Accelerated, Real-Time Aging
For 4 Construction Adhesives

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In 1969, when construction adhesives were new products, the Forest Products Laboratory initiated a long-term test fence exposure of 17 construction and panel adhesives in “T” panel configuration. Based on early observations of the test fence specimens, and other considerations, four adhesives were selected in 1970 for extensive evaluation of their aging behavior.

The influence of heat and moisture on the strength of joints bonded with the four adhesives was studied using accelerated aging concepts. Analysis of the data by the rate process method resulted in estimates of the bonded joints’ useful life. These analyses, particularly for mastic adhesives, are seldom straightforward. There are pitfalls for the unwary. In the present study additional specimens prepared at the same time were placed in outdoor exposure and in continuous wet and continuous dry exposures at normal service temperatures. Now, estimates of service life based on 11 years of service exposure provide an opportunity to evaluate applicability of estimates of service life from accelerated aging exposure. The purposes of this study were 1) to determine if the rate process method of accelerated aging was useful for evaluating the long-term performance of mastic construction adhesives and 2) to generate information about the long-term performance of selected adhesive types.

**Background**

Uncertainty about the durability of new building products necessarily limits the range of their usefulness. For example, engineered plywood diaphragms bonded with construction adhesive may show improved performance over diaphragms fastened only with nails or staples; but, nails or staples will still be required as a safety factor because the long-term strength and stiffness of the adhesive are unknown. More efficient use of construction adhesives as structural fasteners requires detailed knowledge of their ability to transfer stress between load-bearing building components under actual service conditions for the life of the structure involved. Building a prototype structure and monitoring the adhesive’s performance for 20 years or so is uneconomical and impractical. Even if an adhesive manufacturer could afford to do this in every imaginable service environment, new adhesive technology would render the application obsolete before the test was completed. The problem is, how to use relatively quick tests to estimate the effects of long periods of time on an adhesive’s stress transfer capabilities.

One technique is the “torture test” in which bonded specimens are exposed to a very hostile environment for some period of time, after which the strength of the adhesive is measured and compared to the strength of the adhesive in unexposed specimens. The boil-dry-boil test for exterior plywood specified in U.S. Product Standard PS 1-74 (1) is one example. This test provides exposure to three of the primary bond-degrading factors: heat, moisture, and stress. The problem with this test is that it does not provide an indication of the rate of strength loss, how the rate changes with time or temperature, or which of the degrading factors is the major one. An improved method is the ASTM D-3434 automatic boil test (2) which offers the opportunity to measure rate of loss with respect to the number of boil-dry cycles. The individual contributions of hydrolysis, heat, and swell-shrink stress to the overall degradation rate still cannot be distinguished. However, Weyerhaeuser Co., Takoma, Wash., research has shown good correlation between results obtained by method D-3434 and 11 years of outdoor aging (3).

*Maintained at Madison, Wis., in cooperation with the University of Wisconsin.

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**Table I – Characteristics of Adhesives Used in the Accelerated Aging and Real-Time Aging Study**

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>A</th>
<th>C</th>
<th>E</th>
<th>K</th>
</tr>
</thead>
<tbody>
<tr>
<td>Elastomer base</td>
<td>Polyurethane (moisture cure)</td>
<td>Neoprene</td>
<td>SBR copolymer</td>
<td>Fortified reclaim rubber</td>
</tr>
<tr>
<td>Percent solids</td>
<td>99</td>
<td>53</td>
<td>70</td>
<td>58</td>
</tr>
<tr>
<td>Viscosity cP</td>
<td>520,000*</td>
<td>78,000°</td>
<td>450,000*</td>
<td>176,000°</td>
</tr>
<tr>
<td>Liquid vehicle</td>
<td>None</td>
<td>Petroleum distillate, acetone, toluene</td>
<td>Petroleum distillate, acetone</td>
<td>Petroleum distillate</td>
</tr>
</tbody>
</table>

*Brookfield #7 spindle, 4 rpm.
°Brookfield #7 spindle, 10 rpm.
Another approach is the rate-process method. This method involves analysis of data from constant elevated temperature and constant moisture aging. This method offers the opportunity to study the individual effects of heat, water, and chemicals. At the current level of development it does not account for the effects of stress. The rate process method does offer a rational method to estimate the life of joints at service temperatures (4).

None of these accelerated aging problems, by definition, duplicate real environmental conditions nor can they duplicate the random variation of environmental conditions. One is always left in doubt about the interpretation of accelerated aging test results as they apply to a specific environment. Therefore, research such as published by the Weyerhaeuser Co. (5,6), and as described in this report, adds to the pool of knowledge necessary to interpret accelerated aging test results of new adhesives.

