THE EFFECT OF SOAKING PERIOD AND SAMPLE'S SIDE SURFACES ON COPPER SULPHATE RETENTION IN OVEN-DRIED RADIATA PINE¹

(Pengaruh Lama Perendaman dan Bidang Permukaan Samping Contoh Uji Terhadap Retensi Tembaga Sulfat pada Kayu Pinus Radiata Kering Oven)

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ABSTRAK

Penelitian bertujuan untuk menganalisa pengaruh lama perendaman dalam larutan pengawet tembaga sulfat dan bidang permukaan samping yang terekspos terhadap nilai retensi tembaga sulfat pada kayu *Pinus radiata* D.Donn yang dikeringkan dengan oven. Hasil penelitian menunjukkan nilai retensi tembaga sulfat dipengaruhi dengan sangat nyata oleh faktor waktu rendam dan bidang permukaan samping contoh uji yang terekspos selama proses rendaman. Nilai retensi tembaga sulfat tertinggi ($91,10 \text{ kg/m}^3$) dihasilkan melalui proses rendaman selama 1800 detik (30 menit) dengan bidang permukaan samping contoh uji yang terekspos adalah tangensial atas. Perendaman selama 10 detik dengan membiarkan permukaan samping radial contoh uji yang terekspos menghasilkan nilai retensi tembaga sulfat terendah ($6,26 \text{ kg/m}^3$).

Kata kunci: Bidang permukaan samping, lama perendaman, tembaga sulfat, *Pinus radiata*.

ABSTRACT

The experiment aimed to investigate the influence of soaking period and the exposed side surface of the samples on the retention of copper sulphate in oven-dried *Pinus radiata* D.Donn. The

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result obtained showed that the retention value was significantly affected by soaking period and by which side-surface of Radiata pine samples was exposed to preservatives during treatment. Soaking the samples with their tangential-top side-surface exposed during treatment for 1800 seconds (15 minutes) gave the highest value of copper sulphate retention (91.10 kg/m^3). On the other hand, soaking the samples with their radial side-surface exposed during treatment resulted in the lowest value of copper sulphate retention (6.26 kg/m^3) in oven-dried *Pinus radiata*.

Keywords: Side surface, soaking period, copper sulphate, *Pinus radiata*.

I. INTRODUCTION

Formerly known as a native species to southern California (USA) (Scott 1960), *Pinus radiata* D.Donn has now become an important industrial tree species in other places such as New Zealand and Australia. Nevertheless, Radiata pine is not a high natural durable species. It was recorded susceptible to the attack of sap-stain fungi, *Annobium punctatum* and other boring insects (Lewis 1966).

Treatment with preservatives chemicals is a common way used to improve the durability of low-natural durable species such as Radiata pine. Brennan (1993) stated that the chemicals secured in the sapwood of particular species developed a protection belt around the heartwood, thus could help extending the service life of a particular species to 30 years or more.

The uptake of preservatives is affected by several factors, such as the timber condition prior to treatment process (Kollmann and Côté 1968). According to Bateson (1946), Usta and Guray (2000) and Waterson (1997) drying timber before treatment could increase the treatability of timber. This is due to the additional void spaces available inside the wood as the result of drying process. However, the possibility of pit aspiration to occur during drying process, especially kiln drying, should be also recognized as this phenomenon could then prevent any liquid to transport within the wood. Messner *et al.* (2002) reported that dried Sitka spruce wood has permeability of one to five percent lower than the green timber due to this pit aspiration resulting from the kiln drying process.

Furthermore, which part of the timber exposed to the treatment process is also assumed able to influence the uptake of preservative by the wood. Cassens *et al.* (1995) stated that the treatability of wood varies with grain direction. The penetration ratio between end and side grain is about 15 to 1 for oil-based preservatives and 20 to 1 for water-borne preservatives (Cassens *et al.* 1995). According to Hunt and Garratt (1938) the penetration of preservatives through the side-grain was an important issue for long pieces of wood that were inadequately treated by end-grain-penetration alone to achieve sufficient treatment.

