COMPARISONS OF INTERLABORATORY SWELLOMETER TESTING OF TWO WATER-REPELLENT PRESERVATIVE FORMULATIONS FOR MILLWORK

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ABSTRACT

Water-repellency of preservative formulations used in the millwork industry has long been evaluated by measurement of the dimensional changes in wood treated and then submerged in water according to guidelines published by the millwork industry. Perceptions that this "swellometer" test was highly variable led to a round-robin test of one solvent-borne and one waterborne commercial formulation by five independent laboratories in order to assess the variation in results among labs. These data were then used to address the sample size needed to achieve a 90 percent confidence level in the mean water-repellent value. The standard deviation for both formulations was near 25 percent of the mean for all labs. The range in average water-repellent values from the labs was approximately 10 percentage units. Not surprisingly, parent boards for swellometer samples were the main source of variation. The data analysis suggested that 12 (for waterborne) or 18 (for solvent-borne) individual test wafers would be needed to provide a 90 percent confidence that an average result from any single lab would be within 5 percentage units of an overall average value from five independent labs using the same sample materials. The new Window and Door Manufacturers' Association standard reflects this allowance by establishing the lower limit of water repellency at 55 percent.

Experience has indicated that wood windows and doors (millwork) have extended service life when dip treated with water-repellent preservatives (WRP) (4, 7-9). Both solvent-borne and waterborne formulations are currently used for millwork treatment following guidelines of the Window and Door Manufacturers' Association (WDMA), formerly the National Wood Window and Door Association (NWWDA). The effectiveness of the water-repellency in a given treatment is determined by a test method (the "swellometer" test [5]) that uses dimensional change of treated wood wafers after water submersion. The water-repellency percentage value (WRV) is calculated by dividing the difference between swelling of the treated wafer and the matching untreated control specimen by swelling of the untreated specimen and multiplying by 100. Historically, a value of 60 percent has been the minimum accepted test average. Evaluations of window units exposed outdoors have shown that initial and long-term performance of WRP-treated units are better with initial formu-

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TABLE 1. —Mean water-repellent percentage values for millwork WRP formulations from five independent laboratories using the current swellometer standard.[a]

Laboratory no.	Solvent-borne WRP	Waterborne WRP
1	35.6 A	51.3 AB
2	52.0 C	54.3 A
3	53.6 C	42.5 B
4	44.6 B	45.7 AB
5	47.7 BC	53.0 A
Average	46.7	49.3

[a] Tukey (HSD) at P = 95 percent (STATISTIX 4.1); n = 12 wafer comparisons/mean. Means in each column followed by the same capital letter are not statistically different.

lations with WRV values at 60 or above (2,4,9).

The testing of WRP formulations has followed the WDMA guide (3, which advises testing two treated wafers (with two matched untreated wafers) from each of five different parent boards (with the formulation WRV average therefore based on pooling of 10 wafer comparisons). Testing labs using this method have complained that the test is highly variable in providing results for a given formulation, with repeated tests often giving inconsistent results (within or among laboratories). This drawback to the method has been known since early work using the swellograph method for determining water repellency (1). This variability is also acknowledged in the water-repellency requirements of the wider specification standard (6), which notes that a formulation WRV value of 50 percent permits two retests to arrive at an overall average of WRV (three test average) of 60 percent in order to pass. A round-robin test was therefore proposed by WDMA to better understand the variation in test results from five independent laboratories experienced in using the swellometer test. Once the variation among laboratories was assessed (given a common source of wood wafers and formulations for testing), the data were used to develop better guidelines for sample numbers and a confidence level for the mean WRV of both a waterborne and solvent-borne WRP formulation.

MATERIALS AND METHODS

Four parent boards of ponderosa pine (*Pinus ponderosa* Laws.) sapwood were selected according to the existing test guideline (5) and three test wafers (6.4 by 25.4 by 127 mm: longitudinal by radial by tangential directions) along with three matching control wafers were cut from each to provide a set of 12 wafers for treatment by each participating laboratory with a commercial waterborne WRP. An additional set of wafers was also provided for treatment with a solvent (mineral spirits)-borne formulation. This sample scheme was a compromise dictated by the resource limits of available testing participants with a reduction in parent boards from five (as suggested by the standard) to four, but with increased subsampling from each board increased from two to three to allow more degrees of freedom for analyses.

Sets of wafers and WRP formulations were sent to five laboratories with experience in conducting the swellometer test. Results were returned and analyzed for comparisons of mean WRV by laboratory for each WRP type. A comparison of parent boards averaged over all labs was also done using standard analyses of variance methods (STATISTIX 4.1; Analytical Software, Tallahassee, Florida). Wilk-Shapiro/Rankit plots of WRV (to determine if the WRV variable conformed to a normal distribution) as well as regression residual plots of data were performed to validate use of the statistical models used to generate the analyses of variance. The variance data were then used to arrive at suggested sample sizes for estimation of a 90 percent confidence level for mean WRV determination following standard statistical methodology (3).

