

Archaeological Preservation Research Laboratory Report 2:

Re-treatment of Peg Treated Waterlogged wood

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Purpose: To extract polyethylene glycol (PEG) 3350 from waterlogged wood and cross link residual PEG using CR-20 Passivation Crosslinker and catalyst vapor deposition.

This experiment is being published for the purpose of demonstrating some of APRL's technologies directed at the re-treatment of PEG treated artifacts. These processes are particularly important for composite artifacts in which PEG treated wood must be in contact with iron. Additionally, these processes offer many advantages to the conservator for the stabilization of PEG treated artifacts. There is a growing concern that PEG breaks down or becomes molecularly unstable over time when it is used as a bulking agent in organic materials such as waterlogged wood. It is currently believed that after sixty to seventy years, PEG becomes structurally depleted due to ongoing interaction between the bulking agent, humidity and temperature variations. The end result is that, even with modern curation techniques, extensive pooling and intra cellular migration of PEG may occur. This movement of PEG results in cellular stresses which act to degrade the treated matrix as well as distort and in some cases, destroy the organic host.

To create a sample base for experimentation, hundreds of birch tongue depressors (TD's) were placed into a 4 liter stainless steel beaker and immersed in water. Mesh screen and a heavy weight were placed on top of the TD's in solution to prevent them from floating during the waterlogging process. The TD's in solution were then placed on a plate warmer and heated to a temperature of 110 degrees Fahrenheit for eight hours a day for ten days and then stored in a sealed jar containing freshwater at room temperature. Samples of these TD's were allowed to air-dry at room temperature to determine if the structural integrity of the samples had been compromised. After twenty-four hours, extensive warping and shrinking of all samples was noted which suggested that the TD's had been sufficiently waterlogged for the purpose of testing conservation techniques.

Twenty TD were taken from the waterlogged sample group and placed into a large jar containing fresh water and stored in a vented warming oven set at 158 degrees Fahrenheit. Ten percent increments of PEG 3350 were added to this solution until a concentration of approximately 58% PEG was reached. Throughout this "preservation"

phase, the water level was maintained by adding a small amounts daily to account for evaporation.

#3 PEG TD Experiment.

A single TD was removed from the PEG solution and surface with paper towel to remove free-flowing PEG from all of its surfaces and labeled as sample "3." This sample was then placed into a 50 milliliter graduated cylinder containing 50 milliliters of fresh CR-20 Passivation Crosslinker. A loose fitting cap was placed over the top of the cylinder prior to its placement into a vented warming oven set at 158 degrees Fahrenheit (Figure 1).

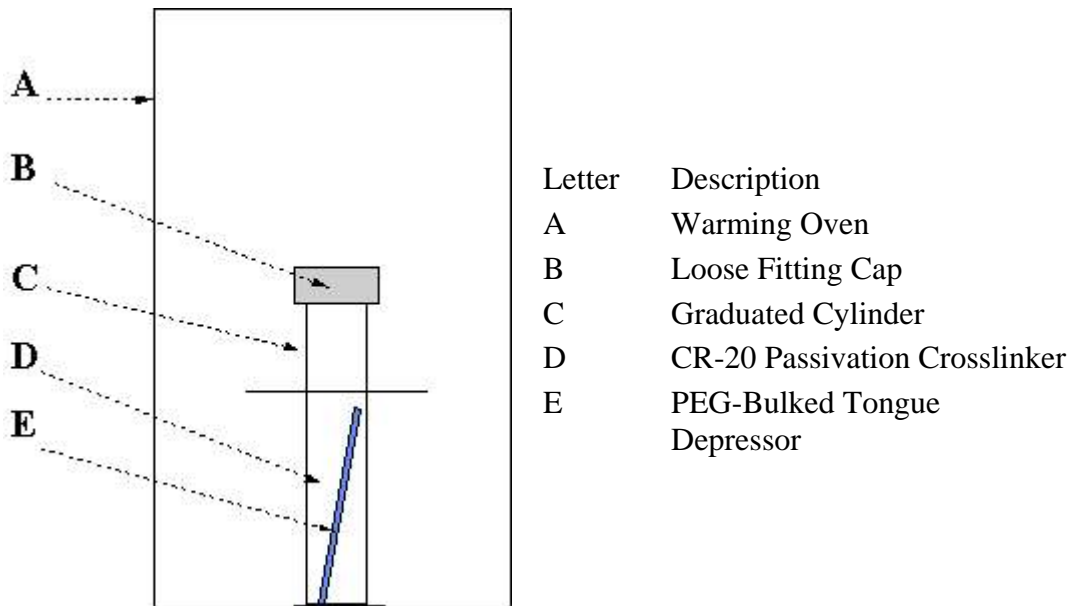


Figure 1. Graduated cylinder set-up.

