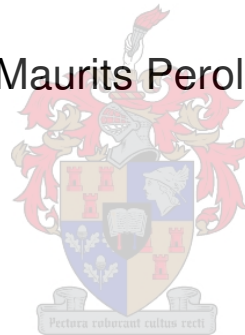


**THE EFFECT OF SODIUM LAURYL SULPHATE ON  
BLUE STAIN, MOULD GROWTH AND SURFACE  
PROPERTIES OF SA PINE**

by

Maurits Perold



Thesis presented in partial fulfillment of the requirements for the degree of  
Master of Science in Forestry (Wood Science) at Stellenbosch University

**April 2006**

STUDY LEADER: Prof. T. Rypstra

CO-STUDY LEADER: G.C. Scheepers

## DECLARATION

I, the undersigned, hereby declare that the work contained in this thesis is my original work and has not previously in its entirety or in part been submitted at any university for a degree.

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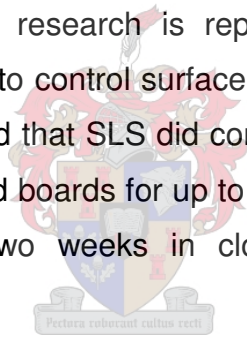
M. Perold



## SUMMARY

Producing high quality end products, rather than focusing on volume production, is slowly but surely becoming the main driving force in the wood processing industry of South Africa. Drying defects such as surface checks and discolouration by yellow stain and kiln brown stain are major factors in softwood timber downgrades when selecting furniture grade timber. Previous efforts to control these defects have focused on schedule adaptation, but as the industry is still mainly concerned with volume production, and because of the varying lumber price, longer schedules to control these drying defects have not yet been that attractive for the larger sawmills.

In ongoing research in this laboratory, a dip treatment of freshly sawn softwood boards in an aqueous solution containing a surfactant called sodium lauryl sulphate (SLS) was used to try to influence the mechanism involved in the development of these stains. Further, complementary research is reported in this study. Firstly, the possibility of using SLS solutions to control surface mould development and bluestain was investigated. Results showed that SLS did control the development of blue stain and mould growth in open-stacked boards for up to three weeks, using concentrations as low as 0.1%, and up to two weeks in closed-stacked timber when using concentrations of 0.2%.



Secondly, it was investigated whether SLS treatment would have a detrimental effect on downstream product quality; in particular, kiln dried boards and glued components for furniture manufacturing. Since SLS influenced fluid water flow during kiln drying, the treatment could have exacerbated the occurrence of surface checking due to altered moisture distribution profiles. Results of this investigation showed that the SLS treatment did not result in increased surface checking.

Thirdly, as furniture quality timber treated with SLS would be glued, (and also finished with surface coatings), it was further considered important to determine if SLS treatment influenced adhesion properties of wood surfaces. Based on shear test results, it was established that SLS did not influence the adhesion properties of wood when glued with two most commonly used glues in the furniture industry i.e. polyvinyl acetate (PVAc) and urea formaldehyde (UF).

## OPSOMMING

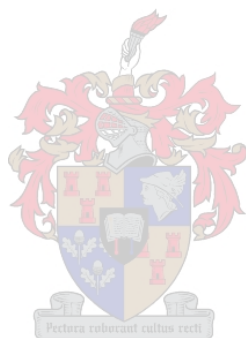
Die algemene neiging wat vandag in die SA saagmeulindustrie bestaan, is om van 'n volume-georiënteerde na 'n kwaliteitgeoriënteerde produksie-ingesteldheid toe te beweeg. Tans is oppervlakkraak en geel- en bruinverkleuring in hout wat vir meubels gebruik moet word dié drogingsdefekte wat die meeste tot afgradering lei. Omdat die bedryf nog grotendeels op volumeproduksie ingestel is, vind toepassing van nuwe navorsingresultate, wat grootliks skedule-aanpassings en langer drogingskedules behels, nog nie sy regmatige byval by die meeste, groter saagmeulens nie.

In voortgesette navorsing wat in hierdie laboratorium uitgevoer is, is die onderdompeling van pasgesaagde naaldhoutplanke in 'n waterige oplossing bevattende 'n oppervlaktespanningsmiddel, natriumlaurielsulfaat (NLS), gebruik om bogenoemde verkleuringsmeganisme te beïnvloed. Verdere, komplementêre ondersoek word in hierdie studie gerapporteer. Eerstens is die moontlikheid ondersoek of die ontwikkeling van blouverkleuring en oppervlaktewamme dmv NLS oplossings beheer kan word. Resultate het aangetoon dat met NLS-konsentrasies so laag as 0.1%, die ontwikkeling van blouverkleuring en oppervlaktewamme in oopgestapelde planke tot drie weke lank beheer kon word. In dig-gestapelde planke kon met 'n 0.2% oplossing tot twee weke lank sukses behaal word.

Tweedens is bepaal of NLS-behandeling enige nadelige effekte op houtkwaliteit verder in die waardeketting kon hê; spesifiek oondgedroogde planke en gelymde komponente wat in meubelvervaardiging gebruik word. Omdat NLS vloeibare waterbeweging tydens droging beïnvloed het en daardeur vogverspreidingsprofiële verander het, kon dié behandeling die voorkoms van oppervlakkraak dalk verhoog het. Resultate van hierdie ondersoek het egter daarop gedui dat vorming van oppervlakkraak nie verhoog is nie.

Laastens, omdat meubelkwaliteit hout gelym word (en ook met oppervlakmiddels afgewerk word), was dit wenslik om vas te stel of die kleefeienskappe van houtoppervlakke deur die NLS-behandeling geaffekteer is. Dmv skuifspanningresultate is vasgestel dat adhesie van die mees algemeen gebruikte

kleefmiddels in die meubelvervaardiging, nl. polivinylasetaat (PVAc) en ureumformaldehyd (UF), nie nadelig beïnvloed is nie.



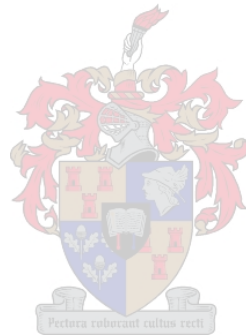
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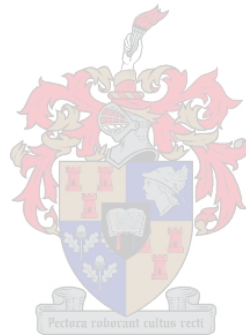
**SALMA** and **THRIP** for supporting my project financially and supplying bursaries;

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## PREFACE

This thesis is presented as a compilation of manuscripts. Chapters 3, 4, and 5 were formatted according to the style of the scientific journal to which it will be submitted.



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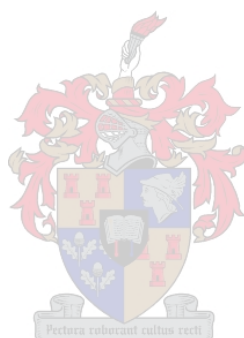
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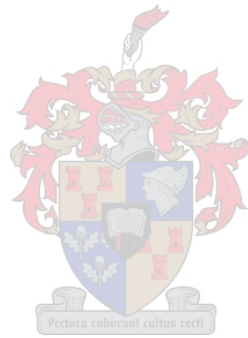
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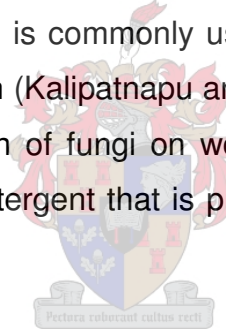
# Chapter 1: Introduction



## 1. Introduction

Blue stain and mould is a common problem for softwood sawmills, particularly during the warm, humid months of the year when fungal infection rates are the highest. To control blue stain, felled logs have to be swiftly transported and sawn so that the lumber can be dried before the fungi get a chance to spread through the wood (Uzunovic *et al.*, 1996). Especially freshly sawn, close-stacked boards, should not be left in front of the kiln too long. Many different chemicals have been developed for the control of blue stain and mould growth on wood. In the past, some of these chemicals were highly toxic, but recent environmental awareness has resulted in a continuing drive to find chemicals and treatment methods that are less harmful to the environment and humans.

In this study, the effect of sodium lauryl sulphate (SLS) on the growth of mould and blue stain was investigated. SLS is commonly used in microbiology as an agent of cell lysis and protein denaturation (Kalipatnapu and Chattopadhyay, 2005) and could thus potentially inhibit the growth of fungi on wood. It is non-toxic, degradable by bacteria, and also a common detergent that is present in many household cleaning products.

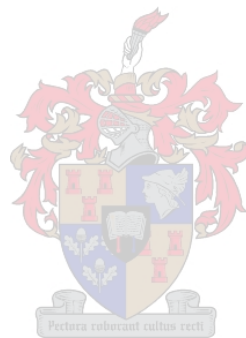


However, SLS treatment could also influence the moisture gradient in drying lumber (Scheepers *et al.* 2006) and act as a surface contaminant, thus yielding a poorer (or improved) surface quality, especially in regard to surface checking and gluability. Consequently, the benefits of fungal control could be offset by the increased occurrence of surface checking and/or inferior adhesive bonding quality of subsequent products. The influence of SLS treatment on the development of surface checks during kiln drying and the gluability of the dried products were, therefore, also investigated.

Thus, the research reports in this thesis describe whether:

1. SLS could be used to control the development of bluestain and mould growth in freshly sawn SA pine species,

2. SLS solutions affected the incidence of surface checking and
3. adhesion properties, of importance in gluing (and finishing), were compromised by the treatment.





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**Uzunovic, A., Webber, J.F. and Dickinson, D.J., 1996.** Comparison of blue stain fungi growing *in vitro* and *in vivo*. *Dissertation, Swedish University of Agricultural Science., ISBN 91-576-5115-9. pp 8 – 20.*



## Chapter 2: Literature review



## 2.1 Surface checks

### 2.1.1 General

Surface checking is one of the most common drying defects in the timber industry today. It occurs in both soft- and hardwoods and is one of the largest quality-reducing factors in the furniture industry. Surface checks are longitudinal failures that usually develop in the wood rays of latewood on the most tangential face or the face furthest away from the pith (Schniewind, 1963).

Radial seasoning checks originate at the interconnection between rays and thick-walled prosenchyma as a result of the higher tangential shrinkage than radial shrinkage (Hale 1957). His explanation for more tangential than radial shrinkage included the fact that radial shrinkage is a summation of the high shrinkage in the relatively high density latewood and the low shrinkage in the relatively low density earlywood. While tangential shrinkage is dominated by the large amount of shrinkage of the latewood and forces in the process the pliant earlywood to shrink with it. The difference in shrinkage between the radial and tangential directions is not a result of a structural difference in the cell walls. The radial shrinkage is prevented by the radially orientated (vertical) rays because individual cells shrink very little lengthwise and hold the relatively flexible latewood in position. The fiber separation would most likely occur in wood that has a large difference in density between the early and latewood.

In two other articles Pentoney (1953) and Peck (1957) also found that earlywood shrinks less than latewood due to the lower density of earlywood. Dinwoodie (2000) said the larger tangential shrinkage is due to the higher shrinkage of the higher density latewood compared to the lower density earlywood. Perré (2003) looked at the role of wood anatomy in the drying of wood and said that surface checks usually initiate in the latewood due to higher shrinkage values of the latewood. Therefore, the possibility of surface checks occurring on the tangential face of boards is much higher when latewood areas are present on the tangential face.

Various other natural factors govern the development of surface checks. The original position of the board in the log, whether sapwood or heartwood, also governs the development of surface checks because of the difference in moisture content. The heartwood of the longleaf pine, for example, has a moisture content range of 30-40 percent while the moisture content of the sapwood exceeds 100 percent (Desch and Dinwoodie, 1996; Hukka, 1989; Haslett, 1998). McGinnes and Dingeldein (1969) reported that the heartwood of the Eastern Redcedar had a mean moisture content of 22% with a range of 19-25%. Therefore, the heartwood will not check because of the low (if any) moisture gradient that develops in the timber and the heartwood needs to be separated from sapwood so that different kiln schedules can be applied to both (Haslett, 1998).

Kiln drying, defined by Henderson (1936) as a method of accelerated removal of water from timber under controlled conditions, is not the only source of surface checking. Surface checks can also develop during air drying. Even the time spent in front of the kiln is a major factor governing the development of surface checks (Simpson, 1991, Harrison, 1968; Hukka, 1989). When wood is exposed to air that has a lower moisture content than the wood it will try to equalize and, therefore, it will lose moisture from the surface and as Wiberg and Morén (1999) had shown, the wet line recedes to about 2 mm below the surface. This process starts as soon as the boards exit the specific processing machine. The time stacked timber spends in front of the kiln will determine the amount of moisture lost from the surface (surface is about 2 mm deep), because it will always be exposed to an environment that has a lower equilibrium moisture content (EMC). This is critical, because when the surface reaches fiber saturation point (FSP) it will start shrinking and stresses will develop in the surface. As soon as the stresses reach the critical level the surface will check.

Air-drying is the oldest method used to dry both fast drying softwoods and slow drying hardwoods. In a few studies done on surface checks it was shown that air-drying resulted in lumber of lower quality (more checks) (Rucker and Smith, 1961) than kiln-dried (Wynands, 1963) and solar-dried lumber (Oliveira, Skaar and Wengert, 1982). The main reason for the increased surface defects in air-drying is because of the lack of atmospheric control, especially relative humidity, which

controls the rate at which moisture evaporates from the surface and, therefore, controls the moisture gradient that develops between the core and the surface. In a controlled environment, such as a kiln, one can control the relative humidity and thus the rate at which water is removed from the surface. The rate at which water is removed from the surface will control the moisture gradient between the surface and the core.

The main reasons for accelerated (forced) removal of water by using kiln drying is to control the dimensional changes the wood undergoes, thereby, reducing the degrade associated with air-drying. Accelerated drying makes wood less likely to deteriorate from fungi and insects. Finally, the lower mass reduces transporting costs (Henderson, 1936).

