### Archaeological Preservation Research Laboratory Report 20:

# Comparison of the Bulking Abilities of Polyethylene Glycol 1450 and PS341 Silicone Monomers

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Organic materials such as waterlogged leather undergo complex changes in the marine environment that makes the process of conservation a challenge. Conserving a leather artifact so that it looks as natural as possible and is not prone to damage due to long term chemical reaction is a problem, that all conservators have to address. (Jensen, 1987). Freeze-drying and solvent dehydration processes, are two commonly practiced methods of conserving archaeological leather, and in both cases, the use of polyethylene glycol (PEG) 400 as a humicant aids in reducing the rate of shrinkage. As free water escapes from the waterlogged leather, contractile forces resulting from this change in state draws protein fibers together, causing hardening and shrinkage within the artifact. Bulking, or replacing water with a suitable material that maintains the diagnostic features of the artifact as well as a natural look to the leather is a necessity if the artifact is to be stabilized and conserved.

To compare the bulking ability of polyethylene glycol (PEG) 1450 and PS341 silicone, a strip of waterlogged leather was cut into two sections, each measuring 5.9 centimeters in length and 4.6 centimeters in width. The leather used for this experiment was taken from a large tanned sample that had been allowed to soak in a solution of sodium hydroxide and water for over a year, as a means of ensuring that the leather was uniformly degraded. Prior to treatment, both pieces of leather were washed in a bath of running water and allowed to rinse in a fresh water bath for seven days to ensure that salts, sediments and chemicals were removed from the samples. After rinsing, both samples were lightly patted dry in paper towel and then each was weighed and measures, taking care to note the points along the length and width of each sample at which measurements were taken. The data for each sample is listed below.

After both samples were measured and weighed, they were placed into a beaker containing acetone. To ensure the rapid removal of free water from both samples, the beaker containing the samples was placed into a freezer-mounted vacuum chamber and a vacuum of 26.5 Torr was applied to the samples for eight hours. Near the end of this process, rapid bubbling had ceased in the container, signifying that the dehydration process was complete. At this point, the used acetone was decanted off the samples and

replaced with fresh acetone. The samples were then stored in solution in the freezer for twelve hours before additional treatments were started.

After dehydration, sample "A" was placed into a clean beaker containing 500 milliliters of PS341 silicone and sample "B" was placed into a beaker with an equal amount of PEG 1450. Both samples were then placed back into the freezer's vacuum chamber and a vacuum of 26.5 Torr was applied to the samples. After seven and one half hours, ethyltrimethyloxysilane, a cross linking agent, was added to form a 5% mixture in the PS341 beaker. A vacuum of 26.5 Torr was then reapplied to both samples for an additional thirty minutes. In total, both samples were treated in their respective bulking agents for eight hours.

While the bulking process was taking place, a warming oven was preheated to 135 degrees Fahrenheit in preparation for curing sample "A" in the fumes of Fascat 2003 catalyst. To ensure that the leather samples would be in contact with the concentrated fumes of catalyst, a containment chamber consisting of an inverted polyethylene pail with its lid serving as the base was placed into the warming oven. In the center of the lid, a flat tray containing two ounces of Fascat 2003 was positioned and a four inch square piece of expanded steel was placed on top of the tray, acting as a platform on which the leather sample could be placed. Once removed from the freezer, both pieces of leather were allowed to drain excess free-running bulking agents from their surfaces and then sample "B" was placed into a vat containing acetone and dry ice as a means of flash freeing the sample. Once frozen, the sample was moved to a frost-free freezer compartment where sublimation of free water in the samples was allowed to dissipate off. Sample "A" was placed onto the mesh screen and with the lid of the containment chamber seated into the base, the warming oven was closed, allowing the leather to be exposed to the fumes of the catalyst.

After three days of curing, sample "A" was removed from the warming oven. The freezedrying time for sample "B" was considerably longer than the time frame required to completely conserve sample "A." The process of freeze-drying this leather sample lasted for three weeks. When sample "B" was removed from the freezer, both samples were reweighed and measured as a means of comparing physical changes that occurred as a result of the treatments. Pre and post-treatment measurements and observations are listed in Figure 1.

After conservation, sample "A" appeared completely normal in coloration and texture, although the leather felt stiff and hardened after treatment. Sample "B" however, was also hard and the surfaces of the leather were coated with what appeared to be a fine hydroscopic layer of PEG. In an attempt to soften the leather samples, Neetsfoot oil was worked into their surfaces and then each sample was manipulated and rubbed. After a few minutes of manipulation, both samples were patted dry of excess lubricant and buffed with a lint free cloth.

