder, without disturbing the weave structure beneath the polymeric material.

    <u>Solubility Parameter</u>. A thermoplastic polymeric material can be characterized by the solvents which dissolve or swell it. For each solvent or resin, there are intermolecular forces (dispersive, dipole-dipole, and hydrogen bonding),and each of these factors can be given a numerical value [8, 26]. In order to establish this "solubility parameter" for various latex backings, different solvents were tested on a latexed (Iranian) carpet fragment: acetone (47/32/21), ethanol (36/18/46), xylene (83/5/12), toluene (80/7/13), and methyl ethyl ketone (53/26/21). The last, methyl ethyl ketone, performed the best. The polymeric material was softened and slowly dissolved. However, the experimental submersion of two fragments of the Iranian carpet in MEK for 15 hours yielded carpet fragments that retained enough polymeric material to remain stiff, despite the high liquor ratio involved. The spent liquor was decanted and allowed to evaporate and the residue was retained for future tests.

Tests were also conducted on the two types of latex adhesive found on the <u>verso</u> of the "Brussels" carpet. The latices will be termed Adhesive #1 for the older latex and Adhesive #2 for the second and complete backing. Since the backing on the first fragment had responded best to the higher dispersion forces, fairly high dipolar contributions, and lower hydrogen bonding found in the solubility parameter of methyl ethyl ketone, this and similar solvents were used on Adhesive #1. Acetone (47/32/31), cyclohexanone (55/28/17), cyclopentanone (-), diethyl ketone (-), methyl ethyl ketone (53/26/21), methyl propyl ketone (-),and methyl cyclohexanone (-). These ketones loosened the latex enough to soil swabs and blotters with residue. In addition, xylene (83/5/12) and toluene (80/7/13) were tested. While some residue was removed, these solvents were less effective [7].

The process of testing was repeated for Adhesive #2. The results were equivalent to those of the first fragment but a comparison to solubility parameters of known resins suggest what the latex backing is not, more than what it is (see Figure 1). The latex backings examined are not polyamide ("soluble nylon") once used in textile conservation; they are probably not related to any nitrocellulose coating; it is unlikely they are a pure poly(vinyl chloride). They fall outside the range of "short oil" alkyd resin. They do fit the range of solubility of poly(methyl methacrylates) [26].

Conventional conservation cleaning methods that depend on dissolving the material to be removed are not suitable for the removal of latex backings. Initial work on the solubility parameter of two extant latex repairs made this limitation quite apparent. Whether or not the polymeric material was crosslinked or oxidized, solvent systems that began to remove any fraction of the polymer did so with bad results. The polymeric material that was removed was readily absorbed into the fiber substrate of the adjacent warp and weft. The stiff object actually becomes more rigid with the removal of the latex from the verso surface. Solvents applied to non-porous surfaces can remain on the outside of the object but wool, cotton, or jute--even if the fibers are spun and plied--retain an absorbancy that prevents simple removal with solvents by swabs or blotters. Thus, the conventional methods used to remove aged painting varnishes cannot be transferred to this particular polymeric removal problem.
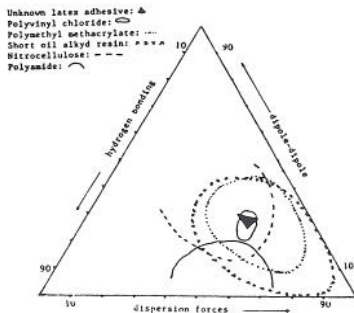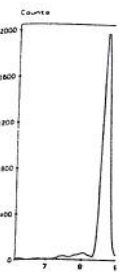

Figure 1. Solubility Perameters.