Surface checks initiate during the early stages of drying when the wood is still green (an average moisture content of above 100%) (Simpson, 1991; Hukka, 1989). When the wood is exposed to environments with a lower moisture content, the free water in the wood surface will evaporate and the surface will dry out. As soon as the surface reaches a moisture content of about 30% (FSP) the bound water in between the fibres will evaporate and the wood will start shrinking (Skaar, 1988).



During the surface evaporation a moisture gradient will develop between the surface and the core (Schniewind, 1963) and, although the gradient is necessary for the wood to dry, an excessive gradient will cause the surface to dry out much quicker than the core and large tensile forces will develop in the surface (Pratt, 1974). Once the tensile forces exceed the mechanical strength of the wood, a check will develop in the surface (Rice, 1994). The length and the depth of these checks are determined by the extent of the drying conditions – the more severe the drying rate, the bigger the moisture gradients between the core and the surface and the more severe the checking.

Tension develops in the surface as the surface dries and the wetter inner regions (core) are placed under compression. Stress reversal takes place as the drying process continues and the core reaches FSP. The surface does not allow the core to shrink and tension develops in the core placing the surface under compression.

The surface checks that developed in the surface are now closed as the core compresses the surface. This is generally the final stress development and leads to the final state in which the board is casehardened. Casehardened boards are not favourable seeing that they distort as soon as they are processed further – like being sawn, moulded, etc. It is, therefore, necessary to condition the boards once they finished the final stage of drying (Pratt, 1974).

Surface checks are not always visible after drying due to stress reversal and subsequent casehardening of the surface during drying, which closes the checks that developed earlier (Rémond *et al.*, 2003). Surface checks become visible once the surface is planed and exposed to changing environments, especially changes in the relative humidity (Pratt, 1974, Simpson, 1991).

The surface checks open up due to initial unnatural internal swelling as a result of mechanical inhibition of the external swelling. According to Stamm (1964) the natural process cell lumens follow, when the bulk wood is swelling or shrinking, is to remain constant in size. This is because individual fibers tend to shrink inwards to the center of the fiber and not to the center of the cell lumen because internal fibril wrappings tend to restrain internal swelling. As soon as a dry piece of wood is held in a clamp and exposed to water or water vapour, the outward swelling is very little so the swelling primarily occurs into the cell lumen resulting in a decrease in the size of the lumen. Now, when the wood sample is dried again the wood sample will shrink normally (dimensions will decrease in size) and will shrink proportional to the amount of internal swelling that occurred. Therefore, the dry dimensions will be less than the original dry sample.

In a study on the dimensional stability of Douglas-fir plywood as it was affected by internal and external dimensional changes, Chow and Wellwood (1968) wrote that glue lines restrain the external swelling of the plywood to some degree and thus swelling may be into the cell lumens. Their results indicated a ratio of external swelling to internal swelling of about 1. Overall, the glue line restraining effect resulted in a decrease in the external swelling and an increase in the internal swelling.

In 1992 Hart *et al.* examined the causes of open surface checks at the end of a drying run in oak as well as find ways of eliminating the defect or reducing the losses due to the defect. In the article they referred to the mechanism of internal swelling as the *axe-handle effect*. When the portion of the dry handle, the portion which is tightly wedged into the metal of the axe head, is exposed to moisture it results in internal swelling and, when it is allowed to dry out again, it will shrink externally and the axe head will become loose. This is a phenomenon that has probably been known for as long as wooden handles have been in use. Importantly, the authors also said that a less commonly known fact is that a dry portion of a board may provide the same restraint as the metal portion of the axe head does, causing internal swelling when exposed to moisture. Therefore, internal swelling of a dry shell will occur when the dry, shrunken core restrains it from swelling externally (the state called casehardening) and subsequently result in external shrinkage of the previously wetted surface. It is this phenomenon that causes closed surface checks to open when exposed to moisture because the dry core restrains the dry shell from swelling externally and thus swells internally. The resulting shrinkage occurs as normal shrinking does (external shrinkage) and results in opening the previously closed checks.

### **2.1.2 The measurement of the surface checks**

The new quality mark, the ZA Dry-Q, developed in South Africa states the procedure to expose and measure the surface checks in the already dried timber. In this investigation the procedure and guidelines of the ZA Dry-Q Kiln Drying Management System were followed because the research was done for the industry and the ZA Dry-Q was developed for the SA industry.

The method is as follows: Once the dried timber is removed from the kiln it is left inside to reach room temperature and then two millimeter are planed off the most tangential face or face furthest from the pith. The boards are then submerged in water for about ten seconds, removed while allowing most of the water to run off the surface and then placed in the sun outside to allow the surface to dry out again. Once the surface of the boards is completely dry and the checks have re-opened, the checks are measured and counted.

### 2.1.3 Methods to reduce surface checking

The most commonly known and widely used method is schedule adaptation. Surface checks occur due to too steep moisture gradients that develop between the surface and the core in the early stages of drying and, because the schedule determines the rate of moisture evaporation from the surface that determines the moisture gradient, the schedule can be adapted to ensure an optimal surface evaporation. This can be achieved by increasing the relative humidity in the drying environment surrounding the boards with the use of steam (Hukka, 1989) at the beginning of the drying run so that the wood loses moisture at a slower rate and the development of the stresses in the board is more gradual from the surface to the core (Pratt, 1974).

Only once the timber reaches fibre saturation point (FSP) can the humidity be lowered and the temperature increased to provide more rapid drying conditions (Walker, 1993). Due to rapid market growth, many companies were forced to produce more in a shorter time and, because kiln drying is the longest step in the process chain, the operators always cut the kiln drying shorter and shorter, which automatically led to increased drying defects.

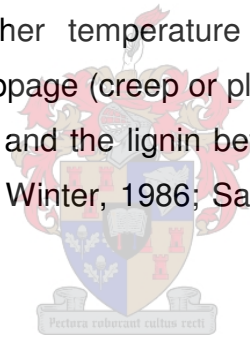
Some research done on oak lumber using moisture pallets (Hart and Gilmore, 1985) was tested successfully for the control of surface checks occurring in hardwood lumber, but the cost of this technique far exceeded its benefits. This technique “added” a surface to the wood and, thus, moisture had to travel through the plywood before it could evaporate. So, by doing that, they basically shifted the resultant tensile stresses due to shrinkage from the surface of the board to the surface of the plywood ensuring the plywood absorbed all the tensile forces. This resulted in much less surface checks occurring because the speed at which moisture evaporated from the surface, which determined the development of tensile forces due to the moisture gradient between the surface and the core, was controlled.

The use of accelerated drying schedules has produced some outstanding results contrary to the general belief that high temperature drying results in higher degrade (especially surface checks). A study done by Saito (2003) looked at determining the



optimal drying temperatures for Sugi (*Cryptomeria japonica*) boxed heart square lumber. The main aim was to find temperatures that would produce products as fast as possible. In order to do so, a temperature range of 50 to 135°C was tested and compared in terms of surface and internal checks, shrinkage, colour and durability. Saito found that higher temperature schedules (80-100°C) produced timber with less surface checks than lower temperature schedules, but produced wood with a darker coloured surface and had 3.5 mm more shrinkage than air-drying. The optimal temperature range for drying Sugi was recommended to vary between 50°C and 70°C.

Espenas (1971) found that wood dried at higher temperatures had a lower equilibrium moisture content (EMC) and shrunk more than wood dried at lower temperatures showing the possible effect of the high temperatures on the hygroscopicity of the wood. The higher shrinkage was a result of the higher stresses that developed during the higher temperature drying compared to the lower temperature drying as well as slippage (creep or plastic flow) (Beckwith, 1994) of the fibres as the stresses increased and the lignin between the fibres softened (Koran, 1967; Palka, 1973; Irvine, 1984; Winter, 1986; Salmen 1990; Eriksson *et al.*, 1991; Uhmeier *et al.*, 1998).



Another study done by Edvardsen *et al.* (1999) found that wood exposed to high drying temperatures (110°C) had a lower EMC and the dimensional changes smaller than wood dried at lower temperatures (50°C). A possible explanation is the change in hygroscopicity of the polyoses resulting in less adsorption sites as well as the possible reduction in the polyoses content. Even after five cycles of wetting and drying, high temperature treated wood showed less swelling when exposed to moisture and less shrinkage when exposed to a drier environment than lower temperature treated wood.

The use of accelerated drying schedules (high temperature drying) in the drying of construction grade timber has been implemented successfully. However, the defect yellow- and brown stain is associated with high temperature drying (Kapp *et al.*, 2002) and one needs to be sure for which end-use the timber is intended before deciding on a drying schedule.

The quality of the wet sawn surface of boards plays a crucial role in determining the extent of surface check development. The rougher the surface, the easier forces can be concentrated and lead to tears or splits. With a smooth surface the forces are well distributed throughout the whole surface and the whole surface absorbs the tensile impact. During primary processing, the timber surface is exposed to various mechanical forces that could have an impact on the quality of the wet surface. Gaby (1963) looked at the effect of three mechanical processes on the development of surface checks in oak. He looked at band and circular sawn boards as well as the effect of green surfaced boards. He said that the combined effect of ray orientation in oak and the sawing action may be compounding factors that could lead to localized drying stresses that result in the increase in the development of surface checks. His results indicated that circular sawn boards checked much more than band sawn or green surfaced boards.

In two other studies the effect of presurfacing of green lumber on the development of surface checks was also evaluated. In one study Leney (1964) presurfaced oak before kiln drying and compared them with rough sawn boards. The results indicated that the rough surface checked more than the planed boards. Rietz and Jenson (1968) pre-surfaced beech wood before drying and the results indicated a reduction of the average length of the surface checks from 79.2 mm (31.2 inches) to 41.6 mm (16.4 inches).

Cutting thinner boards from the higher density outer sapwood will result in less surface checks. The reason for this is higher density wood shrinks more per percent moisture lost and sapwood has a moisture content of more than 100% (Desch and Dinwoodie, 1996). The higher the shrinkage, the higher the stresses that will develop in thicker boards compared to thinner boards because there is more wood that can shrink and the moisture gradient between the core and the surface will be greater in thicker boards.

In addition to other methods to control surface checks, three ways of dimensionally stabilising wood were formulated, namely 1) by reducing the hygroscopicity of wood so that less water is adsorbed, 2) by forming cross links between the interfibre structural units so that separation of these units is minimised as well as the

accompanied swelling and 3) by depositing a bulking agent within the water swollen structure to reduce the shrinkage (Stamm, 1956). One way of reducing the hygroscopicity of wood was to heat wood to high temperatures, breaking down the more hygroscopic polyoses and resulting in the formation of insoluble polymers from the sugars and furfural formed (Seborg *et al.*, 1953).

The formation of cross links within wood, as shown by Tarkow and Stamm (1953), was accomplished by reacting divinyl benzene with small amounts of acidified formaldehyde vapour. Unfortunately, due to the high acidic conditions under which the reaction took place, the strength properties of the wood were badly impaired.

Wood containing a high resin or water soluble extractive content tends to develop less surface checks due to the phenomenon known as *bulking*. Bulking is when fibres remain swollen after they are dried due to extractives that are deposited within the cell walls as the water evaporates from the fibres. Choong (1969) found that by extracting pine using either hot-water treatment or combined hot-water and chemical treatment resulted in a large increase in shrinkage. The extraction process removed soluble extractives that would otherwise have stayed behind and bulked the fibres resulting in a decrease in the total shrinkage. The extraction process, therefore, resulted in the fibres moving closer together (because now without natural bulking agents) resulting in more shrinkage than generally. They also found that more extractives are present in the heartwood than in the sapwood, which is shown by the higher percentage shrinkage in the sapwood (Choong, 1969) as well as indicating the reason for the higher number of surface checks developing in the mature wood (Pratt, 1974 and Simpson, 1991).

Externally added water soluble solutes also tend to remain behind in between the fibres as the wood loses water and dries. For instance wood containing high concentrations of salts will not shrink as much as wood that is only saturated with water. More salt will remain behind the higher the concentration at saturation of the cell walls just before the cell walls start shrinking. Thus, salts reduce the shrinkage in proportion to the volume the salts occupy within the cell wall (Stamm, 1964).

The principle of bulking and its application in research to control the development of surface checks has received a lot of attention. Because surface checking is a result of the shrinkage of wood and bulking controls the shrinkage of wood, it also controls the development of surface checking. According to Stamm, bulking treatments fall into a few categories (Stamm, 1964, Tsoumis, 1991).

The first is bulking using *salt treatments*. It has been shown that salt reduces shrinkage significantly, but is not very functional because the salt tends to corrode metal fasteners and tools and most of them are not very soluble in water, which is an essential necessity. *Potassium thiocyanate* is one of the most soluble salts and reduces the shrinkage of wood by about 50% (Stamm, 1964; Tsoumis, 1991). Colgrove (1956), Haygreen (1962) and Lund and Taras (1959) also found that *sodium chloride* reduced surface checking, but was also very corrosive on metal fasteners.

A second is bulking using *sugar treatments*, which results in a larger bulking effect than salts. This is mainly due to the greater solubility and the greater partial specific volume of the sugar in aqueous solution. The use of sugars as bulking treatments was tested commercially, but was not financially viable and posed a threat of decay because sugars are highly susceptible to attack by various organisms (Stamm, 1964).

The third bulking agent was a water-insoluble wax. One problem was that water-insoluble wax agents could not easily be deposited into the cell wall and had to undergo a two step replacement process. Firstly, the water in a small swollen specimen was replaced by a liquid such as Cellosolve (ethylene glycol monoethyl ether), which is soluble in water and acted as a solvent for waxes because it has properties related to alcohol and ethers. This occurred at 135°C, the boiling point of Cellosolve, after which the specimen was placed in molten wax, such as beeswax and the Cellosolve allowed to slowly distill off (Stamm, 1964).

The fourth bulking treatment entailed using thermosetting resins. Earlier attempts using phenolic resin varnishes resulted in internal coatings because the resins were too highly pre-polymerized to enter the cell walls. They, therefore, acted by cutting

down the rate of water adsorption. It was realised that a very low degree of prepolymerization was needed to ensure solubility in water. The resin, phenol formaldehyde, met the requirements for water solubility (Stamm, 1964).

