# PEG PENETRATION IN THREE COMMERCIALLY IMPORTANT TASMANIAN EUCALYPT SPECIES

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ABSTRACT

Commercially important species of Tasmanian hardwood timber were immersed in 30% (v/v) polyethylene glycol (PEG) of molecular weights 400, 600, and 1000 and incubated up to seven days at three temperatures (30ºC, 45 ºC, 60ºC). Slices obtained from the incubated timber samples were stained with cobalt thiocyanate to indicate the depth of penetration by PEG 400, 600, or 1000 after incubation from two to seven days at the various temperatures. Analysis of the data showed that there was an observable difference in the rate of penetration between each species of eucalypt used in the trial. Incubation time, temperature, and PEG molecular weight were all factors affecting the rate of PEG penetration in a linear fashion and basic density (BD) was the physical property that best supported the trends in this study. This paper is a baseline study that provides the foundation for the quantification and prediction of the movement of PEG into three species of Tasmanian eucalypt timber.

*Keywords:* Eucalypt, polyethylene glycol, PEG, drying stresses, basic density.

## INTRODUCTION

Polyethylene glycol (PEG) has been used as a method of relieving drying stresses in timber since the British Patent of the work by Morén and Centerwall (Mo och Domsjö Aktiebolag 1952) and the findings of Stamm (1956). The chemical has been found to successfully reduce drying stress in many species throughout the world including (first appearances) Sweden (Mo och Domsjö Aktiebolag 1952), United States of America (Stamm 1956), Canada (Cech 1968), Germany (Schneider 1969), Australia (MacKay 1972), Latvia (Kreicuma and Svalbe 1972), China (Lo 1974), Japan (Ishimaru 1976) Korea (Hoffmann 1990) and Turkey (Alma et al., 1996).

Although PEG is successful in preventing checks and permanent deformation of timber that can occur during drying, the cost of the chemical made the process uneconomical, despite the increase in timber degrade in its absence. However, as more trees are harvested at earlier ages from plantations, there may be a need to reinvestigate chemical methods of aiding the drying of timber. The drying schedules used to successfully kiln-dry old growth timber are simply too harsh for regrowth timber, and drying defects in the form of surface and internal checks are a common malady. Furthermore, younger trees have smaller trunk diameters, and in order to saw boards of greater width, the log must be backsawn. Backsawn (aka flatsawn) timber has the largest face from the tangential aspect of the tree: the orientation most susceptible to shrinkage and drying defects owing to differential drying rates.

With these obstacles to navigate, there may be a justification in the added expense of a chemical pretreatment to alleviate drying stresses in regrowth timber. Industrial timber treatments such

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as fungicides and pesticides are common, which suggests that it is possible for a business to incur the added expense of a chemical treatment regime and still be economically viable. This would particularly be the case if the mechanism of a known successful method, such as PEG, can be determined and then later replicated with inexpensive analogues. In an industrial setting, total impregnation of timber is neither required nor desired, as it would be intended that the chemical be removed from the timber before sale. Consequently, the thinnest coating that can provide optimum assistance to the kiln-drying of timber would be the ultimate goal. The starting point was to investigate penetration rates of PEGs in three commercially important hardwood species in Tasmania.

## METHOD

### *Preparation of samples*

Samples of *E.delegatensis, E. obliqua,* and *E. regnans,* were cut at a commercial sawmill into dimensions of 25 × 35 × 300 mm "sticks" in the backsawn (tangential) orientation and immediately wrapped in clingwrap plastic to maintain the moisture content. One stick from each species was selected for each combination of the following parameters: a sample incubated from 2 to 7 days in 30% (v/v) solutions of PEG 400, 600, and 1000 at temperatures of 30ºC, 45ºC, and 60ºC for a total of nine sticks.

Preliminary experiments confirmed that greatest absorption of PEG occurred through the timber end-grain, particularly in the latewood, and this was independent of the proportion of latewood to earlywood. Consequently, all samples to be treated with PEG were first end-sealed with 2-part epoxy adhesive (araldite).