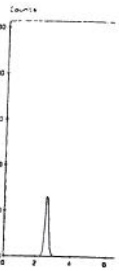Figure 2. spectrum adhesive Iranian c

Figure 3. spectrum c adhesive r tertiary b of "Brusse

Figure 4. spectrum of

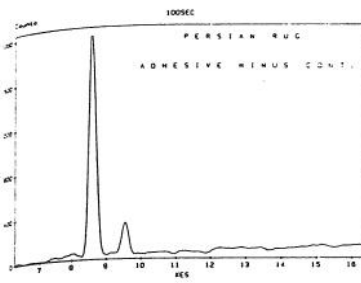Figure 5. In of unknown a from Iranian

Figure 2. X-ray fluorescent spectrum of unknown adhesive removed from Iranian carpet.
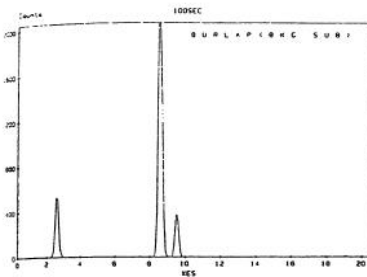

Figure 3. X-ray fluorescent spectrum of unknown adhesive removed from tertiary backing (repair) of "Brussels" carpet.
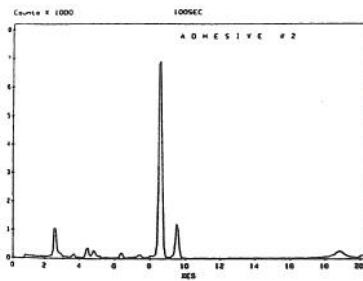

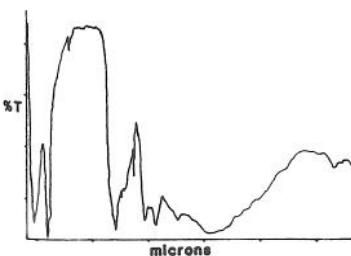Figure 4. X-ray fluorescent spectrum of Adhesive #2.

Figure 5. Infrared spectrum of unknown adhesive residue from Iranian carpet.

X-Ray Fluorescence. While some polymers will crosslink without inorganic agents, the detection of typical inorganic additives for latex could indicate the presence of an inorganic crosslinking agent, and suggest the type of polymer in question. If the latex adhesive were a "true" latex, of natural rubber, few inorganic ions would be present. If the latex were entirely synthetic, the inorganic materials found might indicate which polymer is present.

Three sets of polymeric samples were taken to the Analytical Laboratory in the Conservation Department of the National Gallery. There, a Kevex energy dispersive X-Ray Fluorescence unit is set up with barium and rhodium targets; the collimator source is 6mm; the detectors vary from 2 to 6 mm. In all tests, an accumulation time of 100 seconds was deemed sufficient.

One carpet fragment was analysed both in an undisturbed area and in a latex coated area. When the two results were compared, and the inorganic background of the carpet subtracted from the residue/carpet fluorescence, the result showed no trace of inorganic elements on the specimen, a result which indicates that the polymer has an entirely organic chemical composition. Next, the latex adhesive residue from the methyl ethyl ketone immersion test of the Iranian carpet fragment was tested. The spent liquor had been allowed to dry and had been held on an aluminum foil surface. Again, the foil was analysed and subtracted from the final result. The residue showed a substantial amount of zinc (Figure 2). Although the results of X-ray fluorescence on thin polymeric films cannot be calculated in a quantitative manner, the strength of the peaks do suggest the presence of enough zinc to accelerate the polymerization of isoprene or to crosslink a poly(acrylic acid) [1].

A piece of burlap which had been used as a tertiary backing on the Brussels carpet was assayed. After subtraction for the impurities within the burlap itself, the adhesive residue on its edge also revealed a large amount of zinc, together with an indeterminate amount of chlorine (Figure 3). In fact, the quanity of zinc required resetting the instrument to incorporate the top of the peak. The chlorine is "attenuated" (reduced in intensity) because its X-rays are absorbed by the air, and the amount of chlorine cannot be determined [1]. The presence of the chlorine could indicate a polyvinyl chloride copolymer in the latex.