Another factor contributing to the success of wood treatment is the treatment method applied to impregnate the preservative into the wood. The simplest method commonly used to impregnate the preservative chemicals into the wood is soaking process. The success of wood treatment with this method is affected by, among others, the period of soaking. Muga (2001) found that Radiata pine soaked in furfuryl alcohol

resin for 10 or 20 minutes absorbed the resin greater than when it was soaked for 1 or 5 minutes in the same resin solution.

The preservative types used seemed also affecting the treatment of wood with soaking process. Many experiments indicated that longer soaking period of wood in water-borne preservative solution commonly resulted in large amount of preservatives absorbed by the wood (Abdurrohim and Ginoga 1993; Yetty and Barly 1990; Barly and Permadi, 1987; Abdurrohim *et al.* 1987; Barly and Abdurrohim 1986).

The experiment aimed to investigate the effect of applying different soaking period and exposing different side surfaces on the retention of copper sulphate in oven-dried *Radiata* pine.

II. MATERIALS AND METHOD

A. Material and Equipment

The main materials used were 60 pieces of *Radiata* pine samples measuring 35mm x 35mm x 300mm. Those samples were prepared in flat-sawn board type. The chemical used was Copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) with the strength solution at 2% and silicone gel. The equipments used were plastic boxes for soaking process, balance, timer, humidity oven and writing tools.

B. Site and Time

The experiment was carried out at School of Forest and Ecosystem Science, Institute of Land and Food Resources, University of Melbourne-Creswick, during the period of May-July 2003.

C. Procedure

1. Sample preparation and drying process

As can be seen in Figure 1, each sample had 4 side surfaces (2 radial and 2 tangential faces). However, since the two radial faces were difficult to be differentiated from one another, then it was decided to use only 1 radial side. Thus, for this experiment, there were only 3 side surfaces used which were tangential top, tangential bottom and radial. The 60 samples were divided evenly into these 3 groups based upon which side surface would be left unsealed. In order to properly assess the ability of a specific side surface in absorbing the preservative, for example the tangential-top surface, the remaining surfaces (tangential-bottom and radial surfaces) and end-surface of each sample were sealed thoroughly with silicone gel. With this sealing process, it was expected that the effect of preservative penetration through the surfaces other than the unsealed surface could be eliminated.

All samples were dried for 3-4 days in humidity oven at temperature of 40°C and relative humidity of 50%, followed by 1-day storage at temperature of 60°C and relative humidity of 55%. The purpose of this drying condition was to achieve an average final moisture content of 8-10% for the samples.

Figure 1. The determination of side surfaces of Radiata pine samples (tangential-top, tangential-bottom, and radial)

Gambar 1. Penandaan permukaan sampling pada contoh uji kayu Pinus radiata (tangential-atas, tangensial-bawah, dan radial)

2. Soaking process

The chemical used for this experiment was 2% Copper sulphate. The soaking process was designed in 4 different times: 10 seconds, 5 minutes, 15 minutes and 30 minutes. Samples from each side-surface group were divided equally into these 4 different soaking times. Those samples were weighed before and after each soaking process.

3. Data collection and analysis

The data collected in this experiment were samples dimension, initial weight prior to treatment and final weight of samples after the soaking process. These data were used to calculate the retention value using the formula below:

$$\text{Retention (kg/m}^3\text{)} = \frac{(\text{final weight} - \text{initial weight}) \text{ (kg)}}{\text{Sample dimension (m}^3\text{)}} \times \text{solution strength (\% w/w)}$$

The data on retention of the Copper sulphate in Radiata pine were analyzed using analysis of variance (ANOVA) test provided in Minitab 11 software. The data was set as two-way experimental design with the soaking period as the first factor (A) consisting of 4 levels (10 seconds, 5 minutes, 15 minutes and 30 minutes) and the side surfaces of sample as the second factor (B) consisting of 3 levels (radial-top, radial-bottom and tangential). The experiment model was as follow (Gasperz, 1991):

$$Y_{ijk} = m + A_{ik} + B_{jk} + (AB)_{ijk} + E_{ijk}$$

$i = 1, 2, 3, 4; j = 1, 2, 3; k = 1, 2, 3, 4, 5$

Where:

Y_{ijk} = Retention of copper sulphate on side surface-j of sample-k previously soaked for -i period

m = mean of population

A_{ik} = influence of soaking period- i on copper sulphate retention of sample- k

B_{jk} = influence of side surface - j on copper sulphate retention of sample- k

AB_{ijk} = influence of interaction between soaking period- i (A_i) and side surface- j (B_j) of sample- k

E_{ijk} = experimental error

Further least significant difference test (Fisher's test) was carried out following the ANOVA test if the ANOVA test above gave significant result. The Fisher's test was carried out using Minitab 11 software in order to examine any significant difference of the mean values between and within the observed factors (sample's side surface, soaking period and interaction of both factors).