The fifth wood stabilizing process was developed because treating wood with dimension-stabilizing resins resulted in the wood getting brittle to such an extent that it could not be used for its intended purposes. It was found that the embrittlement could be eliminated if single molecules, having only one reactive group, could replace the hydroxyl groups of the wood. Less hygroscopic acetyl groups, that have a considerably larger volume, was used with pyridine as the catalyst (Stamm, 1964).

The sixth and most commonly used wood stabilization treatment is polyethylene glycol or PEG. It consists of a series of polymers of glycol in which water was eliminated between two or more of the glycol groups resulting in ether linkages between ethyl groups. PEG with a molecular weight of 600 is highly soluble where PEG with a molecular weight of 6000 is soluble to a limited extent. PEG's with a molecular weight of 1000 and lower are excellent bulking agents. Research done by Stamm (1959) indicated that PEG with a molecular weight of 1000 effectively stabilized wood by reducing the shrinking and, therefore, eliminating the checking problem associated with shrinking of wood when an amount equivalent to 30% of the dry weight of the wood is used. Less is required to control surface checking. Stamm said that PEG can easily be leached from the wood but that surface coatings do an excellent job of keeping them inside the wood. Using Stamm's results, Mitchell and Wahlgren (1959) successfully treated Walnut gunstocks and reduced the shrinking and swelling properties of the wood making the product much more dimensionally stable. It resulted in gunstocks with no checks, splits or collapse. Wood treated with PEG had a considerable decay resistance under nonleaching conditions, which is surprising because PEG is non-toxic. PEG prevents the uptake of water vapour necessary to support decay and possibly dehydrates the fungi making them ineffective. The very low molecular weight PEG's not only resulted in a 100% shrinkage reduction but also tended to swell the wood as it dried (Stamm, 1964).

Other ways of dimensionally stabilizing wood included treating wood with glycerol before kiln drying. Northway *et al.* (2003) found that the glycerol treatment reduced

surface checking more than 10% and that the surface checks in the treated wood were shallower and narrower compared to the longer and deeper surface checks in the untreated wood. The treatment did however influence the surface hygroscopic nature of the wood allowing the wood to take up and lose moisture more readily, which will hinder the in-service behaviour of wood treated with glycerol. The glycerol had penetrated to a depth of between 1 and 3 mm at the end of drying and conditioning. In practice this layer will be removed during further processing and at the same time the hygroscopic effect of the glycerol treatment as well as any shallow surface checks will be removed.

Harrison (1968) tested a sodium alginate solution and found it was very effective in preventing surface checking in sawn timber that was air-dried. Sodium alginate is a plant gum obtained from seaweed. In Australia it is obtained from kelp off the east coast of Tasmania. The treatment was tested on numerous refractory hardwoods and successfully controlled surface checking in all of them. The treatment had no effect on the machining and the physical properties of the timber.

In order to test the hypothesis that a reduction in the rate of surface moisture loss would reduce surface checking, Rice, Wengert and Schroeder (1988) tested six coating types on unseasoned, presurfaced oak wood. Of these six coatings selected, PVAc was the most effective coating to reduce surface checks and included additional benefits like quick drying and ease of application.

## **2.2 Surface characteristics influencing bonding of adhesives and wood finishes**

When analyzing the surface characteristics of timber one needs to specify whether it is a macro-, micro- or molecular level of characterization. Quantifying and qualifying surface checks usually occur through that what is visible to the naked eye, although detailed examination has proceeded to the macro level when e.g. the effect of rays on the development of surface checks are studied. In studying the phenomena of

adhesion or how one substance “holds onto” another, one has to go down to the molecular level to understand the science involved.

In the South African Bureau of Standards Code of Practice, “The Terminology and classification of adhesives for wood” (SABS 0183), *adhesion* is defined as:

“The state in which two surfaces are held together through molecular attraction (specific adhesion), or through chemical reaction between an adhesive and an adherent, or through an interlocking action (mechanical adhesion), or through a combination of any of these.”

As a phenomenon, adhesion and its application in gluing and wood finishing technology have received a lot of attention over the last few decades. By replacing, to a great degree, the more classical mechanical attachment techniques such as bolting, riveting, nailing and screwing, adhesion has gained a competitive advantage mainly because it saves weight, allows a more even stress distribution and probably most importantly, offers better aesthetics since the joint (glueline) is practically invisible. Another equally important field of adhesion application is in surface coatings, which include paints and varnishes (Schultz and Nardin, 1994).

Within the phase boundary there are two basic but very important systems that one has to distinguish in order to understand what happens in the phase boundary (Liptakova, *et al.*, 2000). One is the *solid – liquid* system, which is suitable when studying the phase boundary between wood and coating materials when they are applied and still in the liquid phase. The second is the system of *two solids* with different physical and chemical properties. In the second system, the phase boundary formed after the drying of the coating on the substrate surface. In the second system the adhesion of the solid coating to the wood as well as the cohesion of the basic constituents of the system, determines the properties of the newly formed system (Liptakova, *et al.*, 2000).

The phase boundary is a common term used when analyzing what happens between the wooden surface and the coating material in terms of adhesion. In all adhesion applications the phase boundary significantly influences the quality of the bond that develops between the solid and the adherent. Work done studying the nature of

intermolecular /surface forces in the phase boundary played a very important role in formulating and understanding concepts developed to evaluate the phase boundary. The interaction of surface forces on the phase boundary cause coating materials to adhere to and spread over the wooden surface (Liptakova, *et al.*, 2000).

### **2.2.1 A brief overview of the theories and mechanisms of adhesion**

Due to the variety of applications of adhesion in various scientific fields including macromolecular science, physical chemistry of surfaces and interfaces, material science, mechanics and micromechanics of fracture and rheology, various theories and mechanisms of adhesion have been developed to better understand each field of application. These include:

1. Mechanical interlocking
2. Electronic theory
3. Theory of boundary layers and interphases
4. Adsorption (thermodynamic) theory
5. Diffusion theory
6. Chemical bond theory (Schultz and Nardin, 1994).

Depending on the nature of the solids in contact and the conditions of formation of the bonded system, each of the theories given is valid to some extent.

#### **2.2.1.1 Mechanical interlocking**

This theory considers the mechanical keying or interlocking of the adhesive into the cavities, pores and asperities of the solid surface to be the most important factor to determine adhesive strength. However, when considering very smooth surfaces (ex. surfaces containing no pores, grooves or microscopic cavities) this theory cannot be considered universally applicable. Therefore, by improving both the surface morphology and physiochemical surface properties of the substrate and the adhesive, a high level of adhesion could be achieved. Fortunately, the morphology of wood allows mechanical interlocking to occur due to the vast numbers of pores



and cavities present at any wooden surface. By simply increasing the surface roughness, one can create improved wetting conditions to allow even better penetration of adhesive into the pores and cavities, thereby enhancing mechanical interlocking (Schultz and Nardin, 1994).

### **2.2.1.2 Electronic theory**

This theory rests upon the suggestion that the substrate and the adhesive are involved in an electron transfer mechanism that has different electronic band structures. It could result in the formation of a double electrical layer at the interface and, therefore, the adhesive-substrate layer can be analyzed as a capacitor. The separation of the two “plates” of the capacitor leads to an increasing potential difference until a discharge occurs when the system fails. It is, therefore, considered that the attractive electrostatic forces across the electrical double layer create the adhesive strength. But, after intensive research it was found that the electrical phenomena often observed during failure processes are the consequence rather than the cause of high bond strengths (Schultz and Nardin, 1994).

### **2.2.1.3 Theory of weak boundary layers (WBL): Concept of interphase**

The formation of an interfacial zone exhibiting properties that differ from those of the bulk materials can be brought about by alterations and modifications of the adhesive and/or adherent. The main factor in determining the level of adhesion even when failure appears to be interfacial is the cohesive strength of the weak boundary layer. This theory considers cohesive failure within the weaker material near the interface a more favourable event compared to a fracture along the adhesive-substrate interface.

The WBL theory has two main criticisms against it. Firstly, purely interfacial failure for many systems has been proven not to occur through experimental analysis. Secondly, the existence of a WBL can not be the cause of a cohesive failure in the vicinity of the interface in at least one of the materials in contact (Schultz and Nardin, 1994).

#### **2.2.1.4 Adsorption (thermodynamic) theory**

This theory is based on the belief that the adhesive will adhere to the substrate because of interatomic and intermolecular forces established at the interface, provided that an intimate contact is achieved. Van der Waals and Lewis acid-based interactions are the most common interfacial forces. The surface free energies of both the adhesive and adherent will determine the magnitude of these forces. The criteria of good adhesion essentially become criteria of good wetting because, generally, the formation of an assembly goes through a liquid-solid contact step and although this is a necessary condition, it is not a sufficient condition.

According to the generalized Lewis acid-base concept, the electron acceptor and donor interactions could be a major type of interfacial force between the adhesive and the substrate. Hydrogen bonds, which are often involved in adhesive joints, can be incorporated into the approach (Schultz and Nardin, 1994).

#### **2.2.1.5 Diffusion theory**

The diffusion theory of adhesion is based on the assumption that mutual diffusion (inter-diffusion) of macromolecules across the interface creates the adhesion strength of polymers to themselves (auto-hesion) or to each other. This mechanism implies that the macromolecular chains or chain segments are sufficiently mobile and mutually soluble. The mechanism of adhesion implied is very important especially in the field of welding.

The diffusion phenomena do contribute a great deal to the adhesive strength involving polymer-polymer intersections. However, sufficient solubility of both polymers and sufficient mobility of the chains is required for the inter-diffusion of macromolecular chains (Schultz and Nardin, 1994).

#### **2.2.1.6 Chemical bonding theory**

The contribution to the level of adhesion between both materials can significantly be improved by chemical bonds that form across the adhesive-substrate interface.

These bonds are generally called *primary bonds* where Van der Waals forces are called *secondary force interactions*. The difference merely exists on the basis of the relative strength of the two types of bonds. The strength of Van der Waals forces generally does not exceed 50 kJ/mol whereas the strength of covalent bonds (primary bonds) is in the order of 100 to 1000 kJ/mol.

Importantly, the reactivity of both the adhesive and the substrate will determine the formation of chemical bonds. Coupling agents, or adhesion promoting molecules, are used to improve the joint strength between the adhesive and the substrate and are the most important adhesion fields involving interfacial chemical bonds. Coupling agents are able to react chemically on both ends, with the polymer on one end and the substrate on the other, thereby creating a chemical bridge.

No single model or theory can describe the complex field of adhesion. However, it is generally accepted that the adsorption or thermodynamic theory defines the main mechanism promoting the widest applicability. It describes the development of physical forces at the interface and the achievement of intimate contact. Both are important in achieving subsequent interlocking, inter-diffusion and chemical bonding, thereby further increasing the adhesive strength. One can finally consider that three terms relating, respectively, to (1) the interfacial molecular interactions, (2) the mechanical and rheological properties of the bulk materials, and (3) the characteristics of the interface express the measured adhesive strength of an assembly (Schultz and Nardin, 1994).

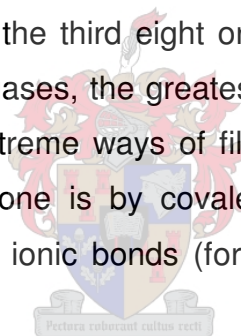
### **2.2.2 The nature of atomic and molecular forces**

Most of the phenomena involved in adhesive and surface coating technology have their origin on an atomic and molecular level of surface interaction. Therefore, a good understanding of the type of forces involved in the chemical bond at atomic and molecular level is necessary, because it not only covers the basics of adhesion in adhesive technology but also incorporates the background of the processes involved in surface coating technology. As seen from the previous section, various factors

play a role in creating a good bond, and understanding the role atomic and molecular forces play in the adhesive process, is probably the most important of those.

Molecules have intramolecular attractive and repulsive forces that originate from the electron charge and spin. Repulsive forces exist because electrons tend to stay as far apart as possible due to their like charges and, if unpaired, because of their like spin. Attractive forces, on the other hand, exist because electrons attracted by atomic nuclei due to their opposite charge tend to occupy the region between two nuclei, while opposite spin permits two electrons to occupy the same molecular orbital (Collett, 1972).

When discussing the chemical bond one has to visualize a positively charged nucleus surrounded by electrons arranged in concentric shells or energy levels. Each shell can accommodate a certain maximum number of electrons: the first shell can take two, the second eight, the third eight or eighteen, and so on. When the outer shell is full, like with inert gases, the greatest stability is reached. If the outer shell is not full there are two extreme ways of filling it and each way result in the formation of a chemical bond: one is by covalent bonds (formed by sharing of electrons) and the second is by ionic bonds (formed by the transfer of electrons) (Collett, 1972).



The covalent bond is a very important type of bond in adhesion. The sharing of electrons occurs if an electron orbital of one atom overlaps an orbital of another. For this to take place, each atomic orbital must have only one electron so that when the bond initiates, the two atomic orbitals merge together to form a single bond orbital that contains both electrons. The formation of a covalent bond leads to the development of energy.

Polarity exists in covalent bonds of which the two nuclei do not share the electrons equally. This results in one end of the bond being relatively negative and the other end relatively positive, therefore, a resultant negative and positive pole. This type of bond is called a polar bond or said to possess polarity. These polar bonds result in the formation of polar molecules and subsequently determine the melting point, boiling point and solubility of solutions. If the center of negative charge does not

coincide with the center of positive charge a molecule has got polarity and is called *dipole*; two equal and opposite charges separated by space.  $H_2$ ,  $O_2$  and  $Br_2$  are molecules with zero dipole moments and are non-polar because the two identical atoms have the same electronegativity and share electrons equally (Collett, 1972).

The transfer of electrons results in the formation of ionic bonds, but they are not as important in adhesion as the covalent bond. Sodium chloride is an example of an ionic compound, because sodium has one free electron in its outer valance shell and chlorine needs one electron to fill its outer valance shell. Therefore, sodium chloride is formed by the transfer of sodium's one electron to chlorine's outer valance shell resulting in sodium carrying a positive charge and chlorine carrying a negative charge. The ionic bond is thus the electrostatic attraction between oppositely charged ions (Collett, 1972).

