ON THE REVERSIBILITY OF CONSOLIDATION TREATMENTS OF DETERIORATED WOOD WITH SOLUBLE RESINS

by Arno P. Schnlewind*

Extensive experiments have been carried out on reversibility of treatments with Butvar B98, Acryloid B72, and AYAT, using both polar and non-polar solvents. Most experiments were made using Soxhlet extraction, but removal by soaking, with and without agitation, was also investigated. With resin loading levels after treatment in the range from 21.5 to 27.7 percent, residual resin levels after extraction and before correction for removal of wood extractives ranged from -0.97 to 6.0 percent.

Introduction

The question of reversibility of conservation treatments is one of the most basic concerns of conservators. It is a question that arises in connection with all types of treatments, including consolidation treatments of deteriorated wood artifacts. Although true reversibility in a strict interpretation of the term does not exist, reversibility remains a guiding principle and an ideal to be pursued (1, 2).

Selection of the type of consolidant is the most important step in determining if a consolidation treatment can even remotely be considered reversible. The use of thermosetting resins, for instance, makes the treatment irreversible as soon as they are cured. Thermoplastic resin solutions, on the other hand, which solidify by evaporation of solvent, do offer a chance of approaching reversibility. The present study was initiated to examine the reversibility of consolidation treatments with soluble resins for deteriorated wood.

The principal factors that must be taken into account, namely the redistribution and removal of wood extractives that are soluble in the solvent used and the removal of wood -- moisture by water-miscible solvents, were previously discussed (2). Preliminary experiments had shown that bacterially degraded Douglas-fir treated with a 15% solution of Acryloid B72 in acetone retained an average of 23.6% of polymer. Subsequent extraction yielded a residual resin content of -0.10%, i.e. an apparent loss. Correction for an extractive content of 0.61 percent removed by the same extraction procedure from a control set then gave a final residual resin content of 0.5% (2). Expressed another way, 2.1% of the consolidant impregnated into the samples remained after extraction. Since it is most unlikely that an equally intensive method of extraction (Soxhlet apparatus) could ever be employed with actual artifacts, this residual confirms that the consolidation treatment was not reversible in the strict sense. On the other hand, 0.5% of residual consolidant in wood will not have a significant effect on wood properties nor is it likely to interfere with any subsequent treatment. The treatment with Acryloid B72 can therefore be considered as virtually reversible for all practical purposes.

***Professor of Forestry and Acting Director, Forest Products Laboratory, University of California, 1301 south 46th Street, Richmond, CA 94804**

Material and Methods

The wood used for the experiments was taken from the bacterially degraded surface layers of Douglas-fir foundation piles that had been in the ground for approximately 70 years (2). Sections of these piles had been stored outside for six years allowing them to dry and weather during that time. Over 60 such sections were available, and from these four sections of relatively sound and four of the most severely degraded appearance were selected for study.

The pile sections were cut into a large number of specimens 2 inches long, 1 inch wide, and approximately 1/4 inch thick, one of the wide face being the undisturbed surface of the pile. The specimens were. conditioned to a nominal moisture content of 12% in a controlled temperature and humidity room. Specimens were kept in 8 lots representing the 8 piles selected for study. Experiments were made in sets of eight specimens, one being selected randomly from each of the 8 lots. The general appearance of the specimen material was illustrated in a previous report (2).

Resins used were a polyvinyl butyral, Butvar B98, an acrylic, Acryloid B72, and a polyvinyl acetate, AYAT. Consolidant solutions were made with acetone or toluene for both AYAT and B72, and with methanol or a mixture of 40% ethanol and 60% toluene (weight basis) for B98. Resin concentration was 15% (weight basis) in every case.