This sample remained in heated CR-20 Passivation Crosslinker immersion for 24 hours and then allowed to remain in the solution at room temperature for 5 hours. Following CR-20 Passivation Crosslinker immersion the surface was lightly wiped with a paper towel. Figure 2 is a photograph showing the large volume of free flowing PEG that had been removed from the tongue depressor. As the CR-20 Passivation Crosslinker solution cooled, the PEG remained in suspension, appearing as a fluffy white substance.

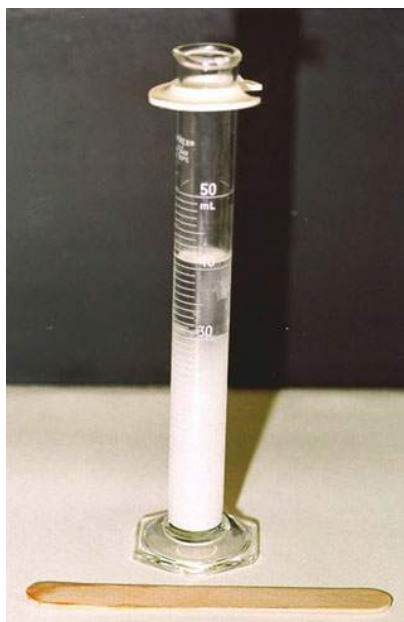


Figure 2. Graduated cylinder with PEG suspended in CR-20 Passivation Crosslinker solution. The re-treated tongue depressor is displayed in front of the cylinder.

The sample was then placed into a tall containment chamber. A catalyst tray was created using an aluminum sample tray that was covered with a 1.75 inch square piece of aluminum expanded screen. This screen formed a platform on which the sample rested, positioning above the catalyst tray during catalyst vapor deposition processing. Fifteen grams of CT-32 were placed in the catalyst tray, the TD positioned on the screen and the body of the containment chamber placed in position. Several small pieces of paper towel were placed on top of the screen before the treated TD was placed in the unit to absorb free-flowing PEG that otherwise might contaminate the catalyst. Once assembled, the unit was placed into a vented warming oven set at 160 degrees Fahrenheit. The sample remained in catalyst vapor deposition for 24 hours. Following treatment, the sample was removed from the oven, transported to a vented fume hood and opened.

Observations

After bulking with the PEG 3350 the treated TD was dark brown in color. The edges of the TD appeared to be translucent and the surfaces of the sample felt waxy and smooth to the touch. Once the CR-20 Passivation Crosslinker treated sample was removed from solution, the solution the graduated cylinder was nearly half filled with a fluffy looking white material which is suggested to be extracted PEG. One sample in 50 milliliters of CR-20 Passivation Crosslinker produced approximately 25 milliliters of this white material.

The tongue depressor itself had been transformed. Instead of being dark brown as in its post bulking state, it was a light gray-brown color, which is much closer in coloration to that noted for the control samples. This sample appeared to retain all of its diagnostic

attributes. No shrinkage was noted and after several weeks of exposure to air, the sample did not warp or change structurally.

#4 PEG TD Experiment

Sample #4 PEG TD's were treated using the same methodology and procedures as were used for sample #3 PEG TD. Instead of one TD in treatment, #4 TD experiment used two PEG 3350 bulked TD's that were treated in 100 milliliters of CR-20 Passivation Crosslinker. After treatment in this solution, the TD were catalyst vapor treated using 15 grams of TPT Titanate instead of CT-32.

Similar results were noted after the CR-20 Passivation Crosslinker treatment as were noted for #3 TD except that with double the CR-20 Passivation Crosslinker silane and two TD's in treatment, there was nearly double the amount of white fluffy extracted material when the TD's were allowed to sit in room temperature for several hours. In total, there were 45 milliliters of this material in the graduated cylinder once the samples were removed.

#5 PEG TD Experiment

Sample #5 PEG TD's were treated using the same methodology and procedures as were used for sample #3 and #4 PEG TD's. Only one TD was treated in this experiment. This samples was placed into 50 milliliters of CR-20 Passivation Crosslinker following the same methods as were used for #3 PEG TD experiment. After treatment in this solution, the TD's were catalyst vapor treated using 15 grams of CT-30.

Similar results were noted after CR-20 Passivation Crosslinker treatment as were noted for #3 PEG TD. In total, there were 45 milliliters of this material in the graduated cylinder once the samples were removed.

#6 PEG TD Experiment

#6 PEG TD was treated using the same exact procedures as were used for all of the other TD experiments except that CR-20 Passivation Crosslinker treatment time was reduced from 24 hours to 6 hours. After CR-20 Passivation Crosslinker treatment, this sample was catalyst vapor treated using 3 ounces of CT-32 catalyst instead of 15 grams catalyst as was used with the other samples. After six hours of CR-20 Passivation Crosslinker treatment, 20 milliliters of fluffy residue were noted in this graduated cylinder.