Atoms (structural units of the molecule) are held to each other by covalent bonds in compounds not held together ionically. The forces holding molecules (the structured atoms) together in non-ionic compounds are intermolecular and generally much weaker than the forces holding ions to each other. *Dipole-dipole interactions* and *van der Waals forces* are two kinds of intermolecular forces. The attraction of the positive end of one polar molecule to the negative end of another polar molecule results in *dipole-dipole interactions*. Hydrogen chloride is an example of where the relatively positive hydrogen of one molecule is attracted to the relatively negative chlorine of another. Polar molecules are generally held to each other more strongly than non-polar molecules as a result of dipole-dipole interaction. The *hydrogen bond* is the most powerful kind of dipole-dipole interaction.

Van der Waals forces exist between molecules of non-ionic and non-polar compounds that can coalesce and solidify. Van der Waals forces have a very short range, acting between portions of different molecules which are in close contact, i.e., the surface of molecules (Collett, 1972).

### 2.2.3 The role and interactions of different forces in adhesive joints

In polymer molecules, the result of the interatomic forces is found to be maximized. The presence of molecular dipoles strongly affects the actual forces at play during the adhesive process. When two materials are held together at their surfaces, the force is referred to as an *adhesive force*. Adjacent molecules of a single material are held together by forces called *cohesive forces*. Unlike charge attractions between molecules result in the development of both adhesive and cohesive forces. The molecules will be closer together the more positive or negative the charged sites are and depending on the number of charged sites available. The adhesive joint will, therefore, fail either cohesively or adhesively depending on the strength of positive or negative charge and depending on the number of charged sites of the molecules and substrate (Collett, 1972).

### 2.2.4 Factors important for developing a strong bond

The development of a strong adhesive joint depends on three necessary requirements, namely (1) good wetting by the adhesive liquid, (2) solidification of the liquid adhesive and (3) sufficient deformability to reduce the effect of elastic stresses during the curing (Baier *et al.*, 1968).

#### 2.2.4.1 The wetting phenomena

A number of definitions were developed to describe the phenomena of wetting and Patton (1970) used the term wetting to describe the processes of *adhesion*, *penetration* and *spreading* which he said are three distinctly different types of wetting. *Adhesion* in this context simply means the wetting conditions that apply when “face to face contact” occurs and thus strictly is an interfacial phenomenon. Wetting conditions that are in play when a liquid works its way up along the walls of a solid capillary are referred to as *penetration* and wetting conditions involved when a liquid flows out over a surface are referred to as *spreading* (Collett, 1972).

*Adsorption* is another phenomena occurring during the adhesive process and the counter-part of the wetting process. *Adsorption* is defined as the adhesion of gases, vapours or liquids that are in contact with a solid surface, thus it is the adhesion of the molecules of these fluids to the surfaces of the solid bodies with which they are in contact. The main sorption process involved in the wetting process, however, is the up-take of a liquid by a porous solid within its gross capillary structure as a result of the action of surface tension forces of the liquid. This process is called *absorption*. Importantly, the phase involved in the wetting process during absorption, compared to the gases, vapours and liquids of adsorption, is only the liquid phase (pure liquids, solutions or dispersions).

The process of wetting involves the replacement of an area of the solid-air interface by an equal area of the solid-liquid interface. The quantitative measurement of the wetting process lies within the interface exchange and the most widely used method for quantifying the wetting potential of a solid surface is through the measurement of the *contact angle* and the best experimental approach to assessing the phenomena of wetting. The *contact angle* is an indicator of the attraction of a liquid to a solid surface. The measured contact angle reflects the equilibrium state that a drop of liquid reached after the three surface tensions acted upon it. The three surface tensions are (1) at the interface of the liquid phase and the vapour phase, (2) at the interface of the solid phase and the liquid phase and (3) at the interface of the solid phase and the vapour phase. The smaller the contact angle measured, the more a liquid has the tendency to spread and adhere to a solid surface and, therefore, the better the wettability of that surface (Collett, 1972). Also, the rougher a surface, the easier a liquid will wet the surface. That is why wood surfaces sanded with sanding paper have better wettability than wood only planed.

#### **2.2.4.2 Solidification of the liquid adhesive**

A very important factor involved in forming a good bond is the viscosity of the liquid adhesive used in the process. When a liquid adhesive is applied to a clean, smooth surface for the first time, it must be able to spread spontaneously, or spread when heat and pressure is applied. Therefore, the liquid adhesive must not be too viscous

otherwise the spreading will be affected. After the liquid adhesive has spread over the surface (either on its own or through the application of heat and pressure), the decrease in viscosity (or solidification) plays an important role. A liquid must solidify in order to function as an adhesive in the bonding process.

Polymers are the only class of materials that function as solidifying liquids. Polymer molecules have attractive charge sites along their length, are large and fairly brittle. Unlike charge attractions between molecules are the primary forces involved in adhesion and those solely involved in cohesion. Only polymers have enough cohesive strength to give adequate bonds and thus adhesive molecules must be polymeric. Polymer molecules are very big and have thousands of charged sites on their surfaces resulting in high cohesive and adhesive strength (Collett, 1972).

The process of increased viscosity or solidification of the polymer molecules occurs by one or a combination of the following processes:

1. curing or chemical polymerization to either a linear thermoplastic solid or a three-dimensional crosslinked solid, with or without use of heat and pressure;
2. hardening by physical cooling from the melt of liquid state, with or without pressure;
3. loss of a solvent (which may be water) by evaporation which is usually speeded up by the addition of heat; and
4. gelling of a dispersed polymeric solid.

It is very important to understand that the solidification process does not in any way increase the attractive forces present between the adhesive and the adherent molecules. This is because all work of adhesion occurs during the initial wetting spreading and the adsorption of the fluid on the solid surface (DeLollis, 1968).

### **2.2.4.3 Stress consideration in the adhesive joint**

Adhesives shrink during the solidification process resulting in the development of stresses. The extent of the stresses depends on the increase in the strength modulus of the cured resin and differences in the thermal coefficients of expansion of



the adhesive and the adherent. One of the primary factors causing bond degradation is internal stresses resulting from the solidification of an adhesive.

One of two mechanisms leading to joint failure is the subsequent forces involved in causing the release of interfacial stresses. The main cause of post-cure displacement forces working on the adhesive joint is water. In the adhesive process pressure is applied to improve the adhesive process, but result in *plastic* and *elastic deformation* at each point of contact. On release of the pressure the induced elastic deformation leads to the release of elastic strains adding to the stresses induced by the shrinking adhesive and act to weaken the bond. The problem of flexibility and toughness of the cured resin over a wider temperature and moisture range was addressed through the development of polymers with chemically advanced units within the molecules (Collett, 1972).

### **2.2.5 The role of surface contamination and importance of surface preparation**

Two concepts exist summarising the problems associated with attaining good adhesion. They are the concepts of intimate contact between the joined surfaces and the state of cleanliness (or contamination).

The section that dealt with the role and interactions of different forces in the adhesive joint, especially intra- and inter-atomic and molecular attraction provided the background necessary for discussing the two factors above. As mentioned earlier, the first rule for effective adhesion is attaining an intimate contact between the liquid and the solid surface at the interface. Adhesion is caused by forces between molecules in or near the surface of the two contacting materials and these forces are primarily Van der Waals forces. They are of such a small size that they are irrelevant until the “surfaces” of molecules are virtually in contact.

The harder and the denser a solid is and the higher its melting point the greater the surface free energy. Even on high-energy surfaces spontaneous wetting does not take place largely due to surface contamination. This dirt or contamination conceals

the true properties of the surface dealt with. The contact angle of the surface or the attractive force field is the main properties affected by contamination, which is directly connected to adhesion. Even the presence of one condensed, absorbed monolayer of organic compounds or coatings is sufficient to cause a major increase in the contact angle (Collett, 1972).

The most common compound in nature represented in the liquid and in the vapour phase, recognised as a contaminant, is water. It is present everywhere and has a limited permeability for most organic polymers. Even if absorbed liquids (such as adhesives, paints, etc) are bonded at the interface under optimum conditions for bonding, preferential desorption by water of that coating will take place due to the incredible affinity of water for most surfaces. A polymolecular adsorbed layer of water offers little resistance to shearing stresses and, therefore, will usually reduce the adhesive joint strength on the adherent interface. So, if an organic liquid adhesive is not capable of reacting with, dissolving or displacing the adsorbed water on the adherent surface while polymerizing, a weak joint will have formed after setting (Collett, 1972).

Natural contamination can occur on the surface of species containing high extractive content. Once the wood has been processed and the resin canals ruptured, the water-soluble resins/extractives migrate to the surface and inactivate the charged sites by reducing the wettability and changing the curing properties of the adhesives and retarding the hardening of finishes. The drying temperature determines the quantity of resins/extractives migrating to the surface because of solubility effects and the relative humidity conditions that determine the concentration gradient (Hse and Kuo, 1988). Hillis (1986) showed that water-repellent extractives affected the surface tension of the wood surface and in turn, affected the application and penetration of the adhesives that consequently affects bond strength.

Chen (1970) looked at the role extractives played in influencing the glue-wood bond. He showed that surface removal of extractives using a 10% sodium hydroxide solution resulted in significantly improved adhesive joint strength. He reported that the improvement was least for species that had the lowest extractive content. The wettability of the wood surfaces extracted improved and a significant increase in

bond strength was found with an increase in surface wettability. Hse and Kuo (1988) reported that the most common detrimental effect of extractives is the discoloration of finishes. They also said that not all effects are unfavourable and that redwood extractives appear to significantly improve the durability of the surface coating. Troughton and Chow (1971) looked at the role fatty acids played in *inactivation* of the wood surface after they migrated to the surface during drying at elevated temperatures (150°C). They found that most of the fatty acids that migrated to the surface had a chain length of less than 18 carbon atoms and according to previous research done by Hancock (1964) only fatty acids with a chain length of 18 and longer significantly influenced the bond strength. Troughton and Chow (1971) also found that no correlation existed between the amount of fatty acids (on the veneer surface) and bond quality.

Research done by Scharfetter (1971) on the effect of contamination on the gluing of wood showed that substances on the surface of wood acting as contaminants do not need to react chemically with the wood or the adhesive to impair the gluability of the wood. Confirming the statement made by Baier *et al.* (1968), Scharfetter said that prerequisites for the bonding of contaminated wood are preferential wetting of the wood by the adhesive and displacement of the contaminant. Scharfetter also said that liquid contaminants are displaced more readily and do not affect bonding as much as solids. Water being the most common liquid contaminant should be removed by an adhesive that displaces it chemically or reacts with it chemically and eventually forms a coating which polymerizes to seal the pores and form a strong adhesive bond (Collett, 1972).

Scharfetter (1971) also stated that non-polar contaminants are not adsorbed to the wood and if they are adequately transferable, can travel into the capillaries. If not adequately movable, they will remain on the surface of the wood and prevent adhesives from wetting the surface due to their hydrophobic nature.

Research on contamination of the adherent surface lead to the development of monomolecular coatings tailored with the right properties to promote adhesion by the same mechanism that undesirable monomolecular layers of contaminants alter

adhesion for the worse. These coatings are called *coupling agents* and act either through *chemical adsorption* (chemisorption) or through *physical adsorption*.

*Chemical adsorption* is the adsorption process when the binding of the adsorbate to the surface of the adsorbent is strong and the heat of adsorption is comparable to the heat of chemical reactions;

*Physical adsorption* is the adsorption process if the binding of the adsorbate to the surface of the adsorbent is weak and the heat of adsorption is of the same order of magnitude as the heat of condensation of a gas (Collett, 1972).

The extent, to which a chemical reaction at the joint interface is necessary to promote the formation of a strong joint, is not known. As mentioned in earlier sections, many researchers have long maintained that an intimate contact between the adhesive and adherent would be sufficient to develop an adequate high strength joint even without surface-chemical interactions. Therefore, a chemical reaction may not always be necessary seeing that the energy involved in the physical adsorption of the molecules of adhesive to the adherent is more than sufficient to form joints stronger than the bulk adhesive. However, it has become progressively more evident that a chemically-bonded adhesive-adherent interface is of great importance in bonds subjected to heat, water, exterior use or chemical attack.

Coupling agents consist of three parts: (1) one or more polar adsorbing groups capable at least of strong physical adsorption, preferably chemisorption on the adherent surface, (2) a hydrophobic spacer, and (3) a reactive, high-surface-energy group which can be readily wetted by resins. The most common coupling agents are alkoxy organosilanes and chloro-substituted carboxylic acids. The main idea behind the development of coupling agent molecules was to design a molecule sufficiently surface active to chemisorb onto the solid adherent surface while presenting an outermost surface on which the liquid adhesive could spread spontaneously (Collett, 1972).

Martens (1968) said that a coating of glue or paint (or varnishes) will fail by one or more of the following mechanisms if the surface contamination is not properly removed:

1. if the contaminant loses adhesion to the substrate, merely simple mechanical interference with the proper adhesive contact between coating and substrate will result in coating failure;
2. substrate corrosion and/or film degradation occurs when moisture or corrosive chemicals are absorbed through osmosis or by simple wicking action, and
3. electrochemical corrosion of ferrous surfaces may occur as a result of foreign matter, substrate flaws or breaks in the coating.

Efforts to minimize the effect of extractives on gluing and finishing include sanding the surface of the wood to be treated lightly and removing almost all types of surface contamination while at the same time increasing the wettability of the surface as well. Planing is an even more effective way of removing unwanted extractives and at the same time exposing a fresh and highly polar surface to which adhesives and finishes bond most effectively (Hse and Kuo, 1988; Scharfetter, 1971; Martens, 1968). Martens (1968) said that one or more of the following steps could be used to properly prepare a surface for gluing or coating:

1. removing loosely adhering foreign matter, corrosion products or old paint by hand methods (cloth or brush),
2. removing tightly adhering foreign matter that could loosen later and break adhesion or that could act as corrosion areas (power tools like planing),
3. producing a surface roughness to promote mechanical adhesion (etching, profile, anchor pattern), and
4. modifying the surface through chemical treatment to make it more compatible with the surface coating.