RESULTS AND DISCUSSION

SOVENT-BORNE FORMULATION

The WRV values of the formulations tested are in **Table 1.** For the solvent-borne formulation, the mean WRV values could be separated into three groups when compared at the 95 percent confidence level. The results from lab 1 were considered anomalous in the degree of variance from the general results (standard deviation at 42 percent of the mean). Also, these tests were conducted several months after other laboratory testing, possibly increasing experimental error due to wood surface changes. The five lab average was 46.7 percent WRV (below the expected result for standard approval or retesting). The standard deviation value for all labs combined was 12.8 units, providing a coefficient of variation (COV) of 27.4 percent.

Sample size calculations to assure confidence in results from a single laboratory trial were estimated based on common statistical inference (3). If the anomalous results from lab 1 are excluded, the range from highest to lowest average WRV value was nine percentage units. It would therefore seem reasonable to assume the "maximum error of the estimate" of an average to be + or - five WRV percentage units (D). If the assumption that the mean + or - D contains the true mean of repeated trials with the same materials at other competent labs and we wish to observe a 90 percent confidence level for that mean, the sample size (N = number of wafers with pooled parent board variance) can be found by the following formula (3):

$$N = (Z \times S)^2/D^2$$

where Z = 1.645 for P = 90% confidence; S = the standard deviation for all data. Application of this formula provides the estimate of 18 wood wafers required to give a 90 percent confidence that the mean WRV from a given lab would be within 5 WRV units of the mean obtained from a result of 5 labs running the same test with the same sample materials.

WATERBORNE FORMULATION

The interlaboratory average WRV for the waterborne formulation was 49.3 percent **(Table 1).** The means from the labs form two groups that are not significantly different and the standard deviation was 10.9 giving a COV of 22 percent (less variation than for the solvent-borne system). Again, the range from high to low average was approximately 10 WRV units. Application of the formula for sample number determination for a 90 percent confidence value in the mean suggests that 13 wafers would be needed in testing. For both systems, a Wilk-Shapiro/Rankit plot of WRV values from all five labs shows approximation of a line (0.99), and the regression residual plots show the residuals are not grouped. These tests supporting

TABLE 2.—Mean water-repellent values from all five testing labs by parent board (three samples/each of four parent boards/ testing lab).

Board no.	Solvent-borne WRP	Waterborne WRP
1	61.8 A	58.0 A
2	43.6 B	44.9 B
3	40.9 B	41.8 B
4	40.6 B	46.6 B

[a] Means in each column (n = 60) followed by different capital letters are statistically different (Tukey (HSD) P = 95 percent; STATISTIX 4.1).

normal distribution of data confirm that the model used to generate the analyses of variance was valid (including the choice dictated by the standard (5) to pool data from different parent boards to arrive at an overall average for WRV).

PARENT BOARD ANALYSIS

It is realized that wood is a variable material and, therefore, the swellometer standard (5) dictates use of a number of parent boards to try to represent such variation in potential performance of a water-repellent formulation (within a narrow wood specific gravity range). The comparisons of parent board data (including all five labs) for solvent-borne and waterborne systems are shown in **Table 2.** It is clear from the pooled data that parent board 1 is much more responsive to the WRP treatment irrespective of carrier solvent, and represents the most contribution to variation of the data means. Additionally, the other three parent boards likewise are grouped as to WRV response to the WRP treatments when averaged over results from the five labs. This is in agreement with early work (1) that noted variability in replicate wafer water-repellency was primarily due to the differences among individual parent boards, and is not simply related to any easily recognized property such as specific gravity or ring count. Unfortunately, one water-repellent formulation may perform well on one board, while a second formulation may perform better on a different parent board included as a wafer source. For this reason, it is not considered advisable to select all specimens for testing from a single board even though it would reduce variability among replicates (1).

CONCLUSIONS AND COMMENTS

In summary, these data demonstrate that the basic testing of WRP formulations by different laboratories can be achieved to provide a guideline for acceptance of results based on current swellometer methodology. If adequate sample numbers are provided from at least four parent boards, and the COV of the data does not exceed 25 percent, any given lab test average should be within five WRV units of the true mean that multiple labs would derive (given a 90% level of confidence). This approach does not abandon the underlying support for the average WRV level of 60 percent established for millwork in prior work, but merely allows a reasonable margin of estimate of five WRV units as error derived from experimental interlaboratory variation, and is so reflected in the most current standard (10).

A prudent approach to accepting the variability in the current water-repellency test would be to include more than one set of test data for determination of acceptance. A single test with a 55 WRV does not indicate whether the result is at the lower acceptable limit of an average value of 60 percent or the upper limit of a value of 50 percent as a mean obtained by multiple laboratory testing. Also, a single lab result (as noted for lab 1 and solvent-borne systems **[Table 1]**) may vary more than the assumed + or - five WRV units about the generally accepted mean from multiple tests. The COV of 42 percent would invalidate consideration of these test data. To be more certain of the meaning of any single trial of water-repellent formulation, confirmatory tests should be conducted to be sure that the general average is near the expected mean of 60 + or - 5 percent.

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