X-ray fluorescence analysis was also carried out on a small fragment from the Brussels carpet. Once again, significant amounts of zinc and chlorine were detected. In this instance, small amounts of calcium and iron are present also. Finally, a small sample of burlap impregnated with Adhesive #2 was tested. This sample could not be run against a control, since no area of the burlap was unaffected by the latex adhesive. Nevertheless, only zinc and chlorine were present, with a small amount of iron (Figure 4). Thus, the results of the analysis of the Iranian carpet, the adhesive on the tertiary backing of the Brussels carpet, its Adhesive #1 and its Adhesive #2 all appear to have some of the same inorganic components.

Wet Chemical Testing. Sulfur's position is detectable by XRF, but its absence was specifically confirmed by wet chemical testing. A reducing solution was made containing 10 gr of stannous chloride and 10 ml of concentrated hydrochloric acid and added to 55 ml of deionized water. Lead acetate paper was prepared using a supersaturated solution of lead acetate in deionized water. Samples were taken of the adhesive/burlap tertiary backing, a burlap backing blank (no adhesive), the Adhesive #1 on a thread, a blank control thread, and the impregnated Adhesive #2/burlap. Each sample was individually placed in a small petri dish and slowly heated. Each petri dish was covered with a lead acetate paper. No brownish black stain on the paper occurred to indicate sulfur [13].

Infrared Spectroscopy is a standard method of resin analysis. There are, however, problems associated with interpreting the spectra of samples of degraded polymeric mixtures, and with distortion of spectra due to additives. Nevertheless, it was hoped that a comparison with the spectra of known polymers would eliminate some compounds from consideration.

A Mattson Alpha Centauri Fourier transform infrared spectrophotometer was employed. Some of the samples were prepared by simple grinding to a powder that was then spread out on a
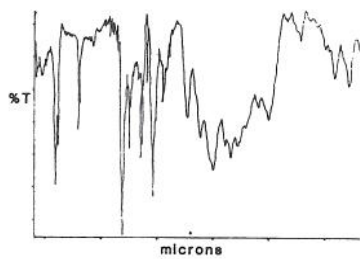
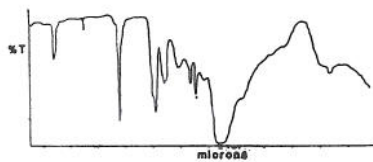Figure 6. Infrared spectrum of Adhesive #2 from "Brussels" carpet.

Figure 7. Infrared spectrum of the tetrapolymer vinylidene chloride/acrylonitrile/methyl acrylate/itaconic acid (91:4.5:4:0.5).
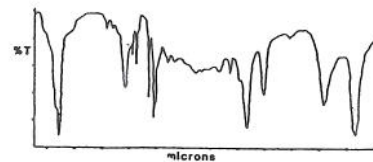

Figure 8. Infrared spectrum of the terpolymer of butadiene/styrene/carboxylic acid (50:45:5)
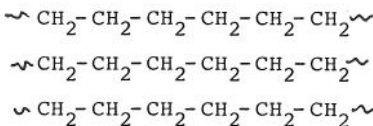

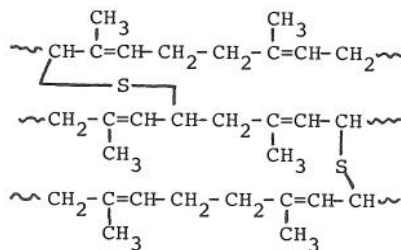Figure 9. The structure of poly(ethylene), a linear polymer.

Figure 10. The structure of vulcanized rubber (poly (cis-1,4-isoprene), a crosslinked polymer.

potassium bromide film; others were dispersed in potassium bromide pellets. Adhesive #2 required dispersion with methylene chloride. A control sample of pure potassium bromide was made as well. The spectra of Adhesives #1 and #2 are closer in appearance to each other than to that of the MEK/latex residue, although the spectra do share some common characteristics (see Figures 5 and 6). All of the samples show some absorbance at 1647-1653 wavelengths; at 1457 or 1458; and at 1375-1386. The common reading at 2360 is that of carbon dioxide. While the Iranian carpet polymer and Adhesive #1 share a possible carboxyl salt peak at 1540, the Iranian carpet polymer and Adhesive #2 share a possible aromatic alcohol moiety with similar peaks at both 3425-3428 and 1239-1243. The two adhesives share three peaks at 1111, 1033, and 908-910, which suggest aliphatic ethers, primary alcohol groups, or ionic carbonates [17, 19].