Research done by Suleman and Rashid (1999) looked at chemically treating the wood surface to improve wood finishing. Of the chemicals tested (sodium hydroxide (NaOH), aluminum chloride and ammonium chloride), sodium hydroxide in concentrations of 1% and 2% showed the best results. NaOH showed the absorption and penetration and the penetration improved with increase in

temperature. The protection of the wood against deterioration (photochemical discoloration due to sunlight) improved a lot.

### **2.3 Description of the two adhesives used in the experiments**

The two adhesives used in this investigation were polyvinyl acetate (PVAc) and urea formaldehyde (UF). They are the two most common glues used in the furniture industry and the research done here was aimed at improving the quality of the wood specifically for the furniture industry.

PVAc cures by means of a condensation reaction during which water evaporates from the resin. The curing process can be accelerated by adding heat. PVAc is produced as a two-part system having relatively low resistance to moisture, and creep can occur in joints over several weeks or months when continuously loaded. With addition of a catalyst to specifically formulated PVAc resins, a high degree of cross linking is induced in the glue system as it dries, giving the system considerable water resistance and making it less susceptible to thermal softening. Only after 14 days of application will one achieve maximum strength and water resistance. Even with added water resistance and strength, PVAc resins remain restricted to non-structural uses like joinery and kitchen furniture due to their inferior load bearing capability (Dinwoodie, 1983).

Urea formaldehyde (UF) was developed in the early 1930's and has been used for a variety of applications ranging from bonding solid wood, plywood and particularly different types of particleboard. It is used in most countries solely for particleboard manufacturing where the use of UF resins for the production of plywood is limited to northern and southern Asian countries. The resin primarily consists of urea and formaldehyde and cures through the process of chemical reaction in contrast to PVAc that cures by means of a condensation reaction. Urea is commercially produced through the reaction of ammonia and carbon dioxide at temperatures ranging from 135-200°C and under pressures ranging from 70-230 atmospheres (1 atmosphere = 101.325 kPa). Formaldehyde is produced by the oxidation of

methanol, which is commercially prepared from carbon monoxide and hydrogen or petroleum (Dinwoodie, 1983).

The molar ratio of formaldehyde to urea varies from 1.2:1 to 2.0:1, but, due environmental legislation restricting the emission of formaldehyde, it is now common practice to use resins containing a molar ratio closer to the lower end of this range. Ammonium chloride or ammonium sulphate are commonly used as catalysts (hardener) to cure UF at elevated temperatures. When using UF resins for room temperature curing, organic acids such as citric, formic or tartaric acid are used as the catalyst and only applied to one of the surfaces while UF is applied to the other. This process is usually used in the production of window frames and gluelam beams (Dinwoodie, 1983). Research done by Vermaas (1966) showed that one could use thermosetting UF but that one had to wait two weeks for the polymerization process to finish before shear tests could be performed.

One of the most important aspects contributing to the success and process of UF gluing is the molar ratio of formaldehyde to urea. Resins with a high F/U molar ratio (1.8:1 - 2.0:1) have a lower viscosity, higher water resistance (durability), higher strength and stiffness and a quicker curing time, but a shorter pot life (i.e. shorter gelation time) and a higher free formaldehyde content than resins with a low F/U molar ratio (1.2:1 – 1.6:1) (Dinwoodie, 1983).

Adhesive technology has led to the development of adhesives that are generally always stronger than the wood it adheres to. Therefore, when testing glues, the values obtained from load failures depend more on the strength of the wood than on the adhesive and cohesive strength of the glue. When testing gluelines, shear tests generally produce higher failure values than tension tests and even higher values than cleavage tests (Vermaas, 1966). Therefore, according to Stanger *et al.* (1965), when investigating the strength of the glue and not the wood, one would rather make use of tension and cleavage tests.

Vermaas (1966) investigated wood failure as an indication of the strength of the glue bond. He said that the breaking loads of a mechanical test and the percentage wood failure are normally the two ways used to indicate glue-bond strength. When using

percentage wood failure as an indication of glue-bond strength it is important that one should compare results within one species. This is important, seeing that density differences will result in one species having more wood failure than the other while the bond strength could be exactly the same for the species with a lower percentage wood failure.

Bergin (1953) studied the mechanisms of bond failure and identified four distinct types of bond failures helping in assessing the glue-bond strength:

1. **Low failing load and a low percentage wood failure** – this result usually indicates that a poor quality glue or gluing technique was used.
2. **Low failing load and a high percentage wood failure** – this implies that the glue is stronger than the wood, but not necessarily a high quality glue. The bond strength is unknown due to the weakness of the wood.
3. **High failing load and a low percentage wood failure** – an indication of a relatively good joint or that strength of the glue and wood are similar.
4. **High failing load and a high percentage wood failure** – indication of a high quality glued joint.

Percentage wood failure alone is not a reliable means of assessing the quality of a joint, because a multitude of factors influence the percentage wood failure.

## 2.4 Summary of aspects directly related to the research

### 2.4.1 Surface checking:

The occurrence and severity of surface checking as a drying defect can be controlled to a large degree through schedule adaptation. The first stage of drying (from green to about 60% moisture content {MC}) is crucial in controlling the development of an excessive moisture gradient between the surface and the core, which controls stress development in the surface layer. Only once the timber has reached an average moisture content of 30-40% can one increase the drying rate by increasing the temperature.



It is important not to expose stacks of green timber before kiln drying to environments that promote drying. Most of the surface checks can occur in lumber standing in front of the kiln waiting to be dried. It is important to get the stacks into the kiln as soon as possible to ensure that not too much moisture is lost from the surface that could lead to stresses and subsequent checks. Controlling the environment in and around the stack will ensure that the optimum kiln schedule can produce good quality dried timber. Merely spraying the stacks with water, using fine mist sprays, will ensure that the relative humidity is kept high enough not to promote rapid drying conditions.

Differences between species play a significant role in determining the optimum schedule. Because of different chemical and morphological compositions, different species will react differently to different schedules. For instance, *P. elliotii* has a greater tendency to develop yellow- and brown stain than *P. patula* or *P. taeda* at higher drying temperatures, because *P. elliotii* has a higher resin content than the other species.

The key to producing good quality dried timber lies in developing a schedule that produces wood that meets customer specific needs and requirements. It is, therefore, necessary to understand the needs of our customers and what they can expect from the production process. Once you know what their needs and requirements are, one must, through trial and error, determine the optimum schedule required to produce timber of the quality expected by your customers. The end-use of timber plays a crucial role in determining the schedule used to dry the wood. For instance, furniture timber has very strict quality requirements and the schedule used to dry furniture grade timber has to be “soft” so that no or very little surface checks develop and that no yellow or brown discoloration occurs.

Stress relaxation in furniture grade lumber during the final step in drying, namely conditioning, is very important. During the drying process stresses develop in the timber due to the way wood shrinks as it loses moisture. At the end of the drying run the surface is normally under a compression stress and the core under a tensile stress. If these stresses are not relaxed, further processing will result in pinching of the saw blades and distortion of the wood. Even worse scenarios due to the axe-

handle phenomenon could cause wood to reveal hidden checks and even cause checks to expand depending on the amount of moisture it is exposed to. This phenomenon is especially prevalent in hardwoods.

The bulking of fibers holds a lot of promise in controlling and even totally eliminating shrinkage and, therefore, also the development of surface checks. Treating wood with bulking chemicals intended for high value applications (like gunstocks) or to protect it from moisture if exposed to excessively fluctuating environments (like in bathrooms or kitchens) could be feasible and should be considered as an alternative to schedule adaptation for small scale productions. The reduction in hygroscopicity induced by some of the treatments could hold a lot of value for exterior use as well.

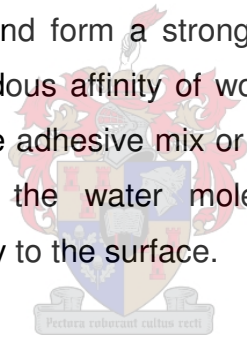
The use of high temperature schedules (100 °C) has in recent years found a lot of useful application. Unfortunately, due to the polymerization effect (yellow and brown stain) brought about by the high temperatures, using high temperature schedules for the drying of high quality timber intended for furniture production, has not been successful. The role high temperatures play in controlling the development of surface checks is attributed to the plasticization of the lignin induced by the high temperatures. As soon as the lignin is softened, the fibers are able to creep (slip) past each other and allow the stresses, due to shrinkage, to be absorbed in the fibrous structure.

#### **2.4.2 The adhesion phenomena:**

Probably the two most important aspects with regards to the adhesion phenomena and the development of a strong bond are wettability and cleanliness. The most important aspect determining the wettability of a surface is the surface roughness. The rougher a surface the better its wettability and the better the spreading and absorption of a liquid by the surface. That is why a planed surface is not as rough as a sanded one and the sanded one has a better wettability. Viscosity also plays a role when it comes to the wettability of a surface. The lower the viscosity the easier the liquid will spread over the surface and migrate into the capillaries.

Contamination of the glued surface is detrimental to the strength of the joint. Contamination not chemically bound to the surface can be removed if it is made mobile enough so that sufficient wetting can occur. Contamination that is chemically bound to the surface can only be removed by sanding or planing the surface exposing a fresh, polar surface to which a coating or adhesive will properly bond to. Otherwise a coupling agent can be used to connect the polar coating/adhesive to the non-polar contaminant on the surface. Extractives are natural contaminants that occur on most wood surfaces. They are usually water soluble and migrate to the surface during the drying process. They can simply be removed by sanding or planing the surface or by “washing” it with a sodium hydroxide solution of 1% or 2%.

Water is possibly the most common contaminant found basically everywhere. The only way this contaminant can be removed is if one uses an adhesive that displaces it chemically or reacts with it chemically and eventually forms a coating which polymerizes to seal the pores and form a strong adhesive bond. In the case of gluing and finishing, the tremendous affinity of wood for water forces one to make sure that the water content of the adhesive mix or coating is not too high, otherwise the sorption sites will attract the water molecules first and not allow the adhesive/coating to bond properly to the surface.



Percentage wood failure alone is not a good enough indication of the strength of the joint. The maximum load failure measured in either Newton (N) or kilogram per square centimeter ( $\text{kg}/\text{cm}^2$ ) combined with percentage wood failure gives a good indication of the strength of the glue and the bond. Generally a high percentage wood failure gives a good indication that the glue bond is stronger than the material it is bonding, but does not always tell us whether the glue used is high quality glue. When it is coupled with a high failure load it shows that it is a very strong joint held together by a very good adhesive. Also, a low percentage wood failure coupled with a low failure load is an indication that a very weak joint has formed either through using poor glue or using the wrong gluing technique.

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**Chapter 3: Effect of sodium lauryl sulphate (SLS) -  
treatment of green lumber on surface mould and blue stain  
development**

**ARTICLE TO BE SUBMITTED FOR PUBLICATION**



# Effect of sodium lauryl sulphate (SLS) - treatment of green lumber on surface mould and blue stain development

## 3.1 Abstract

Wet-off-saw *P. radiata* boards (38×114×1000mm) were sprayed with different aqueous solutions of sodium lauryl sulphate (SLS) and either open-stacked or close-stacked and evaluated for blue stain and mould control over two, three and four weeks. A SLS concentration as low as 0.1% effectively controlled surface mould and blue stain development for 3 weeks on open-stacked boards. When close-stacked, a 0.2% SLS solution effectively controlled blue stain development over a two week period.



## 3.2 Introduction

Blue stain and mould is a common problem for softwood sawmills, particularly during the warm, humid months of the year when the infection rates are the highest. To control blue stain, felled logs have to be swiftly transported and sawn so that the lumber can be dried before the fungi gets a chance to spread through the wood (Uzunovic *et al.*, 1996). Especially freshly sawn, close-stacked boards, should not be left in front of the kiln too long.

Many different chemicals have been developed for the control of blue stain and mould growth on wood. In the past, some of these chemicals were highly toxic, but recent environmental awareness has resulted in a continuing drive to find chemicals and treatment methods that are less harmful to the environment and humans.

In this study, the effect of sodium lauryl sulphate (SLS) on the growth of mould and blue stain was investigated. SLS is commonly used in microbiology as an agent of cell lysis and protein denaturation (Helenius and Simons, 1975; Madden, 1986; Jones *et al.*, 1987; Seddon *et al.*, 2004) and could thus potentially inhibit the growth of fungi on wood. It is non-toxic, degradable by bacteria, and also a common detergent that is present in many household cleaning products.

## 3.3 Materials and methods

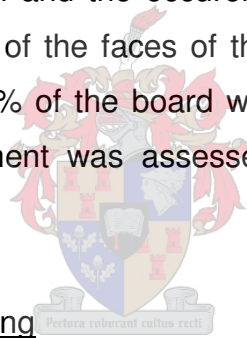
### 3.3.1 Experiment 1: Open-stacking

Three groups, each consisting of ten wet-off-saw *P. radiata* replicate boards (38×114×1000mm), were sprayed with sodium lauryl sulphate (Ionic surfactant, Merck Chemicals, SLS assay: 90% minimum) with solution concentrations ranging from 0.05% (pH 7.1) , 0.5% (pH 7.6) to 5% (pH 7.8). The measured pH values varied significantly. The higher concentrations yielded pH values of 7.5-9 for a single solution concentration. Another group of ten was sprayed with water (pH 6.6) to function as a control group to indicate the possible effect of water as a solvent alone.

A fifth group was not treated, and also functioned as a control group. Thus, the five experimental groups were:

1. No treatment - First control group.
2. Sprayed with water - Second control group.
3. Sprayed with 0.05% SLS solution
4. Sprayed with 0.5% SLS solution
5. Sprayed with 5% SLS solution

The five groups (of ten boards each) were open stacked in a room with ambient conditions conducive to bluestain and mould development. After a period of two weeks, the mould growth on the faces was measured visually. Two 2 mm was then planed off the two faces of each board to also visually assess the amount of bluestain. For this quantitative measurement, the face of each board was divided into 10 sections along the length and the occurrence of surface mould and/or blue stain was noted in each section of the faces of the board. E.g. if three of the ten sections contained bluestain, 30% of the board was judged to be bluestained, etc. The degree of mould development was assessed in the same manner prior to planing the boards.