Eighteen samples (6 from each stick and species) were placed in a sealable container and then filled with a 30% PEG solution (MW 400, 600, 1000, Union Carbide Corp.) and placed in a water bath at the desired temperature (30ºC, 45ºC, 60ºC). A seventh sample of 40 mm was cut from the stick and oven-dried to determine the initial moisture content of the timber. From 2 to 7 days, a sample from each species was removed from each container.

### *Cutting sections*

As PEG is water-soluble, it is important that the samples be exposed to atmospheric moisture for as little time as possible to remove the possibility of displacing PEG by added hydration of the timber. Following incubation, the samples were removed and padded dry to remove excess PEG solution. A purpose-built, microtome-like, slicer was used to cut three 2 mm-thick sections from each sample. The sections were placed immediately in a 105ºC oven for 24 h to allow complete drying and then placed in the staining solution.

### *Staining solution*

Van der Hoeve (1948) adapted Gnamm's (1943) ammonium cobalt thiocyanate method for the qualitative *in vitro* testing of alcohols, ketones, and esters to produce a subsidiary test for compounds involving polyethylene oxide. Young and Wainwright (1981) stained untreated samples and samples treated with PEGs 400 or 3350 with cobalt thiocyanate to successfully view the difference in penetration by PEGs of significantly different molecular size. Cobalt thiocyanate binds specifically to PEG and following incubation, clearing with xylene removes any free PEG and free cobalt thiocyanate. However, any PEG/cobalt thiocyanate complex remains *in situ* and appears the blue color of the cobalt thiocyanate compound. Positive and negative controls consisted of stained treated and untreated slices. After clearing with xylene, the positive control retained the blue color of the stain, while the negative control was its natural timber color.

### *Stain preparation*

The method largely followed that of Young and Wainwright (1981). Dry ammonium thiocyanate (0.2 g) is added to 0.2 g of cobalt nitrate hexahydrate and dissolved in 10 mL of octanol.

Once dissolved (although some residue is expected), the solution is then diluted in another 30 mL of octanol.

### *Visualizing penetration*

The oven-dried samples are placed in separate vessels of staining solution; the containers are sealed with clingwrap plastic and kept at 4ºC overnight. Following incubation, the samples were rinsed in three successive rounds of fresh xylene and then allowed to dry in a fume hood.

### *Measuring penetration*

Depth of penetration was measured using digital vernier callipers from the edge of the sample to the cessation of the blue color at four evenly-spaced points along the four sides of each sample slice. Therefore, for any one sample, incubated for any one length of time, temperature, and molecular weight, 48 data points (4 measurements × 4 sides × 3 slices) were taken. This large sample size was necessary to account for the inherent variability that can be found in timber, and multiple slices ensured that the penetration pattern did not occur by chance.

### *Analysis*

Average values were taken over the 48 data points for each combination of parameters, and the means and standard errors were calculated. Confidence intervals (C.I.) were calculated based on the number of data points using Eqs. (1) and (2) with the desired C.I. established at 95% ($\alpha = 0.05$). This was to determine that the sample size was adequate to produce statistically meaningful results.

$$CI = \left(t_{\alpha/2,n-1}\right)\frac{SD}{\sqrt{n}} \quad (1)$$

$$\%CI = \left(CI / \bar{x}\right) \times 100 \quad (2)$$

where: n = number of samples
$\bar{x}$ = average result

The average values for each set of parameters were plotted, a line of best fit was applied and an $R^2$ value calculated to determine the linearity of the data. The slope of the line was a measure of the rate of penetration, and it was the penetration rate, rather than distance, that was particularly in focus.

Repeated measures ANOVA were performed using the generalized linear model command with case clustering and robust standard error estimation (Stata 8.2, StataCorp, College Station, Texas 77845 USA). The maximum likelihood coefficient estimates were used to calculate predicted mean depth penetrance for the different exposure conditions (see Table 3).