Parallels can be drawn between the spectrum of the vinylidene chloride/acrylonitrile/methyl acrylate/itaconic acid tetrapolymer (91:4.5:4:0.5, Figure 7) and those of Adhesives #1 and #2 more easily than to the spectra of Neoprene, cis-1,4-polyisoprene, styrene/ butadiene (62:38) or a terpolymer of butadiene/ styrene/carboxylic acid (50:45:5, Figure 8) [20, 21, 24].

Theory.

Polymers are divided into two fundamental groups, linear and non-linear, i.e. crosslinked, polymers (see Figures 9 and 10). The emphasis in conservation materials research has been to develop the use of linear, thermoplastic, soluble polymers and to proscribe the use of crosslinking, thermosetting materials for all but special cases [8]. Polymers of both types can be produced by several methods: bulk polymerization (with only reagents and products), solution polymerization, suspension polymerization, and emulsion polymerization. Latex traditionally described the emulsion product of natural rubber, poly(cis-isoprene) dispersed in an aqueous medium. The final, dried product was crosslinked into a three-dimensional network, sometimes by means of sulfur (see Figure 10). Today latex is a generic term for the milky result of an emulsion polymerization where 40-80% solids (polymer) are dispersed, rather than dissolved, in a liquid (usually water) [3, 4, 25].

Tufted carpet technology uses such latices as adhesives to secure unknotted pile yarns to a primary, woven substrate. Styrene-butadiene rubber copolymers called "SBR's" have replaced natural rubber as the latex adhesive selected. Although butadiene can be crosslinked across its remaining unstaturated bond, a simple, self- curing system has been developed by adding a third carboxylated component polymer. It is this XSBR that is important to the study. If the carboxylic acid groups on each chain react with a divalent metal salt, bridges between XSBR polymer chains are formed. The result is a crosslinked, stable, solvent resistant polymer. Technically, these latices are ionomers; that is, they are polymers with ionic salt bridges which can be _reversibly_ crosslinked (see Figure 11) [9, 10, 12, 22].

The analysis of the polymeric material suggested a new treatment to remove the latex backing. Solubility parameter testing indicated that the material could be partially, but not entirely, dissolved. X-ray fluorescence showed that the divalent metal crosslinking agent, zinc, was present. Infrared spectra demonstrated that natural rubber latex (polyisoprene) was not present and sulfur was not found during the wet chemical testing.

Instead of wholesale dissolution by a single solvent or group of solvents, a sequential process seemed appropriate. First, the crosslink in the polymer is broken, the crosslinking agent is flushed out, and the uncrosslinked polymeric residue is rinsed out and dried. A second solvent system is then introduced to separate the remaining polymeric material from the object. This second solvent is not chosen on the basis of being a "good" solvent, because the goal is not to dissolve the polymer but only to loosen it from the object. A "good" solvent would dissolve the polymer and allow it to migrate into the fibers of the textile. Therefore a "poor" solvent is used, one that would be on the edge of the solubility perameter so that absorption into the fibers could not take place [27]. At this point, the polymeric material could be treated like any other foreign solid soil particle and abstracted by a combination of gentle agitation and flushing.
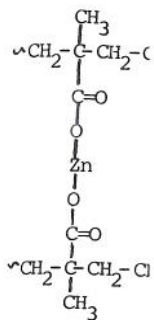