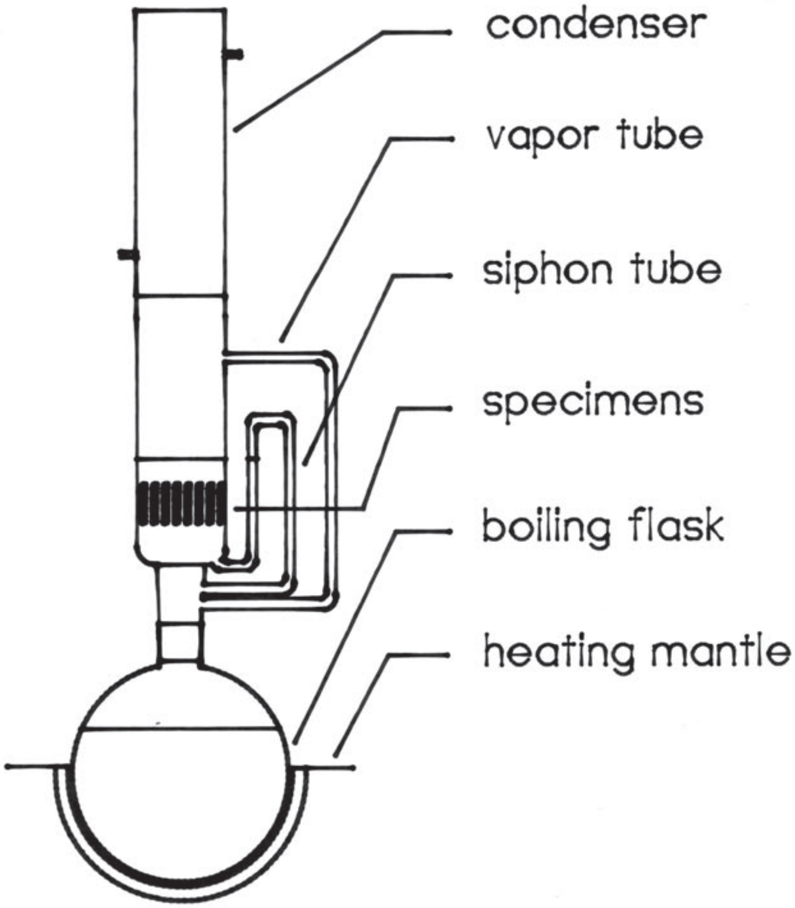

Fig. 1. Soxhlet extraction apparatus.

Specimens to be treated with consolidant were weighed first and then soaked for 2 hours in resin solution. They were then vacuum impregnated according to the procedure described previously (2), followed by another 30 minutes of soaking in resin solution. Specimens were then removed from the solution, blotted, allowed to dry, and finally conditioned to constant weight in the controlled humidity room. They were then weighed to determine the percentage of consolidant loading.

Extraction was carried out in one of three ways, namely by Soxhlet apparatus or by soaking in solvent, either with or without agitation. Soxhlet extraction would be a practical procedure for only the smallest of objects, but it is a very intensive extraction method that can establish whether resins can be removed at all. The Soxhlet apparatus is illustrated in Fig. 1. Solvent in the flask below is heated to boiling by an electric heating mantle. The resulting vapor is condensed and rips over the specimens located in the sample chamber. When the sample chamber becomes filled, u the solvent is siphoned back into the flask to begin a new cycle. Each cycle of filling and siphoning required approximately 75 minutes for acetone and methanol as solvents with lower boiling points, while toluene with its higher boiling point required approximately 95 minutes. Extraction was continued for 4 days, or approximately 77 cycles for acetone and methanol and approximately 60 cycles for toluene.

The mixture of ethanol and toluene was not used for Soxhlet extraction because the composition would change during boiling. Ethanol and toluene do form an azeotrope but at a composition other than the recommended ratio of 40/60. Soaking was therefore the best alternative for this particular solvent, but it was also used with other solvents because it has a far greater chance of being applicable in practical situations. Specimens were placed on a coarse wire mesh grid suspended from the top edge of a beaker as shown in Fig. 2. Another grid was placed over the specimens and a weight was added to prevent specimens from floating above the solution surface. Soaking was carried out either in still solvent or with agitation provided by a magnetic stirrer as also shown in Fig. 2. Soaking was carried out over 10 days, old solvent being exchanged for fresh one at 2-day intervals.

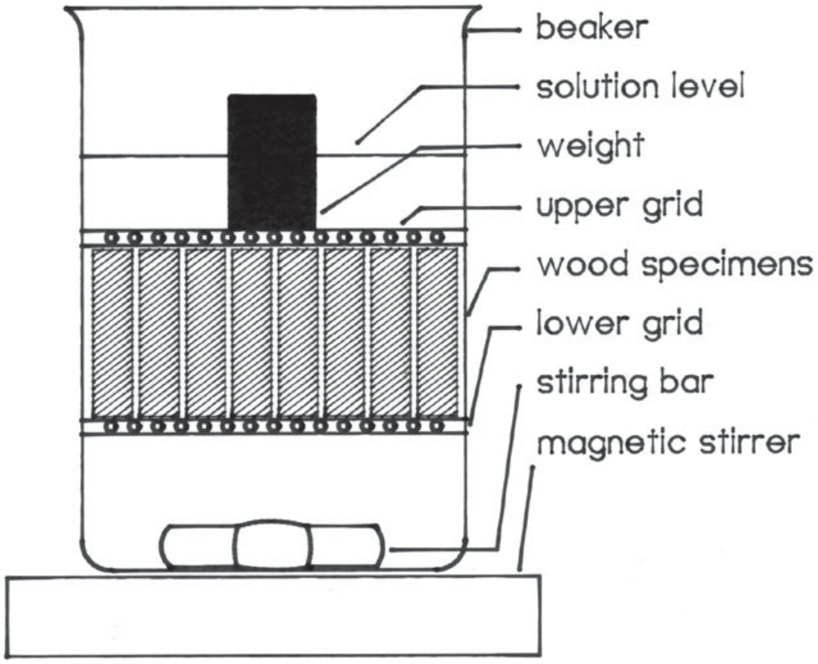

Fig. 2. Arrangement for extraction by soaking.