### 3.3.2 Experiment 2: Open-stacking

This experiment was a follow-up of the first to be able to make adjustments to rectify some of the shortcomings of the first. The aim was to find the lowest effective SLS concentration. An attempt was also made to select boards that contained mostly sapwood and no reaction wood, since these factors influence bluestain development. To lessen the possibility of “premature” bluestain infection, 0.5 m of each end of the original 3 m boards were cut off, used for moisture content determination and then discarded.

SLS treatments were done with concentrations of 0.1%, 0.2% and 0.3%. To eliminate the effect of pH, another 0.3% SLS solution was prepared and the pH adjusted with acetic acid to pH 6.6, which is the same pH level as the water used in the experiment. The five experimental groups were treated as follows:

1. Sprayed with water - Control group
2. Sprayed with 0.1% SLS solution
3. Sprayed with 0.2% SLS solution
4. Sprayed with 0.3% SLS solution
5. Sprayed with 0.3% pH 6.6 SLS solution (pH adjusted with acetic acid).

The mould growth was assessed visually after three weeks and then 2 mm planed off the faces to visually evaluate the blue stain.

### 3.3.3 Experiment 3: Close-stacking

Three groups consisting of twenty *P. radiata* boards (38×114×1000mm) each were prepared. Once again, care was taken to select samples with a low probability of being infected by cutting 0.5 m off each end of the original 3 m boards.

One group was sprayed with 0.2% SLS, and another with 0.2% Triton X-100™ (t-octylphenoxypolyethoxyethanol, Sigma Chemical Co.) that was a non-ionic surfactant compared to the anionic nature of SLS. The third group was not treated.

The groups were treated as follows:

1. No treatment - Control group.
2. Sprayed with 0.2% SLS solution
3. Sprayed with 0.2% Triton X-100™

All three groups were close-stacked separately and covered with plastic and evaluated after two and after four weeks. For blue stain evaluation, they were not planed as in the previous two open stacked experiments.

### 3.4 Results and discussion

#### 3.4.1 Mould development

The effect of various SLS treatment solutions on the development of surface mould is listed in Table 1.

**Table 1 Effect of SLS treatment on the development of surface mould (n=10/group)**

Experimental conditions	Group #	Treatment	% of face area affected after		
			2 weeks	3 weeks	4 weeks
Open stacked	I	Control	<b>13.0</b>	n/a	n/a
	II	Water	<b>6.5</b>	n/a	n/a
	III	0.05% SLS	<b>26.5</b>	n/a	n/a
	IV	0.5% SLS	<b>0.0</b>	n/a	n/a
	V	5% SLS	<b>0.0</b>	n/a	n/a
Open stacked	I	Water	n/a	<b>22.0</b>	n/a
	II	0.1% SLS	n/a	<b>0.0</b>	n/a
	III	0.2% SLS	n/a	<b>0.0</b>	n/a
	IV	0.3% SLS	n/a	<b>0.0</b>	n/a
	V	0.3% pH6.6 SLS	n/a	<b>0.0</b>	n/a
Close stacked	I	Control	<b>4.0</b>	n/a	<b>45.8</b>
	II	0.2% SLS	<b>0.0</b>	n/a	<b>50.5</b>
	III	0.2% Triton X-100	<b>19.5</b>	n/a	<b>84.8</b>

n/a: data not available; boards were not evaluated for that period

As expected, the treatment of open stacked boards with water only, led to an increase in infection; from 6.5% after two weeks to 22% one week later. A comparison of the open and closed stacked controls after two weeks shows that,

probably because of the reduced presence of oxygen and retention of higher moisture contents, the closed stacked infection (4%), was not as much as that experienced under the open stacked conditions (13%). Extended close stacking of the controls from two (4%) to four weeks (45.8%), caused a noticeable increase in surface mould growth.

It appears that SLS concentrations from 0.1% and higher seem to work favourably but that an increase of concentrations >0.2% should be considered if longer protection periods are required.

After two weeks, the 0.2% Triton X-100 solution (74.8%) did not appear to have taken sufficient care of infection developing in close stacked boards.

### 3.4.2 Bluestain development

Table 2 Effect of SLS treatment on the development of blue stain (n=10/group)

Experimental conditions	Group #	Treatment	% of face area affected after		
			2 weeks	3 weeks	4 weeks
Open stacked	I	Control	<b>69.5</b>	n/a	n/a
	II	Water	<b>82.0</b>	n/a	n/a
	III	0.05% SLS	<b>95.0</b>	n/a	n/a
	IV	0.5% SLS	<b>9.0</b>	n/a	n/a
	V	5% SLS	<b>8.0</b>	n/a	n/a
Open stacked	I	Water	n/a	<b>97.5</b>	n/a
	II	0.1% SLS	n/a	<b>2.0</b>	n/a
	III	0.2% SLS	n/a	<b>7.5</b>	n/a
	IV	0.3% SLS	n/a	<b>4.5</b>	n/a
	V	0.3% pH6.6 SLS	n/a	<b>0.0</b>	n/a

Close stacked	I	Control	<b>68.3</b>	n/a	<b>96.3</b>
	II	0.2% SLS	<b>3.0</b>	n/a	<b>50.3</b>
	III	0.2% Triton X-100	<b>74.8</b>	n/a	<b>97.8</b>

n/a: data not available; boards were not evaluated for that period

It seemed as if water only and solutions with a low SLS concentration such as 0.05%, assisted in the penetration of the blue stain fungus into open stacked boards, producing values such as 82 and 95% after two and 97.5% after three weeks of close stacking. Similar order of magnitude values were measured in close stacked boards after two weeks (74.8%), increasing with stacking time (97.8%). Adjustment of pH apparently improved control from 4.5% down to 0% after three weeks.

SLS concentrations > 0.2% appeared to offer blue stain control for two weeks in closed stacked boards. However, this was not sufficient to offer protection for 4 weeks.

As with mould, the 0.2% Triton X-100 solution did not adequately control the development of blue stain fungi.



### 3.5 Conclusions

Blue stain and mould development were effectively controlled by SLS treatments. Concentrations as low as 0.1% yielded effective control for 3 weeks on open-stacked boards. When close-stacked, a 0.2% solution yielded control for 2 weeks. Higher SLS concentrations controlled bluestain and mould development on close-stacked boards for up to 4 weeks. SLS also yielded better protection than the non-ionic Triton X-100™.

SLS may be a viable non-toxic alternative for the short-term control of mould and blue stain. Possible application areas could include log storage, air-drying and green lumber storage.



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**Chapter 4: The influence of sodium lauryl sulphate (SLS) -  
treatment on the occurrence of surface checks in  
softwoods**

**To be submitted for publication in SA Forestry Journal**



# The influence of sodium lauryl sulphate (SLS) - treatment on the occurrence of surface checks in softwoods

## 4.1 Abstract

The number and length of surface checks was determined on the most tangential face or the face furthest from the pith in 700 mm x 114 mm x 38 mm *P. radiata* and *P. elliotii* boards after they were treated with aqueous sodium lauryl sulphate (SLS) solutions and dried using three kiln schedules.

According to statistical evaluation, SLS had no effect on the number of surface checks present in *P. radiata* and *P. elliotii* after kiln drying when using a 95% confidence level. When comparing the control groups of experiments that were dried using the same schedule (*P. radiata* and experiments EII 1, 3 and 4) a significant difference existed between the *P. radiata* control group and the three *P. elliotii* control groups in terms of the total number of checks, indicating the difference between the two species. This will influence the furniture industry's choice of species when selecting the most appropriate for furniture manufacturing.

When comparing the control groups of the five *P. elliotii* experiments, it was found that, although the number of checks decreased from the lower temperature schedule (experiment EII 2) to the higher temperature schedule (experiment EII 5), no statistical significant difference existed on a 95% confidence level. However, although Group II (a water-soaked sample) of experiment EII 5 and experiment EII 2 (non-soaked and covered sample) statistically differed from each other when using a 93% confidence level, one can't be 100% confident that high temperature drying can control surface checking because the samples were treated/handled differently and, therefore, warrants further investigation.

## 4.2 Introduction

Surface checks are one of the most common defects associated with the drying of softwoods. Although not always visible to the naked eye, they become more apparent when the lumber is planed and the surface is exposed to changing environments, especially changes in the relative humidity (Pratt, 1974). The development of surface checks is correlated with the moisture gradient that exists between the surface and the core (Schniewind, 1963). Surface checks develop during the early stages of drying when the surface is placed under a tensile force as the surface wants to shrink towards the core but as the core is still green and fully swollen, this is impossible. When these tensile forces exceed the mechanical strength of the timber, checks develop in the surface (Rice, 1994). The length and the depth of these checks are determined by the drying conditions – the faster the drying rate, the larger/steeper the moisture gradients between the core and the surface, and the more severe the checking. The development of surface checks is the main factor limiting the use of accelerated drying schedules or high temperature drying (Gaby, 1963).

Because surface checking also occurs during air drying (Simpson, 1991), an important factor governing the development of surface checks is the time stacked green lumber spends in front of the kiln door. When wood is exposed to air that has a lower moisture content than the wood it will try to equalize and, therefore, it will lose moisture from the surface and, as Wiberg and Morén (1999) had shown, the wet line (from where the water evaporates) recedes to just below the surface. Early moisture loss from the surface is critical, because when the surface reaches FSP it will start shrinking and stresses will develop in the surface that will cause surface checking. It was shown that air-dried lumber is of lower quality (more checks) (Rucker and Smith, 1961) than kiln-dried (Wynands, 1963) and solar-dried lumber (Oliveira, Skaar and Wengert, 1982).

It has been shown that sodium lauryl sulphate (SLS) treatment can provide short-term control of mould and blue stain growth on open-stacked green softwood (Perold, 2006). However, SLS treatment could also influence the moisture gradient in

drying lumber (Scheepers, 2006) and thus yield a poorer (or improved) surface quality, especially in regard to surface checking. Consequently, the benefit of fungal control could be offset by more surface checking.

The aim of this investigation was to ascertain the influence of SLS treatment on the development of surface checks during kiln drying of two SA pine species using different kiln schedules.

### 4.3 Materials and methods

#### *Sampling and preparation*

Rough sawn, 2.4 m x 114 mm x 38 mm *Pinus radiata* boards and *Pinus elliottii* boards were sourced from the Southern Cape plantations. Approximately 10-15 cm was cut from one end of every board to remove wood that could possibly have lost some water in transit and/or during storage. Test samples, 0.7 m in length, as well as moisture content samples (used to determine oven-dry weight of each 0.7 m sample) were cut from the boards. Boards were end-sealed with a liquid polyurethane-waterproofing compound and randomly divided into four groups having twenty 0.7 m replicates each.

#### *SLS treatment*

The different sodium lauryl sulphate (SLS) treatment concentrations (on a mass to mass ratio) and procedures for all the different experimental groups are listed in table 1 below.

**Table 1 SLS treatments of different experimental groups**

Species	Treatment			Drying Schedule
	Group #	Solution	Application	
<i>P. radiata</i>	I	Control <sup>1</sup>	Covered <sup>2</sup>	Schedule 1
	II	0.05% SLS	Sprayed <sup>3</sup>	
	III	0.5% SLS	Sprayed	
	IV	5% SLS	Sprayed	

<b><i>P. elliotii</i></b> <b>(EII 1)</b>	I	0.5% SLS	Dipped <sup>4</sup>	Schedule 1
	II	Control	Covered	
	III	0.5% SLS	Soaked <sup>5</sup>	
	IV	H <sub>2</sub> O	Soaked	
<b><i>P. elliotii</i></b> <b>(EII 2)</b>	I	Control	Covered	Schedule 2
	II	H <sub>2</sub> O	Soaked	
	III	0.5% SLS	Dipped	
	IV	0.5% SLS	Soaked	
<b><i>P. elliotii</i></b> <b>(EII 3)</b>	I	Control	Covered	Schedule 1
	II	1% SLS	Dipped	
	III	0.5% SLS	Soaked	
	IV	1% SLS	Soaked	
<b><i>P. elliotii</i></b> <b>(EII 4)</b>	I	Control	Covered	Schedule 1
	II	5% SLS	Dipped	
	III	2% SLS	Soaked	
	IV	5% SLS	Soaked	
<b><i>P. elliotii</i></b> <b>(ELL 5)</b>	I	H <sub>2</sub> O	Soaked	Schedule 3
	II	H <sub>2</sub> O	Soaked	
	III	2% SLS	Soaked	
	IV	5% SLS	Soaked	

<sup>1</sup>Control: These boards were not treated

<sup>2</sup>Covered: These boards were wrapped in plastic until the drying runs commenced

<sup>3</sup>Sprayed: These boards were sprayed with an excess (formulation ran off surface) SLS formulation.

<sup>4</sup>Dipped: These boards were wrapped in plastic for three days and then, just before the drying run, dipped for 10 seconds in the different SLS formulations.

<sup>5</sup>Soaked: These boards were kept in either water (H<sub>2</sub>O) or the SLS formulations for three days.

In the fifth *P. elliotii* experiment (EII 5), because bluestain already infected some of the boards in each group, both Group I and II acted as control groups and were soaked in water for three days. A control group that was kept under a plastic cover for three days in the humid conditions that existed at that time, to ensure as little as possible moisture was lost, would only have made the infection worse and influenced the results.

### *Drying*

Tables 2, 3 and 4 contain the three different kiln schedules used. All boards were stacked using 25mm stickers.