#7 PEG TD Experiment

This TD was treated using the same methods and times as were used for #6 PEG TD (six hours of CR-20 Passivation Crosslinker immersion and CT-32 vapor treatment for 24 hours). The only difference is that this sample was immersed in a 100 milliliter solution of CR-20 Passivation Crosslinker with 3% CT-32 added.

Results for all of these experiments are noted in the table below:

Sample(s)	Chemistry	Post Treatment Tongue Results
#3	CR-20 Passivation Crosslinker CT-32 vapor	<ul style="list-style-type: none"> - Slightly more brown than controls - No warpage noted - Slight swelling noted - 25 ml white residue in suspension
#4	CR-20 Passivation Crosslinker TPT Titanate vapor	<ul style="list-style-type: none"> - No warpage - Slight swelling noted - Close in color to controls - 45 ml white residue in suspension
#5	CR-20 Passivation Crosslinker CT-30 vapor	<ul style="list-style-type: none"> - Good color - Slight swelling noted
#6	CR-20 Passivation Crosslinker CT-32 vapor	<ul style="list-style-type: none"> - Gray-brown in color - Slight swelling noted - 20ml white residue in suspension
#7	CR-20 Passivation Crosslinker 3% CT-32 CT-32 vapor	<ul style="list-style-type: none"> - Slightly gray-brown - Slight swelling noted - 15 ml white residue in suspension noted

#8 PEG TD Experiment

#8 Peg TD experiment combined some of the processes used for the other TD experiments however, some processes were adopted so that the tongue depressor could be molded to the shape of the side curvature of a small tin.

While removing a TD that had been bulked with PEG 3350 from its warm solution, it was noted that the TD was very flexible. This flexibility was lost once the TD cooled to room temperature. Using gloves and a hot air gun, this sample was slowly warmed. The side surface of a one pint tin can was also warmed with a hot air gun and while both TD's were still hot, one was wrapped around the side of the can and held in place until returned to room temperature (approximately 20 minutes). Rubber bands were then stretched around the TD positioned so that they held it in position against the side of the can. The TD wrapped can was then placed inside a 5 quart can with a weight placed on top of the 1 pint can to prevent it from floating. Approximately 1 liter of CR-20 Passivation Crosslinker was poured into the 5 quart can to submerge the wrapped TD. Once a loose fitting lid was positioned on top of the larger, outer vessel, the unit was placed in a vented warming oven set at 158 degrees Fahrenheit for 4 hours. The entire unit was then removed from the oven, and moved to a vented fume hood, the small can and TD were removed from the CR-20 Passivation Crosslinker solution.

Still wrapped on the small can, the TD was then placed in a large containment chamber resting on expanded aluminum mesh, over a catalyst tray containing 3 ounces of CT-32 (Figure 3). With the lid of the containment chamber in position, the unit was placed into a vented warming oven for approximately 18 hours (overnight). The sample in its containment chamber was then removed from the oven and opened under a vented fume hood. The rubber bands were removed and the shaped TD slid easily off the side of the tin can.