## DISCUSSION

### *Suitability of the staining method*

The consistency of results showed that the adapted method of Young and Wainwright (1981) is suitable for the measurement of PEG in Tasmanian oak (the marketing term for 3 species of eucalypt; *E. delegatensis, E. regnans, E. obliqua*) up to at least MW of 1000. The blue color contrasted very well with the color of timber and allowed for easy measurements of each section. It would be anticipated that this method would be suitable for detecting PEG in other species of eucalypt.

### *Variability in results*

Error analysis showed that the high number of data points was able to overcome the natural variability found in timber. With the t-score ($t_{0.025,\ n-1=\ 47}$) obtained from a t-table, Eqs. (1) and (2) were used to predict whether the sample size was sufficient to return a desired confidence interval based on the variability in the data set.

Fifty-four values for %CI are presented in Table 1. Of these, 38 (70%) are above the 95% C. I. and of the 16 values beyond the C. I., seven are out by less than 1%, and the greatest departure from the set C. I. is only by 3.5%. These results are excellent and indicate that the data can be interpreted with confidence.

TABLE 1. *% confidence values (from Eq. 2) with confidence set at 95% for Day 2 and Day 7 for samples incubated in 30% (v/v) of PEG at molecular weights of 400, 600, and 1000 at temperatures of 30ºC, 45ºC, and 60ºC (Temp 30, 45, 60). A single letter indicates species:* E. delegatensis *(D),* E. obliqua *(O), and* E. regnans *(R).*

| | | 400 | | 600 | | 1000 | |
|---|---|---|---|---|---|---|---|
| Temp | Species | Day 2 | Day 7 | Day 2 | Day 7 | Day 2 | Day 7 |
| 30 | D | 92.06 | 93.41 | 95.65 | 97.60 | 94.59 | 96.67 |
| | O | 91.50 | 96.69 | 92.59 | 96.28 | 96.19 | 95.42 |
| | R | 95.32 | 94.19 | 94.03 | 93.15 | 97.54 | 95.31 |
| 45 | D | 96.37 | 96.44 | 93.97 | 96.63 | 94.61 | 98.02 |
| | O | 95.32 | 96.11 | 94.21 | 96.30 | 92.25 | 94.69 |
| | R | 94.33 | 93.29 | 97.39 | 96.66 | 93.73 | 96.93 |
| 60 | D | 97.26 | 98.31 | 97.38 | 98.06 | 96.83 | 98.55 |
| | O | 96.09 | 98.06 | 96.25 | 98.40 | 94.04 | 97.37 |
| | R | 97.37 | 96.93 | 96.80 | 97.35 | 95.09 | 97.80 |

### *Diffusion into the timber*

There were four parameters being considered with respect to PEG diffusion into the timber: incubation time, incubation temperature, PEG molecular weight, and difference between species.

### *Incubation time*

The average PEG penetration values for each parameter are shown in Table 2. In the greater majority of cases, the figures running across each row are additive, indicating that with time, the amount of penetration in each sample increased. This effect was independent of the treatment conditions or the species of timber used. Data from this table can be arranged to show the relationship between data sets for a given temperature, PEG molecular weight or to observe any differences between species.

### *Incubation temperature*

Figure 1 shows that temperature had a significant effect on the amount of PEG penetration. Although the pattern of movement into the timber is similar at each temperature, the total penetration distance is noticeably different as the temperature changes. This was expected since as more heat energy is applied to the system, there is greater kinetic energy emitted by the moving molecules and the molecules move faster in the direction dictated by the concentration gradient.

### *PEG molecular weight*

Figure 2 shows the differences in penetration rate of PEGs of differing molecular weight. In each case, the depth of PEG diffusion into the timber was inversely related to PEG molecular weight, i.e., $400<600<1000$. This was anticipated, as the larger and bulkier molecular weight species would have greater difficulty in navigating into cell spaces. Furthermore, with a larger charge, the heavier molecules will be incumbent with a larger solvent shell, and thus movement on a molecular basis may be impeded. There may be structural factors that impede some molecular weight PEGs but not others, and these will be investigated in the near future.