Figure 11. an ionomer, of ethylene acid, after

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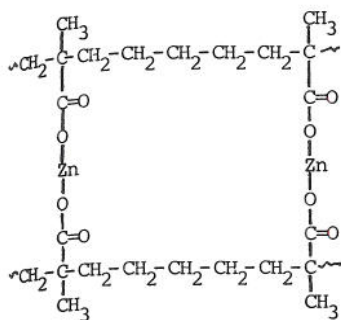could not
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Figure 11. The structure of an ionomer, the copolymer of ethylene and methacrylic acid, after crosslinking.
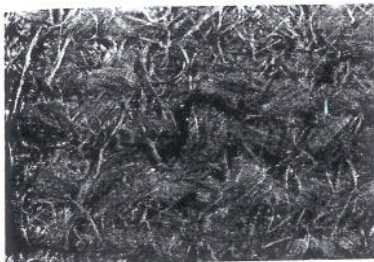

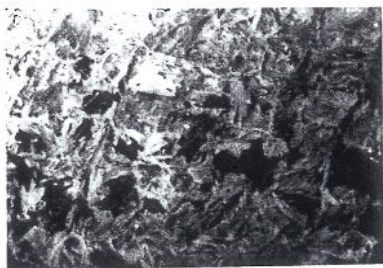Figure 12. The latex coating on Sample #1 before treatment, magnified.

Figure 13. The latex coating on Sample #1 after two hours submersion in 10% ammonium acetate, magnified.

There is a commerical precedent for the first part of this theoretical treatment. Acrylic floor waxes are often formulated with an ammonia-zinc complex. As the ammonia evaporates, the zinc ions are freed to form crosslinks between the acrylic acid units of the polymer chains. The final finish is a crosslinked, durable polymer, but one that can be removed, when it yellows or ages, by the application of a dilute solution of ammonia [27].

However, wool substrates would be adversely affected by ammonia. Alkali can seriously degrade any protein; it can hydrolyse peptide bonds and amide side-chains. Wool is particularly susceptible to alkali damage since the disulfide bonds which provide the cystine crosslink can be destabilized by alkali [16]. An alternative ammoniacal agent, often used with wool dyeing, is the ammonium salt of acetic acid, ammonium acetate. This is actually neutral at room temperature (3.5 g/l has a pH of 6.96). When used in dyeing neutral metal complex dyes, the addition of ammonium acetate (3% based on the weight of the sample) insures a slightly acid bath as the ammonia evaporates (the same solution will have a pH of 6.38 at 80°C.[5, 16].

Experimental.

Four samples, each approximately one inch by three quarters of an inch, were cut from a loose portion of the tertiary backing of the Brussels carpet (see Figure 12). Each sample had about the same amount of latex backing upon the verso. Each sample weighed between 0.14 and 0.16 gr. Solutions of 10% ammonium acetate in deionized water and of 10% ammonium hydroxide in deionized water were made up. The samples were treated at room temperature (70°F ± 2°F) to the following solutions:

Sample 1) 5 ml 10% ammonium acetate (circa 3.2% o.w.s.)
Sample 2) 5 ml 10% ammonium hydroxide (circa 3.2% o.w.s.)
Sample 3) 2.5 ml ethanol + 2.5 ml 10% ammonium hydroxide
      (circa 1.6% each component o.w.s.)
Sample 4) 2.5 ml ethanol + 2.5 ml 10% ammonium acetate
      (circa 1.6% each component o.w.s.).

The samples were observed under low powered magnification at various intervals (see Figure 13). After fifteen minutes of submersion, the latex backing had become opaque and the color of milk. Its distribution and density was strikingly visible. The samples all showed greater flexibility--a softer handle--than that exhibited prior to submersion. In addition, all four samples had small areas of their latex surface dotted with orange spots; the density of this unexplained orange coloring increased at the highest points of the yarn; its occurrence was not related to the density of the original latex film. After one hour, Samples 2 and 3 exhibited indications of separation of the polymeric layer: the latex was beginning to break up into parts, although these parts seemed to remain attached to the fabric substrate.

