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ANALYSIS OF DATA OBTAINED IN THE COURSE OF A PROGRAM TO DEVELOP A WATER REPELLENCY FORMULATION FOR TREATING MILLWORK

Ron Bobker
Everdry Forest Products Ltd
226 Randall St. Oakville ON L6J 1P7

Abstract

A screening method was set up to enable candidate water repellency formulations to be tested. Once promising formulations had been identified, they could then be tested under more rigorous conditions. The procedure adopted involved testing wood treated with a new formulation against untreated wood, and wood treated with a standard formulation. From the large amount of data generated it became clear that there was a great deal of variability in the standard method of comparing the swelling of a treated wood wafer with an end grain matched wafer of untreated wood. There appears to be much less variability in the comparison between wood treated with a standard formulation, and with a test formulation. There appears to be no correlation between the uptake of treatment fluid and water repellency within the range tested. A parameter designated the Coefficient of Improvement has been developed to compare the swelling of wood treated with a known, or standard formulation and wood treated with a developmental formulation. A formulation was identified that gave very good results when tested in accordance with a rigorous test procedure: WDMA Test Method 2.99

1. Introduction

The results described in this paper are based on an R & D program undertaken by Everdry Forest Products Ltd to develop an improved water repellency formulation for wood preservative products used to treat wood used in the millwork (i.e. windows and doors) industry. As this industry normally uses formulations dissolved in a light oil solvent, the work undertaken focussed on solvent based as opposed to water based systems.

Standards for wood treatment

Although there is not, in Canada, any formal requirement to treat wood, companies that wish to conform to the CSA A440 standard for windows are required to treat wood components with a water repellent preservative. CSA has not established its own standards for wood treatment; instead it uses the standards established by US Window and Door Manufacturers' Association, WDMA. The water repellency performance and its method of determination is given in the WDMA test method T.M. 2-99, which states that a treatment must confer a minimum water repellency efficiency of 55% when tested in the prescribed manner. The criterion of water repellency efficiency refers to the efficiency of the treatment in controlling swelling of a treated wood wafer compared with the swelling of an end-grain matched untreated wafer, the swelling being measured in a direction tangential to the annual rings. It is important that the wafers are prepared from flat sawn sapwood, with the grain oriented appropriately. The water repellency[1] is evaluated using the equation:

$$\mathbf{WRE} = \frac{\mathbf{U_t - T_t}}{\mathbf{U_t}} \times \mathbf{100}$$

Where: WRE is the water repellency efficiency expressed as a percentage,

U_t = % swelling of the untreated wafer at time t, and:

T_t = % swelling of the treated wafer at time t

Procedure adopted by Everdry in measuring water repellency

Although WDMA T.M. 2-99 is the standard procedure to be used for judging whether or not a formulation meets the Standard, implementing this method requires, ideally, 125mm long wafers of flat sawn Ponderosa Pine sapwood which has to be conditioned in a controlled temperature-humidity atmosphere prior to testing. It is difficult to obtain Ponderosa Pine in the correct configuration, and the preparation and measuring processes are time consuming, and need special equipment. Accordingly Everdry developed a screening method for evaluating water repellency in a less rigorous manner, but which allows for rapid screening different formulations to be compared with each other and with a standard formulation. Once promising formulations

[1] Although the measured parameter is referred to as "water repellency" it could be more accurately described as "swell resistance". A treatment that effectively controls swelling wood from swelling may, or may not correlate with the ability of the surface of the treated wood to shed water from its surface.

were found, they were tested more-or-less in accordance with the WDMA T.M. 2-99 method[2] at the University of Toronto, Faculty of Forestry Laboratories.

2. Methodology

Preparation of wood

As it is virtually impossible to find pine sapwood planks with the correct grain orientation in a lumber yard, it was necessary to acquire pine logs at a sawmill and cut them appropriately. This meant testing the logs with a before cutting to identify the sapwood zone, which is very difficult to detect visually.

Steps involved:
Red Pine and White Pine logs were selected at a sawmill.
Logs were tested to detect boundary of sapwood and heartwood[3] (Fig 2).
Planks were cut from the sapwood of the log (Fig 3).
Planks kiln-dried to MC (moisture content) < 25%
Planks cut to a suitable size - any undesirable portions (bark and knots) were removed (Fig 4).
Wafers were sliced from planks with a band saw (a typical wafer is shown in Fig 5.