### *Species*

*E. delegatensis, E. obliqua,* and *E. regnans* are common commercially important timbers obtained from old-growth and regrowth forests in Tasmania. Since the 1860s, the three separate species have been marketed collectively as Tasmanian oak despite the fact that oak is of the species *Quercus.* During seasoning (either air-drying or kiln-drying), the species are mixed in the belief that their physical properties, specifically with respect to drying characteristics, are similar. The results of this study show that this view does not extend to the movement of PEG into the wood substance. Figure 3 shows that there is a clear difference in the amount of PEG

TABLE 2. *Average penetration distance (mm, n=48) of PEG for samples incubated in 30% (v/v) of PEG at molecular weights of 400, 600, and 1000 at temperatures of 30°C, 45°C, and 60°C (Temp 30, 45, 60) for between 2 and 7 days. A single letter indicates species:* E. delegatensis *(D),* E. obliqua *(O), and* E. regnans *(R).*

| | | | Incubation Period (Days) | | | | | |
|---|---|---|---|---|---|---|---|---|
| M.W. | Temp | Species | 2 | 3 | 4 | 5 | 6 | 7 |
| 400 | 30 | D | 0.80 | 1.05 | 1.30 | 1.60 | 1.70 | 2.06 |
| | | O | 0.44 | 0.74 | 0.96 | 1.24 | 1.40 | 1.52 |
| | | R | 1.13 | 1.33 | 1.70 | 1.83 | 2.00 | 2.27 |
| | 45 | D | 1.53 | 2.07 | 2.43 | 2.87 | 3.03 | 3.60 |
| | | O | 1.02 | 1.30 | 1.76 | 2.00 | 2.10 | 2.29 |
| | | R | 2.20 | 2.62 | 3.20 | 3.54 | 4.00 | 4.15 |
| | 60 | D | 1.83 | 2.40 | 3.00 | 3.80 | 4.72 | 4.97 |
| | | O | 1.00 | 1.28 | 1.60 | 2.34 | 2.68 | 3.23 |
| | | R | 2.68 | 3.32 | 3.87 | 4.77 | 5.50 | 5.62 |
| 600 | 30 | D | 0.69 | 0.83 | 1.19 | 1.51 | 1.79 | 1.89 |
| | | O | 0.36 | 0.64 | 0.74 | 1.06 | 1.15 | 1.42 |
| | | R | 0.93 | 1.15 | 1.29 | 1.62 | 1.86 | 2.09 |
| | 45 | D | 1.25 | 1.48 | 1.84 | 1.96 | 2.50 | 3.20 |
| | | O | 0.77 | 0.90 | 1.30 | 1.22 | 1.62 | 1.91 |
| | | R | 1.76 | 2.13 | 2.60 | 2.72 | 3.06 | 3.61 |
| | 60 | D | 1.57 | 1.83 | 2.40 | 2.89 | 3.66 | 4.27 |
| | | O | 0.89 | 1.32 | 1.40 | 1.87 | 2.17 | 2.79 |
| | | R | 2.00 | 2.64 | 3.32 | 3.53 | 4.13 | 5.01 |
| 1000 | 30 | D | 0.60 | 0.89 | 1.08 | 1.37 | 1.49 | 1.70 |
| | | O | 0.35 | 0.59 | 0.87 | 0.85 | 1.10 | 1.29 |
| | | R | 0.73 | 1.03 | 1.45 | 1.56 | 1.80 | 1.92 |
| | 45 | D | 0.90 | 1.07 | 1.54 | 1.80 | 2.28 | 2.91 |
| | | O | 0.62 | 0.77 | 1.07 | 1.07 | 1.41 | 1.37 |
| | | R | 1.24 | 1.54 | 1.74 | 2.16 | 2.63 | 3.09 |
| | 60 | D | 1.05 | 1.53 | 1.80 | 2.20 | 3.06 | 3.77 |
| | | O | 0.77 | 1.18 | 1.12 | 1.60 | 1.80 | 1.90 |
| | | R | 1.44 | 2.14 | 2.27 | 2.94 | 3.40 | 3.95 |