Materials

The four adhesives are briefly described in Table I. Although the adhesives are classified only by the base elastomer, it is important to note that the elastomer may constitute less than 50% of the adhesive solids. Detailed information about their formulation is unavailable. Other components, such as fillers, resins, and solvents, often constitute high percentages or otherwise strongly affect the adhesive’s properties.

The four adhesives and their general performance were as follows (7):

- Adhesive A, a one-part, moisture-curing polyurethane adhesive, is very flexible and, of all the adhesives, showed the best recovery after unloading. Adhesive A bondlines and bond strengths were the most uniform of all the adhesives.
- Adhesive C, a Neoprene type and one of the two strongest, is hard and horny but shows good time-delayed recovery. It had a tendency to form honey-combed or starved joints, depending on the uniformity of the adhesive spread and the uniformity of the veneer thickness. This resulted in high bond strength variability.
- Adhesive E, an SBR type adhesive, also hard and horny, was the other strong adhesive, but its recovery was much poorer than that of adhesive C.
- Adhesive K, a reclaim rubber-based adhesive, is hard but less tough. It was the weakest and had the poorest recovery behavior.

Specimen Preparation

The test specimen selected for the accelerated and real-time aging was a two-ply tensile lap-shear specimen patterned after the ASTM D-2339 specimen. It was made with 1/4 inch (3.2 mm) thick yellow birch veneer instead of 1/16 inch (1.6 mm) veneer. Adhesive was extruded onto one 14-by-14-inch (356 by 356 mm) sheet of veneer, then spread as evenly as possible over the surface with a notched trowel. A second 14-by-14-inch veneer was laid on the adhesive. Ten of these 2-ply panels were placed in a 14-by-14-inch hydraulic press under just enough pressure to cause a slight amount of adhesive to squeeze out at the edges. The panels remained in the press for 24 hr and were removed to complete the cure under a 50-lb weight at 27°C/30% relative humidity (RH) for 30 days. Individual specimens (Figure 1) 1-by-3½ inch (25.4 by 82.6 mm) were cut from the 14-by-14-inch panels after the 30-day cure and stored at 27°C/30% RH until placed in an aging exposure. The storage period ranged from 30 to 200 days.

Accelerated Aging

Groups of 40 specimens selected at random were exposed to accelerated aging at four temperatures and two moisture levels, as shown in Table II. At various intervals, depending on the temperature, subgroups of five specimens were withdrawn from each exposure, reconditioned to equilibrium moisture content at 27°C/30% RH, and tested for strength. Ideally, specimens of the eighth subgroup were exposed long enough at the given conditions to have lost 50% of their estimated original strength. This time varied with the adhesive and the exposure, but the longest times were less than 100 days at the lowest temperatures. Testing was performed by pulling the specimen in shear with a wedge action “Templin” grip in a universal testing machine at a 10 mm/min rate of crosshead movement. The appearance of the fractured adhesive and the percentage of wood failure were noted.

Real-Time Service

As mentioned previously, when the accelerated aging study was initiated, identically bonded specimens were also placed on outdoor exposure. Additional specimens were stored in water at 27°C and at 27°C/30% RH. Tests of the specimens stored outdoors and those stored wet at 27°C were made periodically to establish rates of strength loss. Specimens stored at 27°C/30% RH were tested only after the 1 1-year exposure period. Strength tests were conducted in the same manner as in the accelerated aging.

Data Analysis

A plot of the data at one temperature-moisture level should look like Figure 2a. This relationship is often represented by fitting the following equation or a similar one to the data.

\[ \log y = A_1 + B_1 t \]  

where

- \( y \) = shear strength
- \( A_1 \) = shear strength at zero aging time
- \( B_1 \) = degradation rate
- \( t \) = aging time

Similar data and equations would be obtained at other temperatures. Then the time required for the strength to degrade to half its original value (shear strength half-life) at each temperature is estimated visually from each plot or by calculation from the fitted equations. Next, shear strength half-life is plotted as a function of the reciprocal of the aging temperature (in degrees Kelvin) shown for the ideal case in Figure 2b.