Extracted specimens were reconditioned in the controlled humidity room to constant weight. Time to equilibrium varied depending on solvent boiling point. Specimens were then dried in an oven at 103°C for 24 hours, cooled inside weighing bottles in a dry atmosphere, and weighed again. They were then coated with hot wax to measure their volume by the water immersion method for determination of specific gravity.

Parallel extractions were made on control sets not treated with consolidant to determine the amount of extractives that would be removed during the extraction procedure. In one experiment, specimens were extracted with acetone first, treated with B72 in acetone, and extracted again, so that all measurements could be made on the same set of specimens.

Results and Discussion

Moisture content of extracted control samples in equilibrium with conditions in the constant temperature and humidity room and their specific gravity are shown in Table 1. Considerable variability is evident, which is typical for wood in general and in particular for wood that has undergone some deterioration. The grand average moisture content was 13.0 percent, which is somewhat higher than the nominal moisture content of 12% for the temperature and relative humidity settings used. The degree of deterioration is reflected in the specific gravity values. Normal Douglas-fir has specific gravity (based on oven-dry weight and volume, as for the data in Table 1) of 0.51 (3). The grand average specific gravity of 0.41 thus indicates a weight loss of approximately 20%, implying a substantial loss in strength. The average value for the relatively sound piles was 0.47 as compared to a value of 0.35 for the more obviously degraded material. Individual piles may of course have a specific gravity that is either higher or lower than the species average when they are new and without deterioration (cf. pile No. 18 which is higher than average even in the deteriorated state), so that estimates of the degree of deterioration are only possible on the basis of many samples.

Table 1. Moisture content and specific gravity of control samples.

Pile No.	Deterioration	Moisture Content (%) Mean	Moisture Content (%) Min/Max	Specific Gravity Mean	Specific Gravity Min/Max
16E	Moderate	13.1*	11.7/13.8	0.48*	0.43/0.61
17	“	13.0	12.3/13.6	0.43	0.40/0.45
18	“	12.7	11.6/13.6	0.57	0.50/0.63
19	“	13.0	11.2/14.0	0.39	0.32/0.44
3A	Severe	12.1	10.4/13.4	0.37	0.33/0.42
20	“	13.7	12.6/14.6	0.26	0.23/0.28
21	“	12.9	12.3/13.8	0.41	0.36/0.48
22	“	13.2	12.3/14.4	0.36	0.33/0.38

*Means of values from 7 control sets.

Wood contains extraneous substances which can be removed by solvent extraction and are therefore usually referred to as extractives. Extraction of consolidants will therefore be accompanied by removal of extractives, and to account for this effect the extractive content under a given extraction procedure was determined on control sets. The results are shown in Table 2. Methanol clearly removed the most extractives at 1.13% based on oven-dry weight of wood. Toluene and acetone form the next group, with averages ranging from 0.61 to 0.75%. In view of the large variability of individual values (cf. minimum and maximum values in Table 2) the averages must be considered to be essentially the same. As this includes acetone extraction by all three methods, it can be concluded that for this particular solvent the extraction method has no apparent effect on the amount of extractives removed. The last group includes extraction with ethanol and toluene mixture, both with agitation and without it. When agitation was included, the amount of extractives removed was very small, and without agitation there was a weight increase. Since it is impossible to add extractives during the extraction process, the weight increase can only be attributed to adsorption of solvent, in this case the ethanol component since toluene is non-polar and will not be adsorbed. Small amounts of polar solvents such as ethanol, on the other hand, can be adsorbed so tenaciously on wood that they cannot even be removed during drying at 105°C (4).

Table 2. Extractive content of control samples.

Test No.	Solvent	Extraction Method	Extractive Content (%) Mean	Extractive Content (%) Min/Max
A3	Methanol	Soxhlet	1.13*	0.36/4.82
C3	Toluene	Soxhlet	0.72	0.25/1.30
G2	Acetone	Soxhlet	0.75	0.45/0.97
3	Acetone	Soxhlet	0.62	-0.11/1.02
I3	Acetone	Soak and agitate	0.64	0.43/1.09
J3	Acetone	Soak only	0.61	-0.03/0.89
H3	ETOH-Tol	Soak and agitate	0.13	-0.90/1.71
F3	ETOH-Tol	Soak only	-0.31	-1.27/0.62

*Each value is the mean of 8 specimens.