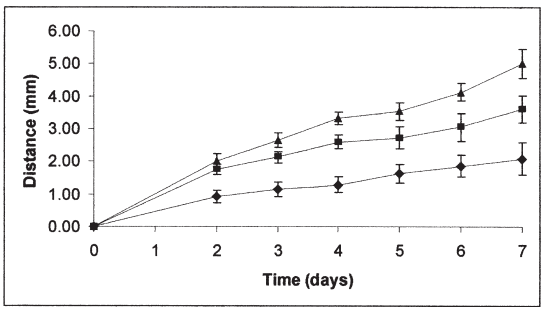


FIG. 1. 30% (v/v) PEG 600 penetration (±SD) in *E. regnans* at an incubation temperature of 30°C (◆), 45°C (■), and 60°C (▲).

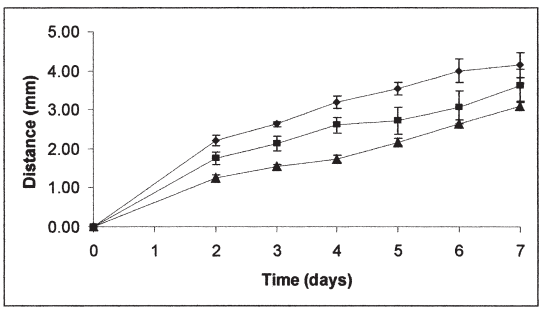


FIG. 2. PEG penetration (±SD) in *E. regnans* at 45°C for 30% (v/v) solutions of PEG 400 (◆), 600 (■), and 1000 (▲).

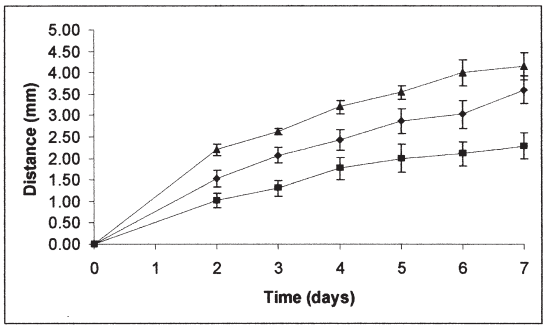


FIG. 3. 30% (v/v) PEG 400 penetration (±SD) at 45°C in E. delegatensis (◆), *E. obliqua* (■), and *E. regnans* (▲).

absorbed over the measured period, and the amount of penetration can be described as *E. regnans*>*E. delegatensis*>*E. obliqua.*

Table 2 shows the contrasts in results between species. Each set of three rows from the top of the table shows the penetration distances for each species at each temperature for each molecular weight. The distinction between each species is clear from the outset. Such a consistent finding across a variety of incubation times, incubation temperatures, and varying PEG molecular weight suggests that there may be a physical parameter (or parameters) that regulate the outcome.

The Australian Standard (AS 2082–2000) lists the physical properties of all commercially important Australian timbers. Of note is the average density of seasoned hardwood species at 12% moisture content. Density is determined by the ratio between the mass of the timber (at a given %MC expressed in kg/m$^3$) and the volume of the timber after drying to 0%MC. This is an average number that may be departed from greatly by two samples of the same species grown under different environmental conditions. *E. delegatensis, E. obliqua,* and *E. regnans* have average densities (kg/m$^3$) of 660, 770, and 625, respectively. When compared to the rate of penetration, there is a noticeable inverse concordance.

The density of the timber is an expression of the amount of wood substance present in the wood structure. Therefore, the higher the average density, the more of the wood mass is actual wood substance, and, more significantly, the less space is available to be occupied by water. The movement of PEG into timber is dependent on moisture being available in the timber to participate in a countercurrent mechanism and to facilitate transport of the molecule through hydrophilic regions of the wood structure by the provision of a solvent shell to the PEG molecule. Timbers of high density, even when saturated with water, may still have a lower %MC than a partly dry timber of lower density. This was certainly the case with these species. *E. obliqua* with an average density of 770kg/m$^3$ showed an initial MC (IMC) that did not exceed 80%, while samples *E. regnans* had IMC values around 100% (i.e., 50% of the total wood mass was water). With less water available to enact the countercurrent mechanism, and more wood substance to penetrate, it may explain the lower rate of penetration in *E. obliqua* compared to the other two species of Eucalypt.