Preparation of test wafers

The trimmed plank was given an identification code, and then was sawn into 7mm thick wafers with a band saw. Wafers were cut into sets of three, and marked A, B and C, together with other information identifying the plank. The A wafer was treated with a test formulation, the B served as an untreated control, and the C wafer was treated with a standard water repellency formulation. In all cases the B wafer was the middle of the set of three, and was therefore end-grain matched with both the A and the C wafers.

Treating and measuring procedure

Five sets of A, B and C wafers were selected for each test. In some of the earlier tests all five sets came from the same plank; in later tests sets were taken from different planks.
The moisture content of the wafers was measured with a resistance type moisture meter; moisture contents were typically < 5%

[2] Because it is very difficult to obtain flat sawn Ponderosa Pine sapwood, wafers from Southern Yellow Pine sapwood were used instead.

[3] The reagent is a 50-50 mixture of sodium nitrite and o-anisidine hydrochloride. The two solutions are mixed and used within 30 minutes. The reagent turns bright red within a few minutes after coming into contact with pine heartwood.

The wafers were treated by being dipped for 30s in the appropriate solvent solution (A in new formulation, C in standard formulation).

Wafers were spread out on an open mesh and the solvent allowed to evaporate off for 7 days; the B samples were also arranged on the mesh, even though they were not treated. The conditioning was in the laboratory under ambient conditions – approximately 22°C. Humidity was not measured, but probably never went above 75%.

Wafers were re-weighed to determine the amount of non-volatiles left in the wood.

Wafer length was measured prior to the wafer being immersed in distilled water to give initial lengths, i.e. at time, t = 0: A_0, B_0 and C_0. A calliper with the capability of measuring to ±0.01mm was used; see Figure 5: note that the length of the wafer is parallel, i.e. tangential to the annual rings.

Wafers were immersed in distilled water[4], for a timed period of 5 minutes, whereupon they were removed from the water, the ends wiped with a paper towel and the length measured; a period of 60s was allowed to remove the wafer, wipe the ends measure and record the length then replace the wafer back in its immersion bath.

The total immersion time was 45 minutes so a total of 9 readings were taken per sample.

The results were entered into a spreadsheet (Microsoft Excel) which was set up to calculate the percent swelling and the WRE. Results were calculated for each individual wafer, and also the mean values of the five sets that were measured. Figures 6 and 7 show the results of the means, and the resulting graphs (time versus % swelling, and time versus WRE).

Once the measurements were completed the wafers were allowed to dry out, and were stored so that subsequent swell measurements could be performed, to estimate the permanence of the treatment.

[4] Wafers were immersed, each in its own bath, at one minute intervals.

FIGURES SHOWING PREPARATION OF WOOD SAMPLES FOR TESTING

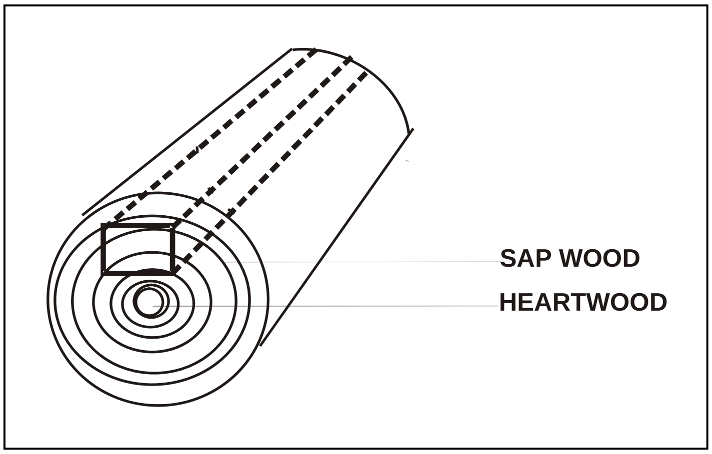

Fig. 1 Schematic of a log indicating the cuts required to obtain a plank with the correct grain orientation for swell testing

Fig. 2 Detecting sapwood boundary

Fig. 3 Cutting flat sawn planks of sapwood

Fig. 4 Preparing kiln dried wood into planks

Fig. 5 Typical wafer

3. Results and Discussions

Figure 6 shows typical results of swell test measurement, and Figure 7 is the corresponding water repellency efficiency calculated using Equation 1: note that these results are the mean of the five wafer samples measured. A total of 116 sets of measurements were performed, using a wide variety of test formulations. This means that a total of 580 A, B and C sets were prepared, and a total of 1740 wafers were cut. Of this 116 there were several repeats once promising formulations were found, to obtain confidence that the results were real, and not due to some unknown random effect or event.