Reversibility of three consolidant resins using polar solvents and Soxhlet extraction can be compared as shown in Table 3. The apparent (uncorrected for extractive removal) residual resin content is negative in every case, but the corrected values range from slightly negative for AYAT to 0.8 percent for B98. The data suggest that AYAT is most readily reversible of the three resins, followed by B72 and B98, in order. In practical terms, however, even a residual of 0.8 percent is not a large and significant amount. Nakhla (5) also found that B72 was more reversible than a polyvinyl butyral (Mowital B30H). B98, on the other hand, has been found to be more effective than B72 in other respects (6, 7).

The second set treated with B72 that is listed in Table 3 was extracted prior consolidation treatment, i.e., the treatment was performed on extractive-free wood. When calculations were made in the same manner as for the other sets, the corrected residual resin content was found to be equal to zero. Direct calculation comparing equilibrium weights after extractive removal and after consolidant extraction, which made corrections unnecessary, gave the identical result.

Table 3. Reversibility of resins using Soxhlet extraction.

Polymer	Solvent	Resin Retention (%)	Residual Resin Uncorrected (%)	Residual Resin Corrected (%)
AYAT	Acetone	22.0	-1.0	-0.3
B72*	Acetone	23.6	-0.1	0.6
B72**	Acetone	23.8	-0.7	0.0
B98	Methanol	21.5	-0.3	0.8

*Regular procedure.
**Specimens extracted prior to consolidation.

The effect of using toluene, a non-polar solvent, rather than the polar acetone for both AYAT and B72 is shown in Table 4. In the case of either resin, the reversibility is not as good when toluene is used rather than acetone. One set of specimens (not shown in Table 4) was treated with B72 in acetone but the consolidant was extracted with toluene. Interestingly, the corrected residual resin content was 0.6%, or the same amount as the set treated and extracted with acetone. This would suggest that although the consolidant loading appears to be about the same for all sets of specimens regardless of treating materials and conditions, the internal distribution may differ depending on the type of solvent used.

Table 4. Effect of polar vs. non-polar solvents.

Polymer	Solvent	Resin Retention (%)	Residual Resin Uncorrected (%)	Residual Resin Corrected (%)
AYAT	Acetone	22.0	-1.0	-0.3
“	Toluene	23.2	1.2	1.9
B72	Acetone	23.6	-0.1	0.6
“	Toluene	22.4	0.2	0.9

Table 5 affords a comparison of extraction methods. One would expect that Soxhlet extraction would be the most efficient, followed, in order, by soaking with agitation and by soaking alone. As the data show, for B72 in acetone there is virtually no difference between the first two extraction methods, soaking with agitation giving slightly less residual than Soxhlet extraction, but soaking alone produced more than twice as much residual resin content. By far the most residual resin was left in the case of consolidation with B98 in the mixture of ethanol and toluene. Agitation helps to reduce the residual resin content in this case also. The residual of 5.7% for soaking alone and even the 2.9% for soaking with agitation are large enough to make it advisable to avoid that particular combination of resin and solvent.

Table 5. Comparison of extraction methods.

Resin	Solvent	Extraction Method	Resin Retention (%)	Residual Resin Uncorr. (%)	Residual Resin Corrected (%)
B72	Acetone	Soxhlet	23.6	-0.1	0.6
"	"	Soak & agitate	24.9	-0.3	0.4
"	"	Soak only	23.7	0.7	1.3
B98	ETOH-Tol	Soak & agitate	25.1	2.7	2.9
"	"	Soak only	27.7	6.0	5.7

The appearance of specimens changed somewhat following consolidation treatment. Since the specimens were blotted immediately after taking them from the treating solution, there was no problem with surface gloss, but specimens became somewhat darker with treatment. There were no discernible differences between resins or solvents. However, following extraction, specimens invariably returned to their original, lighter coloring.

In conclusion, the experiments have shown that consolidation treatments of deteriorated wood with soluble resins are not reversible in the strictest sense. The results also show, however, that the lack of complete reversibility is due in part to the simultaneous removal of wood extractives, and that under certain conditions virtually all of the impregnated resin can be removed again.

Of the resins tested, AYAT was most reversible, followed by B72 and then B98, but the difference between them was not large. The use of polar solvents led to better reversibility than non-polar ones. Soxhlet extraction was most effective, but is not a practical method. When attempting to remove consolidants by soaking in solvent, reversibility was much improved by providing agitation.

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