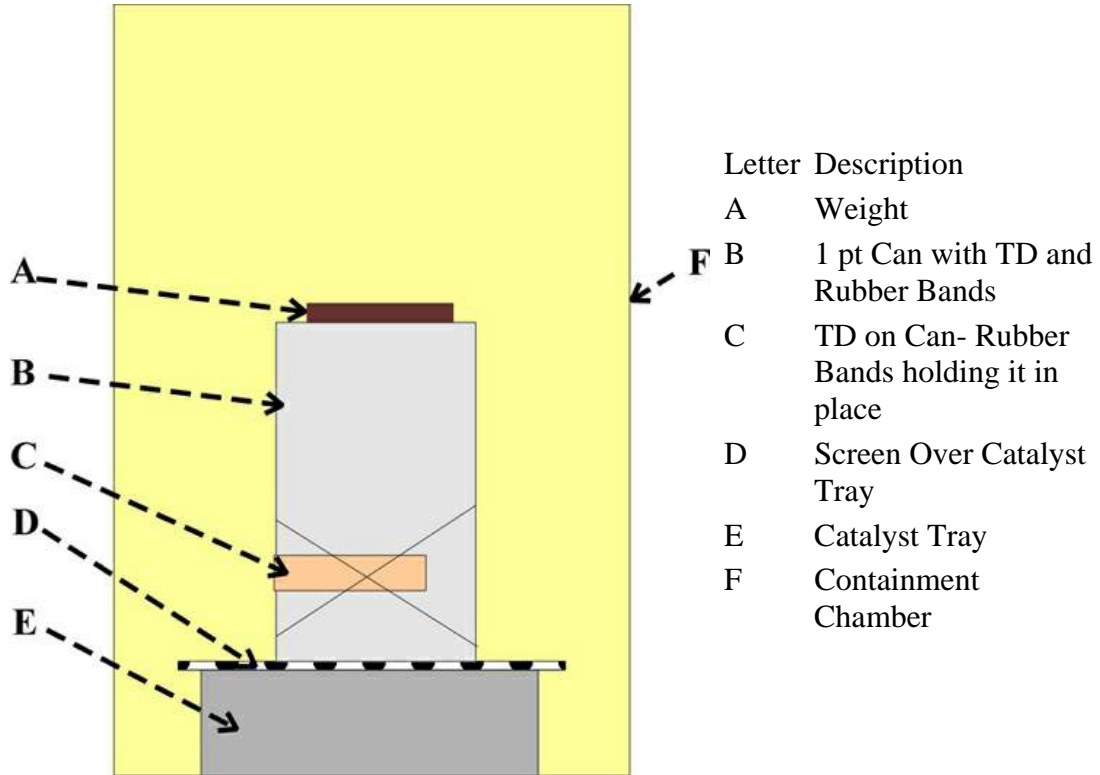
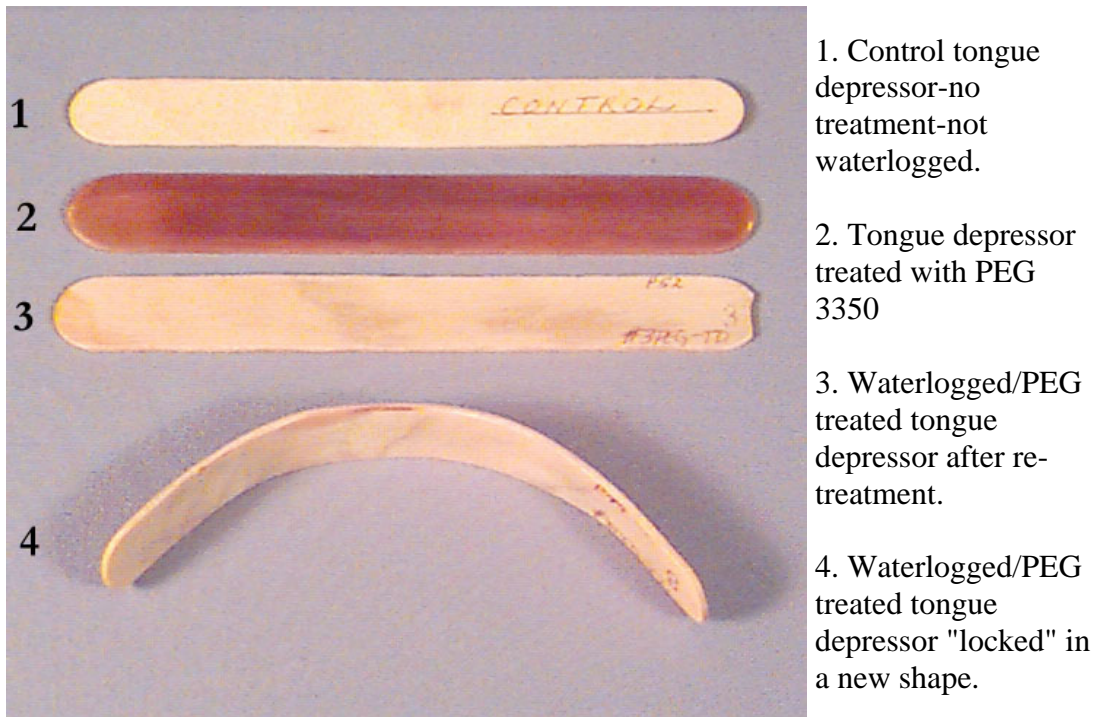


Figure 3 Catalyst Vapor Deposition Configuration



Conclusions

After treatment, the profile of the TD was traced so that the sample could be compared in shape periodically to determine if the TD had been sufficiently locked into this new, semi-circular shape. Over time, the tongue depressor remained stable, and has not changed shape. Sample Number 4, has been heat treated to determine if the tongue depressor will change shape in elevated temperatures. After allowing this re-shaped specimen to sit in a vented oven, preheated to 70 degrees Celsius for five hours, there was no indication that the sample was in any way stressed or changing shape.

Report number three will demonstrate this re-treatment process on an actual artifact which had been treated using polyethylene glycol.

Citation Information:

Smith, C. Wayne

1997 "Re-treatment of PEG Treated Waterlogged Wood," Archaeological Preservation Research Laboratory (APRL), Report 2, World Wide Web, URL, <http://nautarch.tamu.edu/APRL/report02.htm>, Nautical Archaeology Program, Texas A&M University, College Station, Texas. November 5, 1997