On the recto, it was apparent that the latex had also swollen. The interstices of all four samples were filled with the same soft opaque polymer. Although the difference between fifteen minutes and two hours did not seem pronounced for the recto of Sample 3, for the recto of Sample 4, the change over just one hour was clear. The samples were allowed to sit immersed in their respective solutions for a total of four hours. At the end of that time, the pH of all four sample solutions was taken, using Special Indicator Sticks made by E.Merck (Darmstadt, West Germany). The readings were as follows:
Sample 1) pH 7.0 - 7.5.
Sample 2) pH 10.5.
Sample 3) pH 10.0
Sample 4) pH 6.5 - 7.5.
It should be noted that the solutions at this time had a yellowish caste. Sample 2, which contained 10% ammonium hydroxide, had the strongest coloring, and Sample 4, with half ethanol and half 10% ammonium acetate, had the least coloring. Next, all were individually rinsed in deionized water. The samples themselves were separately set onto clean mylar film and cut in half: one half was air dried, one half dried with acetone.

Sample 1 was selected for the second section of the treatment, based on its final pH, its appearance, and the appearance of its solution. Because infrared spectra of the latices associated with the Brussels carpet had conformed in some respects to the tetrapolymer containing poly(vinylidene chloride), a solvent for that polymer was considered: 1,1,1, trichloroethane. This solvent is also a common dryside spotting agent and easily available. It is not a "good" solvent in the sense that it had lower dispersive
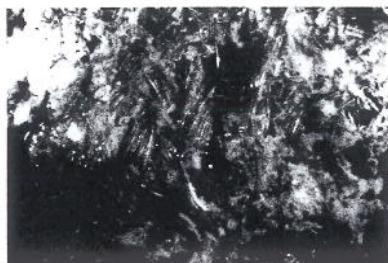
Figure 14. The latex coating on Sample #1 during its first rinse with 1,1,1-trichloroethane, magnified.

Figure 15. The burlap backing of Sample #1 after the final rinse, with no trace of latex on the fibers, magnified.

forces, higher dipolar forces, and lower hydrogen bonding (70/19/11) than the ketones used previously . It did allow the polymeric material to flake off the fabric substrate. Again submersion rendered the latex white and opaque. Under low magnification, the action resembled skin flaking off a sunburned body, but less adhesion was involved (see Figure 14). In fact, the issue of removal was reduced to the problem of flushing away enough of the polymer so that loose flakes were not caught in the loose fibers of the yarns.

Two rinses were able to render Sample 1 visibly free, when dry, of any polymeric material and as flexible to the touch as a fabric which had never been treated with latex adhesive (see Figure 15). At 25X magnification, however, some residue of the latex could be seen in the interstices of the weave. A third rinse rendered the sample virtually clean.

Discussion.

Visual examination was sufficient only to show the existence of a problem, not to explain it nor to suggest a remedy. The analytical methods for identification also had benefits and drawbacks. Solubility parameter tests produced the results that led to the other analytical work, although solvent testing itself did little more than show that solvent treatment alone would not work. X-Ray Fluorescence allowed a definitive identification of certain elements in the polymer but did not explain the quantity of inorganics nor the role those inorganic elements played in the adhesive. Yet, XRF analysis did assist the subsequent interpretation of infrared spectra. The infrared spectra of twenty year old mixed polymer adhesives were not easily interpreted. Alterations in the proportions of polymeric constituents change the spectra and the presence of inorganic substituents further complicate the spectra of the polymer. Knowing which inorganic elements were present helped in the interpretation [20, 21].

The precise nature of the polymeric material remains a mystery. It may be that more sophisticated extraction methods, including separation by size exclusion chromatography prior to infrared spectroscopy might allow distinct components to appear. Yet these methods are not readily available to practicing conservators. Inorganic analysis is easier to obtain. Confirmation by wet chemical testing may be available as well. While the exact nature of the polymer is still unclear, the number of possibilities was reduced to reasonable limits with the analytical methods described. This was enough to develop the course of treatment.

Conclusion.

The treatment described above provides a useful method for reversing damage previously found irreversible. Antique textiles with partially or fully carboxylated latex backings would, of course, require careful examination to determine whether or not the colorants, the weave structure, the yarn conformation, and the overall condition could withstand the initial swelling of fiber and latex. Carpet and textile conservators make decisions of this type on a daily basis and the protocols are well established. No treatment is suitable for all objects, even those of the same type, history or coloring.

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