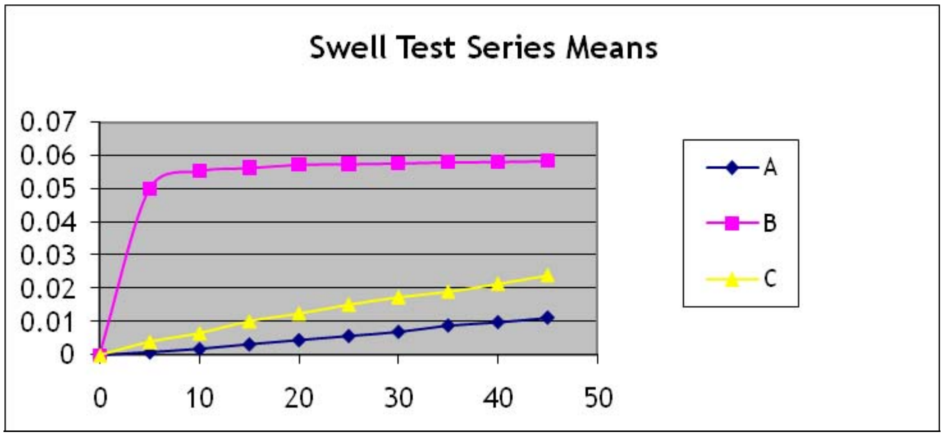

Figure 6: Typical swell test results – raw data, swelling (as a fraction) vs. time in minutes

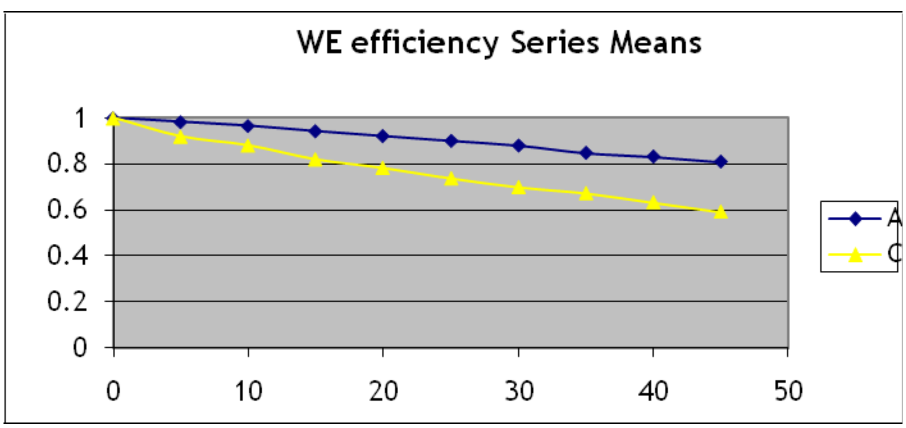

Figure 7: Water repellency efficiencies based on the swell data shown in Figure 6

Discussion of swelling and WR results

It will be noted that the untreated wood samples swelled very rapidly, so that within 5 minutes they reached over 80% of the ultimate swollen length. The amount they swelled varied from wood sample to wood sample, from a low of 3% to a high of 7%. It is also apparent that the water repellent treatment had a significant effect upon the rate of swelling, so the method is an effective means for determining the effectiveness of a treatment. It will also be noted that the method also allows for the comparison of two different water repellency treatments or formulations, particularly after an immersion time of 30 minutes or more.

Statistical analysis of the results

The swell measurements on the individual wafers were mostly quite well defined, the data points all lie on a well defined line. Initially it was thought that the consistency of the results meant that a reliable value for WRE could be obtained from a limited number of measurements. However when the results for 286 measurements on C wafers were examined, it is apparent that there is a wide spread in the values, which means that a lot of measurements would be necessary to make a judgement as to what the actual value is. This distribution is shown in Figure 8:

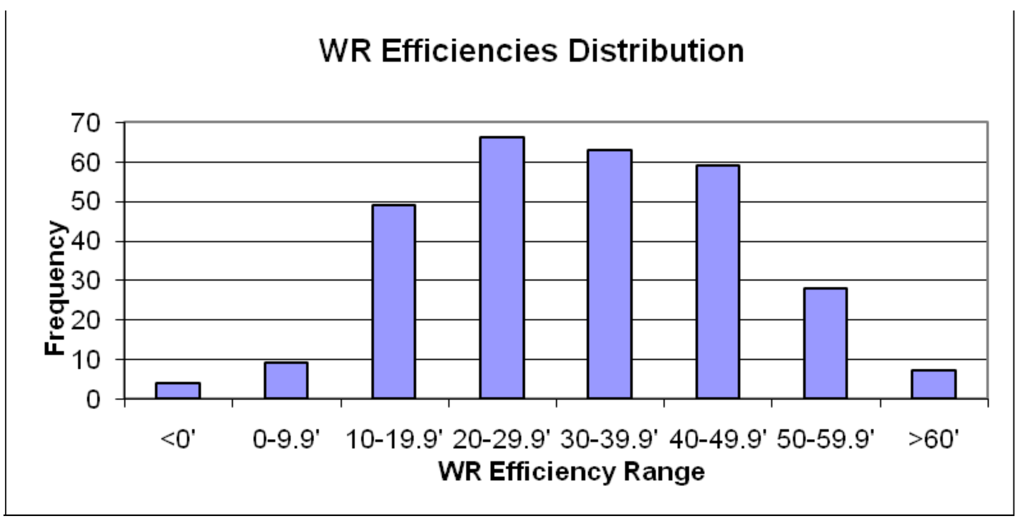

Figure 8: Distribution of water repellency measurements for C wafers (treated with the standard formulation)

No. of samples =286, Mean = 33.65, Standard Deviation = 22.273

The distribution was tested for normalcy using the Jarque-Bera test:

$$JB = \frac{n}{6}\left(S^2 + \frac{(K-3)^2}{4}\right)$$

Where: JB = Jarque-Bera test statistic

n = degrees of freedom
S[5]=skew of data, and
K= kurtosis of data

The JB statistic is compared with the critical X^2 (chi squared) value = 5.991. The distribution can be considered normal if $JB < X^2$. In this instance, JB = 0.673628 hence it can be treated as "normal".The distribution of the swelling of the B (untreated) wafers was examined[6]:

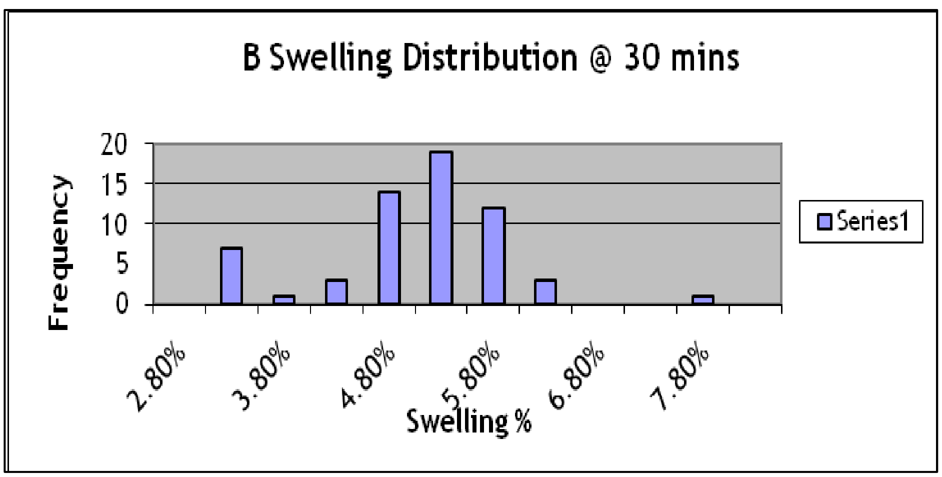

Figure 9: Swelling of untreated wafers after 30 minutes immersion:

Mean = 4.84%, Standard deviation = 0.9%, JB = 3.51 therefore "normal"

[5] The values for S and K are obtained directly from the Excel worksheet, where kurtosis is calculated as K-3
[6] Not for the same sets of measurements as those from which the results shown in Figure 8 were obtained