### *Interactions between variables*

Table 2 shows the maximum average values obtained at each temperature for each species and PEG molecular weight. These data show that the greatest penetration distance is found after the longest incubation period (7 days) after timber of the lowest basic density (*E. regnans*) is incubated in a solution of the lowest molecular weight PEG (400) at the highest incubation temperature (60°C). Similarly, the highest density timber (*E. obliqua*) incubated for the shortest period (2 days) in the highest molecular weight PEG (1000) at the lowest temperature (30°C) yields the lowest amount of penetration. This effect of variables of combination was also shown statistically (Table 4).

Repeated measures ANOVA analysis of the combined effect of molecular weight and incubation temperature (Table 3) showed that both temperature and molecular weight of the PEG had independent effects on PEG penetration depth, and that there was a significant interaction between temperature and molecular weight ($p<0.001$). It was found that increasing temper-

TABLE 3. *Estimate of mean depth for treatment combinations of PEG molecular weight (MW) and temperature. 95% confidence intervals for each combination are shown in italics.*

| | | Temperature (°C) | | |
|---|---|---|---|---|
| | | 30 | 45 | 60 |
| PEG MW | 400 | 2.28<br>*1.90 to 2.66* | 3.43<br>*2.22 to 4.63* | 4.14<br>*2.59 to 5.70* |
| | 600 | 2.12<br>*1.68 to 2.27* | 2.88<br>*1.51 to 4.24* | 3.31<br>*1.43 to 5.19* |
| | 1000 | 2.04<br>*1.61 to 2.46* | 2.74<br>*1.19 to 4.28* | 3.00<br>*0.91 to 5.08* |

ature from 30 to 60 degrees increased the penetrance of the PEG 400 by 82%, compared to 56% and 47% for PEGs 600 and 1000, respectively. Between 62% and 73% of the effect was obtained by increasing temperature from 30 to 45 degrees, with smaller effects for the 45 to 60 temperature rise.

### *Penetration rate*

To better compare data sets, the average penetration values were treated to yield the rate of penetration rather than the total distance penetrated. Penetration rate was determined by applying a line of best fit to the data with the slope of the line representing the ratio of penetration distance and incubation time or penetration rate. This was performed initially over the entire data range (day 0 to day 7), but a change in the distance penetrated was noticed after the two days of incubation (See Figs. 1–3). Day 2 acted as a demarcation point between an initial and final rate of penetration. Figure 4 is presented as an example of the final rate determination and in this case, a slight increase in the gradient of the line can be observed and is concordant with the initial rate of penetration.

$R^2$ values for the lines of best fit ranged between 0.9205–0.9909, and thus the final rates show a strong linearity under most conditions. There appeared to be no consistent or significant deflections in the linear presentation of the data, which suggests that time is not a factor affecting the rate of penetration in Tasmanian oak under the conditions described.

### *Initial v. final penetration rate*

With the rate data calculated at the initial and final phases, analysis was conducted to determine which factors bore the highest $R^2$ value considering all the possible combinations of incubation temperature, PEG molecular weight, species, penetration rate (initial and final), and the difference between initial and final penetration rates.

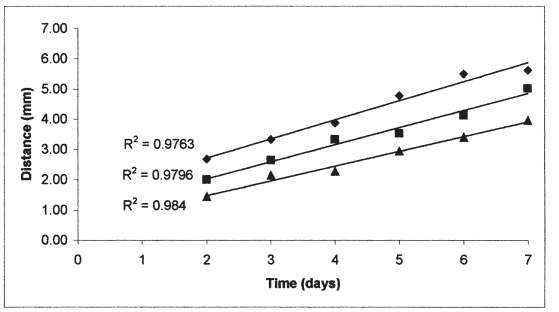


FIG. 4. PEG penetration in E. regnans at 60°C for 30% (v/v) solutions of PEG 400 (◆), 600 (■), and 1000 (▲). Final penetration rate has been determined from the slope of the line of best fit for each data set.