III. RESULTS AND DISCUSSION

Table 1 and Figure 2 showed the retention of copper sulphate in oven-dried Radiata pine. Briefly, it can be seen that soaking period had an effect on the value of copper-sulphate retention on the samples. In general, as can be seen in Figure 2, as the soaking period increased, so did the copper sulphate retention. Thus, the shortest soaking period (10 seconds) gave the lowest value of copper sulphate retention on all side surfaces.

Most researchers found that lengthening the soaking period of wood in a solution of chemical preservatives could improve the absorption of the chemical itself by the wood, which in this context was indicated by highest retention value of the chemical in the wood (Muga 2001; Abdurrohim and Ginoga 1993; Yetty and Barly 1990; Barly and Permadi, 1987; Abdurrohim *et al.* 1987; Barly and Abdurrohim 1986). This is understandable as longer soaking period could accommodate a long interaction between the wood and the chemical being introduced.

Nevertheless, the wood itself apparently has its own limitation in absorbing chemicals and this possibly depends on various factors, such as which part of the wood is being introduced to the chemicals and/or the type of the chemicals used. As can be seen in Table 1, the longest soaking period (1800 seconds/30 minutes) apparently did not result in the highest retention of copper sulphate in Radiata pine wood when only its tangential-bottom surface exposed to the preservatives solution.

Table 1. Average value of copper sulphate retention (kg/m³) in oven-dried Radiata pine
Tabel 1. Nilai rata-rata retensi tembaga sulfat (kg/m³) pada kayu Pinus radiata kering oven

Sample's side surface (Permukaan samping sampel)	Copper sulphate retention (kg/m ³) (Retensi tembaga sulfat) (kg/m ³)			
	Soaking period (seconds) (Lama perendaman) (detik)			
	10	300	900	1800
Tangential -bottom (<i>Tangential-bawah</i>)	11.10	46.53	86.75	75.05
Tangential-top (<i>Tangential-atas</i>)	11.70	35.59	71.18	91.10
Radial/ <i>Radial</i>	6.26	15.56	25.58	32.71

Figure 2. Retention of copper sulphate impregnated on different side surfaces of Radiata pine
Gambar 2. Retensi tembaga sulfat yang diimpregnasikan pada permukaan samping kayu Pinus radiata

The result of analysis of variance (ANOVA) test in Table 2 confirmed that the retention of copper sulphate was not only affected significantly by the soaking period but also by which side surface was exposed to the treatment process. On the other hand, the interaction between which side surface was exposed to preservative and the soaking period was found to have less significant effect on the retention of copper sulphate. In this context, as previously shown in Figure 2, the retention of copper sulphate impregnated on radial surface of Radiata pine was observed lower than that from tangential surface for all soaking periods.

Table 2. The-2-way ANOVA (Analysis of variance) result for analyzing the effect of soaking period and sample's surface on copper-sulphate retention in oven-dried Radiata pine

Tabel 2. Uji ANOVA (Analisa keragaman) untuk pengujian pengaruh lama perendaman dan permukaan samping sampel terhadap retensi tembaga sulfat pada kayu Pinus radiata kering oven

Source of variance (Sumber keragaman)	Df (Derajat bebas)	SS (Jumlah kuadrat)	MS (Kuadrat tengah)	F-value (F-bitung)	P (Peluang)
Soaking period (Lama perendaman)	3	0.0223877	0.0074626	12.44	0.000
Sample's side surface (Permukaan samping sampel)	2	0.0188221	0.0094110	15.68	0.000
Interaction between soaking period and sample's side surface (Interaksi antara lama perendaman dan permukaan samping sampel)	6	0.0066227	0.0011038	1.84	0.111
Error (Galat)	48	0.0288032	0.0006001		
Total (Total)	59	0.0766357			