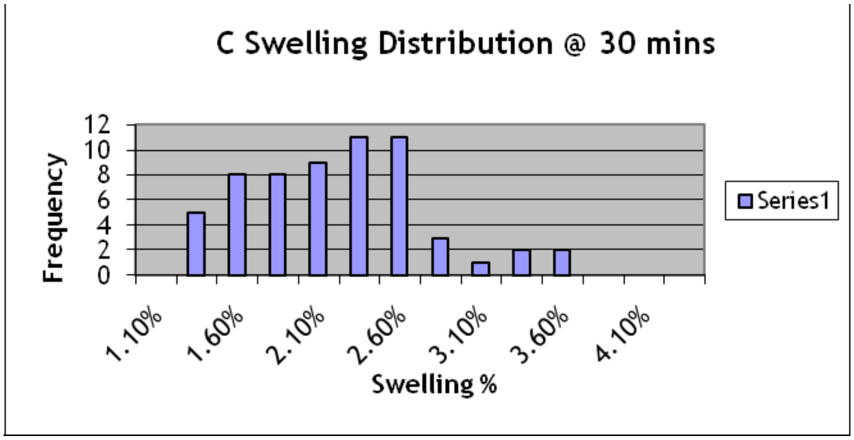

Figure 10: swelling of wafers treated with standard formulation after 30 minutes immersion

Mean = 2.10%, Standard deviation = 0.54%, JB = 1.65 therefore "normal"

Rank correlation of different parameters

It is of interest to speculate whether there is any correlation between the treated wafers (either A or C) and the untreated, B-wafers. In other words is there a negative or positive correlation between the ways in which a WR formulation affects the swelling of a treated piece of wood compared with the way in which the untreated wood swells. The method selected for this was the Kendall Tau correlation coefficient, for which a web-based calculator can be used[7]. For perfect positive correlation, tau = +1 and for a perfect negative correlation the value is -1. A value close to zero indicates no correlation.

Table 1: Correlation between various parameters

Parameters compared		Kendall's Tau
C swelling	B swelling	0.349
C swelling	Retention of treatment liquid	-0.0133
Index of Improvement	C swelling	-0.301

[7] Wessa, (2008), Kendall tau Rank Correlation (v1.0.10) in Free Statistics Software (v1.1.23-r3), Office for Research Development and Education, URL http://www.wessa.net/rwasp_kendall.wasp/

The values of Kendall's Tau indicate there is no correlation between the retention of treatment liquid and the amount the wood swells, and very little correlation between C swelling and B swelling. Before undertaking this analysis there could be an expectation that higher loadings of treatment in a sample would give better resistance to swelling; clearly this is not the case. There is also very little correlation between the degree of swelling of the untreated wood and the effect of the treatment. The index of improvement is a parameter that compares the improvement in swell resistance by a change in formulation. This is discussed in more detail below.

Coefficient of Improvement

Because the water repellency as defined by Equation 1 appears to be a parameter that is subject to much variation, so that a measured value does not give much information. WRE compares the treated wood with untreated wood; however the way in which wood swells, as depicted in Figure 6 indicates that the swelling of the treated and untreated wood is not really comparing the same process. It is suggested therefore that instead of comparing treated with untreated, a more meaningful parameter is to compare the swelling of a wafer treated with the standard formulation the swelling of a wafer treated with a test formulation. A coefficient of Improvement, CoI can be defined as:

$$\mathrm{CoI} = 1 - \frac{C_0\,(A_t - A_0)}{A_0\,(C_t - C_0)}$$

C_t = Length of the C wafer at time t

A_0 = Length of the A water at time t=0

A_t = Length of the A water at time t

It was found that the CoI gave reasonably consistent results as shown in Table 2.

Run #	Composition	Coeff. of Improvement at 30 minutes	WRE C wafers at 30 minutes
77	755	-28.1%	64.5%
79	755	23.6%	57.4%
106	755	27.3%	47.9%
114	755	7.2%	66.2%
116	755	4.8%	61.5%
117	755	9.4%	62.7%

Table 2:Coefficient of Improvement for formulation # 755

In 5 out of the six runs, formulation #755 was more effective in preventing swelling than was the standard formulation. It is not clear why Run # 77 showed the reverse effect. However, Formulation 755 was tested at the University of Toronto, in accordance with WDMA T.M. 2.99, the test being carried out in parallel with wafers treated with the standard formulation (C – series results). The results are shown in Figure 10