**Table 2** Schedule 1 used for the *P. radiata* and the first, third and fourth (Ell 1, 3 + 4) *P. elliotii* experiments

Drying phase	Dry Bulb (°C)	Wet Bulb (°C)	RH (%)	EMC (%)
Warm up 240 min	90	87	89	14.3
Green to 12%	90	60	25.4	3.1
Equalize 300 min	90	87	89	14.3

**Table 3** Schedule 2 for the second *P. elliotii* (Ell 2) experiment

Drying phase	Dry Bulb (°C)	Wet Bulb (°C)	RH (%)	EMC (%)
Initial MC – 10%	60	50	58	8.9
Initial MC – 20%	62	50	53	7.9
Initial MC – 30%	65	50	44	6.8
Initial MC – 40%	68	50	39	5.8
Initial MC – 50%	72	50	32	4.8
To 11 %	75	45	19.5	3.0
Equalize 300 min	75	68	72.5	10.6

**Table 4** Schedule 3 used for the fifth *P. elliotii* (Ell 5) experiment

Drying phase	Dry Bulb (°C)	Wet Bulb (°C)	RH (%)	EMC (%)
Warm up 240 min	90	87	89	14.3
Green to 11%	110	100	70.2	7.3
Equalize 300 min	90	87	89	14.3

### Surface check evaluation

After drying, 2 mm were planed off the most tangential surface or the surface furthest from the pith of each board. The boards were then re-wetted by immersing them in a water bath for about ten seconds so that only the surfaces became fully saturated. The boards were then left outside in the sun to allow the surface of the board to dry completely so that the surface checks could open fully (ZA Dry-Q Kiln Drying Management System, 2005; Hart, Gilmore and Wyatt, 1992). The surface check lengths were measured with a ruler and classified and counted using a classification system that entailed grouping the checks in 20 mm groups so that checks having a length of 0 – 19 mm would fall in the 0 – 19 mm group and checks having a length of 20 – 39 mm would fall in the 20 – 39 mm group, etc.

## 4.4 Results

### 4.4.1 Results of SLS treatment on the development of surface checks

The data of all the experiments were not normally distributed (see Appendix A) and, therefore, a normal statistical analysis of variance (ANOVA) could not be performed. The Kruskal-Wallis ANOVA was used to determine whether any significant differences existed between the different groups because it was the most suitable statistical evaluation for these data sets. In the Kruskal-Wallis test, the boards in each sample were ranked in terms of the total number of checks occurring in each board. The least checked board was ranked first, the second least checked board second, and so forth. Then the average rank of the boards of each sample was determined and compared with each other. The probability (p-value) that the difference in ranks between the data sets occurred by chance (the null hypothesis) was then determined. If the p-value was less than 0.05, it indicated that the null hypothesis could be rejected at a 95% confidence level and that the differences were not due to chance, but due to the different treatments. The bootstrap analysis (see Appendix B for description) was used to determine between which groups the differences existed and whether the differences were statistically significant. The bootstrap was done whether the Kruskal-Wallis showed a statistical difference between the groups or not. This was merely precautionary to ensure all possible human errors were excluded. A Bonferroni test was applied to the bootstrap data to indicate any between group differences and by doing so indicate whether the treated groups differed from the control groups.

All the raw data (number of checks per length group) used in the statistical analysis is given in Appendix C. Table 5 contains a summary of the statistical results and Appendix D contains all the raw data from the statistical results. In Table 5 the Bonferroni p-values are the results of a comparison of the control group and the main treated group, which is the group that was exposed to the treatment the longest (soaked for three days) and using the highest concentration for that specific experiment.



**Table 5** Statistical results (p-values) of all the experiments on a 95% confidence level

Experiment	Kruskal-Wallis Anova	Bonferroni result
<i>P. radiata</i>	0.8766	1.0000
<i>P. elliotii</i> (EII 1)	0.3071	1.0000
<i>P. elliotii</i> (EII 2)	0.7871	1.0000
<i>P. elliotii</i> (EII 3)	0.7465	1.0000
<i>P. elliotii</i> (EII 4)	0.9347	1.0000
<i>P. elliotii</i> (EII 5)	0.2109	1.0000 & 0.5579

#### 4.4.2 Comparing results obtained from the control groups for within and between species differences

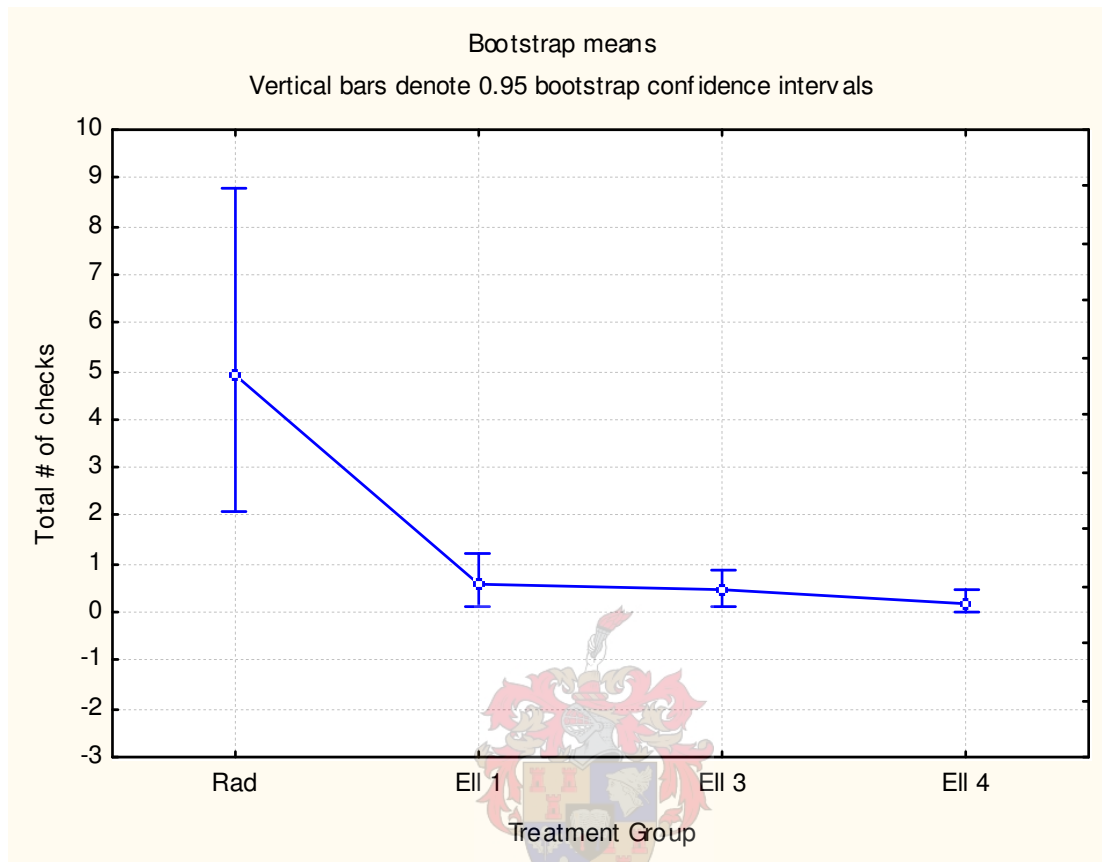
Interesting results were obtained from the control groups of all six experiments. The number of surface checks, obtained from the control groups of four of the experiments (*P. radiata* and 3 *P. elliotii* experiments), was used to compare these two pine species because all four experiments were dried using the same kiln schedule. Table 6 provides the number of checks for each surface check length group as well as the total number of checks for the four experiments.

**Table 6** Comparison of the number of surface checks in controls of two different pine species when using the same kiln schedule (schedule 1)

Check length	<i>Experiments</i>			
	<i>P. radiata</i>	EII 1	EII 3	EII 4
0-19mm	15	5	1	0
20-39mm	36	4	7	2
40-59mm	23	1	1	1
60-79mm	10	2	0	1
80-99mm	7	0	0	0
100mm+	8	1	0	0
<b>Total</b>	<b>99</b>	<b>13</b>	<b>9</b>	<b>4</b>

The Kruskal-Wallis p-value of 0.0110 indicates that, at a 98% confidence level, the differences between the four control groups were not due to chance. The bootstrap analysis (see Figure 1) confirmed that the *P. radiata* control group statistically

differed from all three *P. elliotii* control groups. A Bonferroni test was used on the bootstrap data to produce the multiple p-values in Table 7.



**Figure 1** Bootstrap results of the *P. radiata* and the three *P. elliotii* (EII 1, 3 + 4) control groups using a 95% confidence level to determine significant differences

**Table 7** Multiple p-values obtained from the Bonferroni statistical test that was applied to the bootstrap data of Figure 1

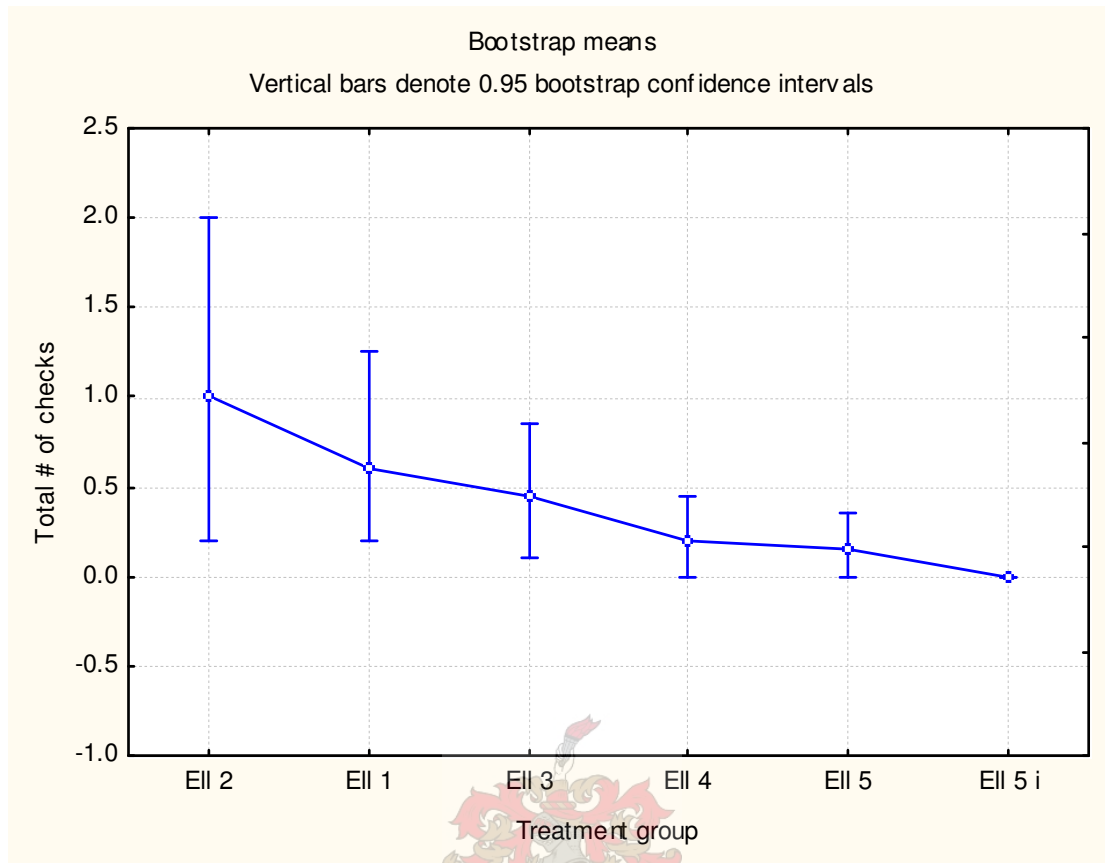
Experiment #	<i>P. radiata</i>	<i>EII 1</i>	<i>EII 3</i>	<i>EII 4</i>
<i>P. radiata</i>		<b>0.0043</b>	<b>0.0025</b>	<b>0.0013</b>
<i>EII 1</i>	<b>0.0043</b>		1.0000	1.0000
<i>EII 3</i>	<b>0.0025</b>	1.0000		1.0000
<i>EII 4</i>	<b>0.0013</b>	1.0000	1.0000	

Table 8 shows the number of surface checks for the control groups of the five *P. elliotii* experiments of which boards from experiment EII 1, 3 + 4 were dried using the same schedule (Table 2), boards from experiment EII 2 were dried using the lower temperature schedule in Table 3 and boards from experiment EII 5 (both Group I and Group II) were dried using the higher temperature schedule in Table 4.

**Table 8** Comparison of effect of different schedules on the number of surface checks in controls of the five *P. elliotii* experiments

Check length	Experiments					
	EII 2	EII 1	EII 3	EII 4	EII 5 (I)	EII 5 (II)
0-19mm	5	5	1	0	1	0
20-39mm	8	4	7	2	1	0
40-59mm	5	1	1	1	1	0
60-79mm	2	2	0	1	0	0
80-99mm	1	0	0	0	0	0
100mm+	0	1	0	0	0	0
<b>Total</b>	<b>21</b>	<b>13</b>	<b>9</b>	<b>4</b>	<b>3</b>	<b>0</b>

Although the total number of surface checks decreased from the lower temperature schedule (experiment EII 2), to the medium temperature (experiment EII 1, 3 + 4) to the higher temperature schedule (experiment EII 5, both control groups), the Kruskal-Wallis anova produced a p-value of 0.1408 indicating no statistical significant difference between the groups when using a 95% confidence level. The bootstrap analysis confirmed the Kruskal-Wallis results (see Figure 2) by producing multiple p-values with the Bonferroni test indicating no statistical significant difference between individual control groups (Table 9). Group II of experiment EII 5 had no checks and, therefore, a 95% grouping of the data was not possible. It is for that reason that the vertical bar does not overlap with the rest of the results.



**Figure 2** Bootstrap results of the *P. elliotii* control groups using a 95% confidence level to determine significant differences

**Table 9** Multiple p-values obtained from the Bonferroni statistical test that was applied to the bootstrap data of Figure 2

Experiment	EII 2	EII 1	EII 3	EII 4	EII 5 (I)	EII 5 (II)
<b>EII 2</b>		1.0000	1.0000	0.3016	0.2098	0.0648
<b>EII 1</b>	1.0000		1.0000	1.0000	1.0000	1.0000
<b>EII 3</b>	1.0000	1.0000		1.0000	1.0000	1.0000
<b>EII 4</b>	0.3016	1.0000	1.0000		1.0000	1.0000
<b>EII 5 (I)</b>	0.2098	1.0000	1.0000	1.0000		1.0000
<b>EII 5 (II)</b>	0.0648	1.0000	1.0000	1.0000	1.0000	

Although experiment EII 5 (II) statistically differed the most from experiment EII 2, no significant difference existed between the two groups when using a 95% confidence level. However, when using a 93% confidence level experiment EII 5 (II) statistically

differed from experiment Ell 2. Therefore, one can be 93% assured that the same results will be obtained when these experiments are repeated.