Remarks (Keterangan): * = Significant effect; Df= Degree of freedom; SS= Sum of square; MS=Mean of square; P=Probability

As shown in Figure 1, the radial surface meant on radiata pine flat-sawn board samples refers to the side surface that is parallel to the rays structure. Based on this illustration, it can be assumed initially that the low level of preservative penetration into Radiata pine through this specific surface is possibly due to the existence of the cell walls which became the first barrier for the flow of preservatives into the wood. Cell walls were seen as one of the pathways for the transportation of water or any liquid within the wood (Stamm 1967 in Langrish and Walker 1993). Nonetheless, this preliminary assumption needs more experiments to validate its accuracy.

It also can be seen in Table 1 and Figure 2 that the retention of copper sulphate impregnated from tangential-bottom surface of the sample was higher than that from tangential-top surface of the sample for soaking periods of 300 seconds and 900 seconds. Further test (Appendix 1) confirmed that significant difference in copper sulphate retention was found between samples with their tangential top and bottom exposed to preservatives during treatment.

In Figure 1, it can be seen that the tangential surfaces on flat sawn board type refers to side surfaces that are perpendicular to rays structure and growth rings. Based on this illustration, it seems that the tangential bottom surface is mostly started with old cells whereas the tangential top surface is started with young cells as the growth of tree in diameter usually occurs on the outer part of the wood. It is a common knowledge that old cells are usually not as permeable as young cells, thus this reason could not be used to explain why the absorption of copper sulphate from tangential-bottom surface was commonly higher than that from tangential top surface. One possible explanation was that the drying process might cause a lot of pit aspiration in young cells, therefore the penetration of copper sulphate was commonly low through the tangential top surface of the Radiata pine wood. Again, this assumption needs more experiments to confirm its precision, including experiment on wood anatomy and structure.

IV. CONCLUSION AND RECOMMENDATION

1. The retention value of copper sulphate in oven-dried Radiata pine was significantly affected by soaking period and by which side-surface was exposed to preservative solution.
2. The longest soaking period (1800 seconds) resulted in highest retention of copper sulphate, except for samples with its tangential-bottom surface exposed during treatment process.
3. Samples with their tangential surfaces exposed to preservative solution had higher retention of copper sulphate than those with their radial surface exposed.
4. The interaction between soaking period and which side-surface was exposed to preservative solution was found to have less significant effect on the retention value of copper sulphate in oven-dried Radiata pine.
5. Further and detailed experiments are required to investigate the exact causes behind the results obtained from this experiment.

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Appendix 1. Least Significant Difference (LSD) Fisher-s test to compare retention value between soaking period groups and side surface groups

1. One-Way Analysis of Variance : Comparison between soaking period Groups

1. Analisa keragaman 1 arah : Perbandingan antar grup berdasarkan lama perendaman

Analysis of Variance for retention

Source	DF	SS	MS	F	P
Soaking	3	0.022388	0.007463	7.70	0.000
Error	56	0.054248	0.000969		
Total	59	0.076636			

Fisher's pairwise comparisons

Family error rate = 0.199

Individual error rate = 0.0500

Critical value = 2.003

Intervals for (column level mean) - (row level mean)

	0.17 minutes	5.00 minutes	15.00 minutes
5.00 minutes	-0.03558 0.00994		
15.00 minutes	-0.06400 -0.01847	-0.05118 -0.00565	
30.00 minutes	-0.06911 -0.02358	-0.05629 -0.01076	-0.02788 0.01765

2. One-Way Analysis of Variance : Comparison between side-surface groups

2. Analisa keragaman 1 arah : Perbandingan antar grup berdasarkan permukaan samping yang terkena larutan pengawet

Analysis of Variance for retention

Source	DF	SS	MS	F	P
Side sur	2	0.01882	0.00941	9.28	0.000
Error	57	0.05781	0.00101		
Total	59	0.07664			

Fisher's pairwise comparisons

Family error rate = 0.121

Individual error rate = 0.0500

Critical value = 2.002

Intervals for (column level mean) - (row level mean)

	1	2
2	-0.02540 0.01493	
3	0.01452 0.05484	0.01975 0.06008

Remarks:

1 = tangential bottom

2 = tangential top

3 = radial surface