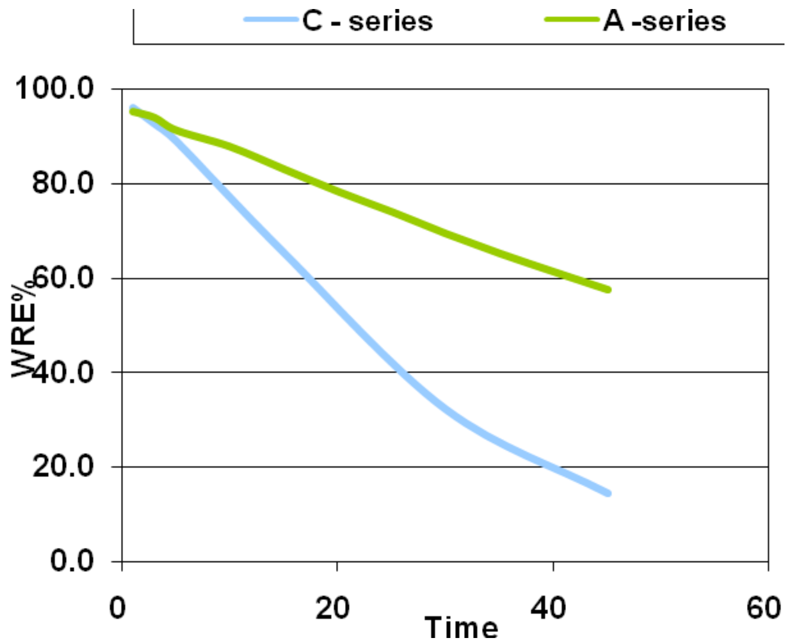

Figure 10: WDMA T.M. 2.99 results for wafers treated with the standard formulation (C-series) and with formulation #755. A-series: wood treated with formulation #755 C-series: standard formulation. Work done at the Faculty of Forestry laboratories at the University of Toronto.

Summary of Findings

Untreated pine sapwood swells quite rapidly in a direction tangential to the annual rings when it is immersed in water; within five minutes it can swell by as much as 5%. It reaches its ultimate swollen dimension within 20 minutes

Wood treated with an effective water repellent swells much more slowly, and will take several hours to reach the same degree of swelling as an untreated piece of wood.

Water repellent efficiency, WRE, is a comparison of the swelling of an untreated piece of wood and that of an end-grain matched sample of wood that has been treated. A WRE value of 100% would indicate zero swelling in the specified time (30 minutes). There is a good deal of variation in wood swelling, and the WRE value as determined in this investigation is reproducible to the extent that the values could be used with confidence.

There appears to be no correlation between the swelling of treated wood and end-grain matched untreated wood, nor is there any correlation between the swelling of treated wood and the amount of water repellent compounds it contains.

A better parameter for determining the effectiveness of a treatment is the coefficient of improvement, CoI, which essentially compares the swelling of a piece of wood treated with a standard, or control formulation, and a test formulation. A zero value for CoI indicates that the wood treated with the test formulation swells precisely the same as the wood treated with the standard. Any value > 0 indicates an improvement. A promising test formulation was found which, in the majority of tests, gave a CoI > 0. Subsequent testing carried out at the laboratories of the Faculty of Forestry at the University of Toronto showed that this test formulation had a WRE of over 70% at 30 minutes.

An effective formulation was developed which proved to have a high WR rating when measured in accordance with WDMA T.M. 2.99.

4. Conclusions

Pine sapwood swells when it comes into contact with water, and the rate at which it swells is fairly uniform: most samples of wood tested reached over 80% of their ultimate swelling within 5 minutes of being immersed. The ultimate swelling reached within 45 minutes varied from about 3% to 7%.

A water repellent slows down the rate at which wood swells; with very effective water repellents the change of length in the tangential direction with time is virtually linear over the range 5 – 45 minutes.

There is a wide variation in the measured values of water repellency. WRE values at 30 minutes varied from -5% to > 60% for wood treated with a standard formulation.

WRE compares the swelling between end-grain-matched treated and untreated pine sapwood wafers. The large variation observed in the calculated values of the WREs is very likely due to the different modes of swelling of the treated and untreated wood samples.

More consistent results are obtained when wood treated with a test formulation is compared with wood treated with a standard formulation, probably because the mode of swelling is more similar.

It is proposed that a future standard for evaluating water repellency should be based upon the comparison of wood treated with a test solution and wood treated with some standard formulation.

Once a standard formulation has been decided upon, it is suggested that the term “coefficient of improvement” should be used for defining the effectiveness of a water repellent formulation.The protocol described here is a useful method for screening water repellency formulations.