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**Table II – Accelerated Aging Conditions**

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Wet (°C)</th>
<th>Dry (°C)</th>
</tr>
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<tbody>
<tr>
<td>A</td>
<td>40, 60, 70, 80</td>
<td>100, 115, 130, 145</td>
</tr>
<tr>
<td>C</td>
<td>70, 80, 100, 149</td>
<td>100, 115, 130, 145</td>
</tr>
<tr>
<td>E</td>
<td>40, 60, 70, 80</td>
<td>100, 115, 130, 145</td>
</tr>
<tr>
<td>K</td>
<td>40, 50, 60, 70</td>
<td>100, 115, 130, 145</td>
</tr>
</tbody>
</table>
This is the Arrhenius temperature dependence relationship which can be represented by fitting the data to the equation:

$$\log t_{1/2} = A_2 + B_2/T$$

(2)

where

- $t_{1/2}$ = shear strength half-life
- $A_2, B_2$ = material constants
- $T$ = aging temperature (°K)

The service life (shear strength half-life at service temperature) can be estimated by extrapolation from the plat or the fitted equation.

Rate equations, equation (1), for the specimens stored outdoors and those stored wet at 27°C were determined by least squares linear regression, in the same manner as specimens subjected to the accelerated aging processes. Similarly, the estimated shear strength half-life was determined from each equation. These estimates are compared with the estimates from wet accelerated aging.

Results

Accelerated Aging. Half-life estimates at the accelerated aging conditions are represented by Figure 3. The figure includes the individual data points and the fitted regression lines for wet and dry environments which are estimates of the half-life/temperature relationships. Extrapolation of the relationships to 27°C resulted in the accelerated aging estimates in Tables III and IV (column 2).

Real-Time Service Aging. In all cases except adhesive A, the joints actually reached half their estimated original strength within the 11-year outdoor exposure or wet storage period. The outdoor exposure strength loss relationship with time for adhesive A had to be projected 2 years beyond the actual 11-year aging period to reach half the estimated original strength. Adhesive A data were quite consistent, and the estimate is considered accurate. The estimates of half-life, wet at 27°C, are given in Table III.

Only one test was conducted on specimens stored at 27°C/30% RH. Degradation under these conditions is normally so slow that measurable strength loss was not expected. The results of the single test expressed as the percentage of the estimated original strength retained after 11 years are given in Table IV.

Comparison of Accelerated and Real-Time Aging Results. The half-life estimates from the accelerated aging tests and those from the real-time tests are in good agreement, except for the adhesive E wet (Table III, columns 2 and 3) and adhesive K dry results (Table IV, column 2). Apparently, the aging mechanism for adhesive E changes between the 27°C of the real time and the lowest wet accelerated aging temperature 40°C. The accelerated aging estimate for adhesive A is also somewhat long compared to the actual wet and outdoor results; however, this difference is within the realm of experimental variation. The estimated half-lives for adhesive A are equivalent to the expected service...
life of a high-quality polyurethane sealant.

For adhesive C, the half-life estimates from accelerated aging and the actual 27°C estimates are in close agreement and certainly not distinguishable statistically. The half-life in outdoor aging is roughly half that in continuous 27°C wet aging. The same is true of adhesive E. The reason for these reduced half-lives in outdoor aging is apparently the addition of swelling and shrinking stress in the outdoor environment. Adhesive A, which is very flexible and able to accommodate these stresses, was unaffected, while adhesives C and E, which are more rigid, were affected. The effect of stresses at the edges of the bond area are evident in Figure 4.

As mentioned previously, 11 years in a dry 27°C/30% RH environment is not enough time to even approach the shear strength half-life. The estimated percentages of original strength retained after 11 years (Table IV, column 3) are high and thus support the long accelerated aging half-life estimates. From accelerated aging, adhesive A should lose 9% in 11 years. In real-time aging it lost 11%. Adhesives C and E apparently gained strength in real-time aging. As in all cases, part of the difference between accelerated and real-time results is due to normal variability, but the 119% and 118% increases for these adhesives are probably due to cross-linking after the initial cure. There was evidence of cross-linking in the accelerated aging strength versus time determinations. The half-life for adhesive K from accelerated aging seems very long in comparison to the other adhesives’ half-life estimates and in comparison to the percent of strength retained after 11 years of real-time aging. In this case, as with adhesive E in the wet accelerated aging exposure, a change in the aging mechanism may occur between the lowest accelerated aging temperature (115°C) and 27°C.

### Discussion

The half-life estimates extrapolated for 27°C from the accelerated aging tests represent the average response of a particular group of specimens. The accuracy and precision of these estimates are certainly open to question. At least five factors confuse the rate process analysis, three pertaining to the strength-time relationship and two pertaining to time-temperature.