TABLE 4. *$R^2$ values as a function of rate variable for species (as basic density (BD) at 12%MC), PEG molecular weight (MW) and incubation temperature (Temp.). Factor mean $R^2$ is the average $R^2$ value for each rate parameter (initial, final, difference).*

| | Average $R^2$ | | | |
|---|---|---|---|---|
| Factor | BD | MW | Temp. | Factor mean $R^2$ |
| Initial Rate | 0.921 | 0.916 | 0.922 | 0.919 |
| Final Rate | 0.800 | 0.711 | 0.904 | 0.805 |
| Rate Difference | 0.650 | 0.772 | 0.468 | 0.630 |

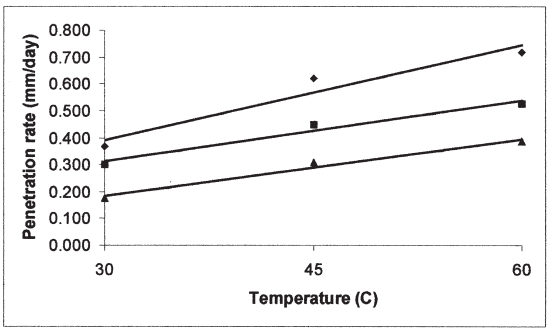


FIG. 5. Initial rate of PEG 1000 penetration by temperature in *E. regnans* (◆), *E. delegatensis* (■), and *E. obliqua* (σ).

For all combinations, the order of correlation from highest to lowest was initial rate>final rate>rate difference (Table 4). Thus the initial rate can clearly be seen as the factor that "sets the pace" for the final penetration depth. For the highest correlating variable (initial rate), the factors with the highest correlation were temperature>species (represented as basic density at 12% MC)>molecular weight.

In most cases shown in Table 5, the rate of penetration increases across the row and decreases down the column. This demonstrates that the rate of penetration increases with increased temperature and decreases as molecular weight increases. This consistency of this observation suggests effectors of penetration rate that are independent of the species being investigated. Furthermore, when PEG molecular weight and temperature are considered collectively, it could be suggested that their effect is additive: in most cases, the highest rate of penetration is found at the highest temperature (60ºC) and the lowest molecular weight (400) and the opposite is also true.

## CONCLUSIONS

This initial investigation has shown that varying the incubation time, temperature, and PEG molecular weight has an impact on the rate of penetration of PEG in Tasmanian oak (*E. delegatensis, E. obliqua,* and *E. regnans*). After two days of incubation, time was not shown to be a factor affecting the rate of PEG penetration as the amount of penetration continued to increase linearly with time. The basic density of the timber appears to be well correlated with the measured rates of PEG penetration, and an explanation has been given to support this association.

A useful goal to attain with this work is to formulate an equation predicting the likely rate of penetration for a given set of parameters. This research has brought factors such as time, temperature, and PEG molecular weight into focus, has highlighted average density as a factor, but only alluded to the impact of differing %MC. The next step will be to examine the effect on PEG penetration for species with samples at differing %MC and also investigating the effects of assorted PEG concentrations.

TABLE 5. *Final PEG penetration rates (mm/day) for each combination of experimental parameters and assessed to three decimal places.*

| *E. Delegatensis* | Temperature | | |
|---|---|---|---|
| Mol. weight | 30 | 45 | 60 |
| 400 | 0.244 | 0.391 | 0.670 |
| 600 | 0.263 | 0.369 | 0.557 |
| 1000 | 0.217 | 0.398 | 0.531 |
| *E. Obliqua* | Temperature | | |
| Mol. weight | 30 | 45 | 60 |
| 400 | 0.219 | 0.257 | 0.460 |
| 600 | 0.204 | 0.222 | 0.358 |
| 1000 | 0.177 | 0.162 | 0.228 |
| *E. Regnans* | Temperature | | |
| Mol. weight | 30 | 45 | 60 |
| 400 | 0.224 | 0.407 | 0.633 |
| 600 | 0.236 | 0.347 | 0.564 |
| 1000 | 0.239 | 0.370 | 0.486 |

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