Observations

Considering that the leather used for this experiment purposely degraded, in the hopes of providing samples that were relatively uniform in their degree of degradation, these samples were ideal tar comparing the results of different bulking techniques. Serving as controls, several test samples, which were allowed to air-dry without treatment, dried to a shriveled, hardened state after several hours. Since sample "A" and "B" did not become distorted, like these test strips, some degree of bulking had occurred in each sample. The observation that sample "A" was slightly harder in its conserved state than sample "B" was surprising at first, because it was assumed that sample "B" would undergo greater shrinkage during the drying process and therefore, be harder in texture and less flexible. Because of the greater diffusion coefficient of PS343 however, PS341 more easily impregnates the cell structures and voids of the leather, resulting in more completely packing the waterlogged matrix (Munnikendam, p.97). The reason sample "A" feels more inflexible is that for the given period of bulking allotted for these samples, PS343 is capable of deeper and more complete penetration into the fibers of the leather where, as Munnikendam has observed, the monomer forms a harder compound which affords greater mechanical strength to the conserved artifact (Munnikendam, p.98). During the process of bulking the leather sample with PEG, free water in the sample is more easily dehydrated than the diffusion ability of PEG to replace it, since the undiluted bulking agent has a lower rate of diffusion than either water or PS343 silicone. This has the potential for causing additional damage to the integrity of the artifact beyond the destruction caused from waterlogging. To more successfully bulk leather with PEG, a process structures around the incremental addition of PEG into a water solution over a longer period of time, would be much more effective.

Predictably, the application of Neetsfoot oil to the silicone treated sample had no effect and the lubricant appears to have been wiped from the surface of the leather during the process of buffing. The application of this lubricant did soften the texture of sample "B" and after a period of manipulation, the sample was more supple. After thoroughly blotting and buffing each sample with a lint-free cloth, sample "B" was noticeably more greasy in texture. The hygroscopicity of the PEG used in conserving this sample may be a concern for long-term curation. With the exception of a slightly higher percentage change in post treatment width for sample "A" (.06%), most of the greatest changes in weight and physical dimensions were recorded for the PEG treated leather sample "B." The conserved weight of this sample had decreased substantially from its per conservation wet weight. Considering that both samples were identical in their cut dimensions the recorded wet weight of sample "B" is questionable since it is 1.1 grams heavier than sample "A." There are a couple of possible explanations for this seeming incongruity in weight. Sample "B" was either holding more free water prior to treatment, or the sample was insufficiently pat dried with paper towels prior to weighing. Because sample "B" has decreased in weight at a rate nearly 7.26 times that of sample "A" and yet has not deteriorated to the state of the control test strips, insufficient surface drying scams to be the best explanation for its weight loss.

#### Conclusions

When choosing conservation processes that are in the best interest of the artifact, the conservator must consider many factors. Tanning processes, species of animal, usewear and environment in which the artifact was found are all important considerations when determining appropriate conservation strategies. If the artifact has been greatly weakened due to processes of decomposition, or if the finished artifact needs increased mechanical rigidity, the strength imparted to the impregnated leather artifact by PS343 bulking may be an important consideration. The greater diffusion coefficient of the smaller molecules of PS343 also makes this an ideal choice for the conservation of larger and thicker leather artifacts, or in situations where the conservator must be concerned about thorough and deep penetration of bulking agents into the collagen fibers of the leather.

There is no doubt that the PEG treated leather sample was softer in comparison to the silicone treated leather, but the potential for problems of hygroscopicity in leather treated with this kind of monomer is an important issue for long-term curation. In terms of overall treatment methodologies, PEG treated samples appear to require more controlled curation, in which temperature, humidity and light sources are necessary considerations. In situations where maintaining physical dimension is critical, the silicone treated leather displays superior ability to easily bulk the leather reducing the rate of shrinkage greatly. Post treatment manipulation and softening of the silicone treated sample however, was much more difficult than the PEG treated leather. The intent of this experiment was to compare the general bulking ability of PS343 silicone and PEG 1450, as well as compare the post treatment results of bulking in terms of aesthetics and practicality. Arguably, treatments and procedures can be tailored for either process, allowing the conservator to accentuate desired characteristic to be imparted to the finished artifact. Quantities of polyethylene glycol 1450 and PS343 silicone used in this experiment were very small and although the cost per unit of silicone compound is more than that of PEG, the wage-hours and lower projected costs of curation and maintenance of the silicone treated leather make it less expensive in the long run, than the PEG treated sample.

| Sample | Pre<br>treatment<br>weight/g | Post<br>treatment<br>weight/g | %<br>Change | Pre<br>treatment<br>length/cm | Post<br>treatment<br>length/cm | %<br>Change |
|--------|------------------------------|-------------------------------|-------------|-------------------------------|--------------------------------|-------------|
| A      | 4.00                         | 3.85                          | -3.75       | 5.50                          | 5.35                           | -2.654      |
| В      | 5.10                         | 3.70                          | -27.25      | 5.90                          | 5.68                           | -3.728      |

#### References

Munnikendam, R.A. 1973, The Conservation of Waterlogged Wood with Glycol Methacrylate, Studies in Conservation, 18, 1973, pages 97-99, The Journal of the International Institute of Conservation of Historic and Artistic Works, London, England.

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