- Joint strength may increase or decrease rapidly upon the first exposure to elevated temperature (Figure 5a). This response may be due to driving off lingering solvents, chemical cross-linking, internal stress relief, or some other short-term response to a temperature rise.
- The long-term degradation rate may change during the aging period (Figure 5b).
- Both the wood and the adhesive degrade in the aging exposure, and most likely at different rates. If the wood is initially stronger, as is usually the case with mastic adhesive joints, the rate of change in joint strength reflects the degradation rate of the adhesive. But as sometimes happens, the wood may be degrading faster than the adhesive and eventually becomes less strong than the adhesive. Then the rate of change in joint strength reflects the wood degradation rate.
- Statistical variability creates unusual and confusing patterns of strength versus time (Figure 5c).

The strength test is, of course, destructive. The strength of one or a group of specimens cannot be followed sequentially through the entire aging exposure. Instead, separate groups of specimens must be exposed and tested at several different aging times. This introduces variability which can be confused with one of the previous factors.

- The aging mechanism may change with temperature (Figure 5d). Just as the visible degradation rate may change with time, the visible degradation mechanism may change with temperature. Thus, the temperature dependence of strength loss may be different at the elevated accelerated aging temperatures and in the interval between...
those temperatures and the lower service temperature. Extrapolation of the shear strength half-life relationship from the aging temperatures to the service temperature would be misleading.

The precision and accuracy of the extrapolated service life estimates are unknown (Figure 5e). The Arrhenius temperature dependence relationship is based on a series of estimates. Rigorous statistical procedures for determining the precision (confidence limits) about the estimated service life are not available. Furthermore, if the adhesive is very durable, the accuracy of the service life estimate cannot be determined from actual exposure because degradation is so slow.

Accuracy, of course, can only be verified by actual experience; however, the estimated half-lives of solid wood specimens in several studies at FPL (8-10) have shown reasonable agreement with the limited published information about the strength of very old wood samples (11).

Results of this study, particularly the wet aging results with adhesives A, C, and K, support the accuracy of the estimates derived from the accelerated aging tests. Rate-process aging analysis is not easy and does not provide a "guaranteed" service life. The analyst, using every bit of mechanical, chemical, and physical information and the best statistical techniques, still must doubt the service life estimate. The experience with adhesives E and K in this study punctuates the need for caution against blind acceptance of estimates derived from accelerated aging tests. But as we add to our knowledge of adhesive joint aging behavior, interpretation of results and recognition of erroneous estimates will become easier.

The half-life estimates derived from this study, the durable performance of two adhesives (A and C), have been qualitatively confirmed through their application in a test structure. In 1968, an experimental adhesive bonded house was built on FPL grounds (12), making extensive use of adhesive C in the exterior wall construction and adhesive A in a unique roof plank construction. Both adhesives have thus been exposed to exterior temperature conditions and fluctuations in humidity for more than 15 years. A recent inspection of the house and joints of each adhesive revealed no adhesives failures, nor was any reason found to suspect a precipitous decrease in the adhesives' performance.

Summary

Estimates of the time required for joints of four elastomer-based mastic-type construction adhesives to lose half of their original strength were made from rate-process analysis of accelerated aging data. The estimates were compared to actual performance of the adhesives over an 11-year period in three service conditions. Good support for the accuracy of the accelerated aging estimates was shown by real-time aging for three of the four adhesives in wet exposures. Aging in a 30% RH environment at 27°C showed very little degradation, but this in itself supported the long-life estimates obtained from dry accelerated aging.

Experience with a number of construction adhesives exposed to accelerated aging conditions in the laboratory and out of doors has revealed a broad spectrum of performance. Of the four adhesives extensively evaluated in this study, a flexible polyurethane was generally the most durable. A Neoprene-phenolic and a styrene-butadiene performed about equally as well as each other but were slightly less durable than the polyurethane. An adhesive based on fortified reclaim rubber was the least durable, particularly in the presence of moisture, in all the studies at the Laboratory. Broad differences in durability performance among the same base-elastomer types have also been noted. These differences are apparently related to other ingredients in the adhesives and to the method of preparing the adhesive and the joint.

Conclusion

Rate process analysis of accelerated aging data can be a powerful tool for evaluating durability. The analysis is not always simple and straightforward; caution and experience are required. Service life estimates from rate process analysis of accelerated aging data agreed with estimates from service exposures in three of four adhesives tested.

Based on both the accelerated and service aging exposures, well-formulated polyurethane, Neoprene-phenolic,
and styrene-butadiene construction adhesives may be expected to provide many years of satisfactory performance in building applications.

References