## 4.5 Discussion

The statistical evaluation of the *P. radiata* results indicated no significant difference between any of the four groups and, therefore, the treatment did not affect the number of checks.

The results of the first *P. elliotii* experiments showed no statistical, significant difference between the different experimental groups and, thereby, indicated no increase and no decrease in the number of checks in the boards treated with the SLS.

Very interesting differences between species were noticed. When comparing the control groups of the *P. radiata* experiment and the three *P. elliotii* experiments (Ell 1, 3 + 4) that were dried using the same schedule, there was a marked difference in the amount of surface checks. Table 6 shows that the *P. radiata* experiment had the most surface checks. Statistical evaluation showed that, in terms of the number of surface checks, *P. radiata* significantly differed from the other three *P. elliotii* control groups (see Figure 1). That was an indication of how species differ and will play a very important role when selecting timber intended for furniture production.

Within species reaction to different schedules was evaluated by comparing the control groups of the five *P. elliotii* experiments. Both water-soaked groups of experiment Ell 5 acted as control groups. The results indicated no statistically significant difference between any of the groups at a 95% confidence level and, therefore, no difference between the three different schedules used in these experiments. Although Group II (a water-soaked sample) of experiment Ell 5 statistically differed from the control (non-soaked and covered sample) group of experiment Ell 2 when a confidence level of 93% was used, it is not 100% certain whether the difference in sample preparation\handling had an effect on the results

and would need further investigation. This result shows the possible role high temperature drying will have in controlling drying defects like surface checks when SLS is used to control mould and blue stain.

One needs to consider the natural occurrence of surface checks in boards and how the original position in the log (sapwood vs. heartwood) as well as the presence of late wood at the surface, influence the development of surface checks. Even boards that came out of the same plank did not show any significant difference in terms of number of surface checks due to the treatment. Irrespective of species or kiln schedule, the SLS treatment, therefore, did not significantly reduce or increase the occurrence of surface checking in the two softwood species.

## 4.6 Conclusion

Although the boards from experiment Ell 5 (II) and experiment Ell 2 were statistically different at a 93% confidence level, one can't be 100% confident that the kiln schedule influenced the surface checking, because the two control groups (from experiment Ell 5 and Ell 2) were not treated the same. The effect of kiln schedule on surface checking, therefore, warrants further investigation.

Differences between species, however, did occur, as indicated by the significantly lower number of surface checks found in *P. elliotii* compared to *P. radiata*.

Treating green lumber with SLS prior to kiln drying did not affect the number of surface checks found in the lumber after drying. Consequently, SLS treatment of green lumber to control mould and blue stain would not result in a greater number of surface checks during drying.

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**Chapter 5: The influence of sodium lauryl sulphate (SLS) -  
treatment on *PVAc* and *UF* adhesive bond quality**

**To be submitted for publication in SA Forestry Journal**





# The influence of sodium lauryl sulphate (SLS) - treatment on *PVAc* and *UF* adhesive bond quality

## 5.1 Abstract

The influence of sodium lauryl sulphate (SLS) on the quality of the glue bond between two test samples of 50 mm x 50 mm x 18 mm was measured using two types of glues, namely polyvinyl acetate (PVAc) and urea formaldehyde (UF). The PVAc samples were glued under constant pressure of 700 kPa and shear broken in an Instron machine and the maximum failure force recorded. SLS only acted as a surface contaminant when applied to already dried wood in concentrations as high as 5%. The industrial use of SLS would most likely not reach that high concentration.

When the treatment was applied to wet wood prior to drying the results indicated a possible influence on the bond quality when concentrations as high as 2% and 5% SLS were used. However, surface analysis indicated a similar average percentage cohesive failure within the control group and treated groups. The average maximum breaking force recorded was, therefore, not an indication of the influence of SLS on the bond quality, but indicated the difference in wood properties of the test samples used for the experiments. Therefore, SLS did not act as a surface contaminant when test samples from wood treated with SLS prior to drying were glued using PVAc.

The UF samples were glued using a combination of 8 days closed assembly (a constant pressure of 700 kPa) and 2 days conditioning. This was determined at room temperature through a process of testing different combinations of closed assembly and conditioning. There was an increase in bond strength as the number of closed assembly days increased. The results obtained from the UF tests indicated that SLS did not influence the bond quality between the test samples and, therefore, did not act as a surface contaminant when applied to wet wood prior to drying when UF was used as an adhesive.

## 5.2 Introduction

The influence of naturally occurring substances in wood or that of “foreign” substances introduced with processing, on the quality of the glue bond, has been investigated widely. E.g. Hse and Kuo (1988) found that when extractives migrate to the surface of the wood during the drying process, they might cause gluing difficulties individually or as combined effects explained in one or more of the following ways:

1. Heavy deposits of extractives on the gluing surface block the reaction sites, thus preventing the chemical anchoring of adhesives;
2. Chemical incompatibility between the extractives and adhesives results in inferior glue bonds;
3. Extractives influence the wettability and polarity of the wood surface so that the wettability-permeability relationship of a particular adhesive is altered; and
4. Extractives affect the setting and curing characteristics of adhesives.

One group of extractives, namely fatty acids, has a chemical structure similar to SLS and in the paper by Hse and Kuo (1988), reference was made to work done by Sinclair *et al.* (1960) who suggested that the fatty acids were chemically anchored to the fibre surface by forming ether bonds with the cellulose.

Scharfetter (1971) studied the influence of chemicals, commonly found in the timber processing industry, on the gluability of wood using a resorcinol-based glue. Substances investigated were paraffin, diesel oil, motor oil, grease, spirits, pentachloro phenol (PCP), creosote, naphthenates, pine resin and wattle tannin. Of all these substances, only creosote, because of the presence of waxes, as well as wattle tannin, had a negative effect on the quality of the glue bond.

Two of the most common wood preservatives are copper-chromium-arsenate (CCA) and boron, which are both water-soluble inorganic wood-preserving salts. CCA is probably the most effective because it reacts chemically with the wood and is

considered unleachable. When wood is treated with CCA and used for glued products, the timber must be kiln-dried before gluing. In addition, the treated wood must be machined before using phenolic and resorcinol glues to remove possible surface irregularities of the timber. Melamine formaldehyde and crosslinked (two component) PVAc adhesives are successfully used for CCA-treated laminated wood products. The main advantage of the boron treatment is the fact that it does not interfere with the bonding quality because it does not alter the properties of the wood and in some cases, even enhanced bonding has been reported (SALMA Manual).

Fire-retardant treated wood, although not commonly used in South Africa, has received a lot of attention with regards to the effect the treatment has on the glue bond quality. In an article by Bergin (1963) three commercially important fire retardants were tested for their effect on the glue bond quality. They were zinc chloride-ammonium sulphate-boric acid (ZAB) and two types of ammonium phosphate-ammonium sulphate formulations (APS I and APS II). Eleven adhesives were used for the tests. Of the three fire retardant solutions, ZAB interfered the most with the glue bond quality. Of the glues tested only the room-temperature-curing urea and melamine adhesives produced reasonably good glue bonds under the different gluing conditions. In general, bonds produced with various combinations of treatments and adhesives, passed both adhesive and plywood specification requirements but showed significant reductions in strength when compared to results obtained from untreated veneers (Bergin, 1963).

In some building constructions, treated timber must be used, and due to reduced log sizes, the only way of producing larger sized timber products has been by means of glue laminating. It has, therefore, made the gluing of treated timber much more important. Although the gluing of treated timber has received some research attention the last twenty years, it has to be an area on which future research should concentrate because the forecast for glued treated timber is one of growth.

Sodium lauryl sulphate (SLS) treatment has been shown to control the development of blue stain and mould growth in open-stacked boards for up to three weeks (Perold, 2006). However, the effect of this treatment on the gluability of wood is not known. The aim of this paper was to investigate whether SLS could be considered a

“surface contaminant” when bonding SA pine with glues commonly used in the furniture industry, i.e. polyvinyl acetate (PVAc) or urea formaldehyde (UF) adhesive.

### 5.3 Materials and methods

Glue bond quality tests were conducted on kiln-dried, SLS sprayed SA pine as well as on wet of saw SA pine, which was SLS dip treated, kiln-dried and then planed.

#### 5.3.1 Kiln-dried SLS sprayed PVAc bonded SA pine

Rough sawn, kiln-dried 3.1 m x 114 mm x 38 mm *Pinus radiata* boards were planed to a thickness of about 18 mm and cut into blocks 50 mm by 50 mm (SABS Method 1044). Four groups (II to V) of ten replicate pairs were given the spray treatments indicated in Table 1 and was compared to a control group (Group I). The samples were placed flat on a metal grid and surfaces that would be glued were sprayed so that it ran off the surface.

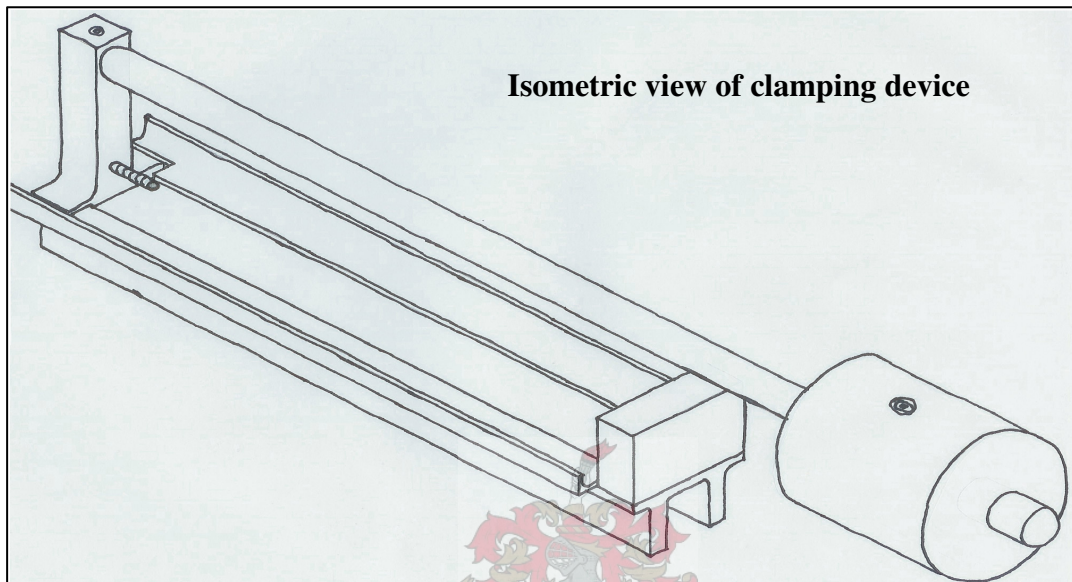
**Table 1** Surface treatments of sample groups (% expressed as mass SLS/mass tap water)

Group number	Spray treatment
I (Control)	None
II	Water only
III	0.05% SLS
IV	0.5% SLS
V	5% SLS

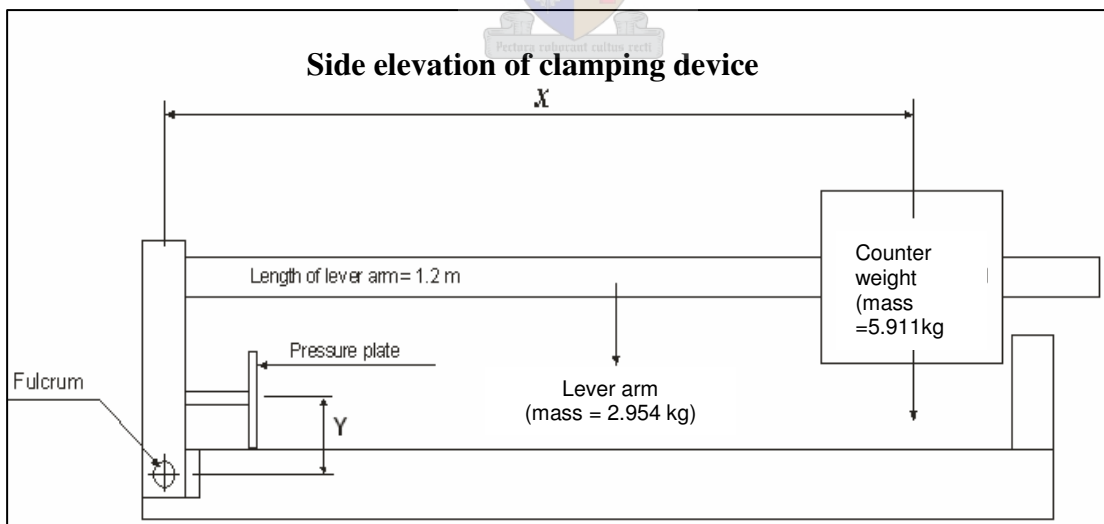
After ten minutes, the groups were placed in an oven at a temperature of less than 50 °C for about 24 hours to dry out the surface again. The samples were then conditioned at 20 ± 2 °C, 65% RH for more than 24 hours to attain equilibrium moisture content.

The samples were glued with a commercially available PVAc (Alcolin, Permo seal; refer to technical data sheet in Appendix E) to create a stepped joint of 40mm by 50 mm (SABS Method 1044) and a glueline pressure of 700 kPa (SABS Method 1029

and 1031) was applied using a custom made clamping device (See Figure 1). The samples were clamped for about 24 hours and then left at constant room temperature for about 24 hours to equalize. The clamping time was more than the curing time prescribed by the manufacturer to ensure that the glue had fully cured (See Appendix E).



**Figure 1** Custom made clamping device used to exert a constant pressure of 700kPa on the glued samples



Calculations to determine the position of the counterweight:

Variables (refer to illustration above):

$X$  = distance of counter weight to exert 700kPa pressure

$Y$  = 0.044 m

Area glued = 0.002 m<sup>2</sup>

Formula:

(Force x distance) + (force x  $\chi$ ) = (force x distance)

(2.954kg x 9.81m.s<sup>-1</sup> x 0.6m) + (5.911kg x 9.81m.s<sup>-1</sup> x  $\chi$ ) = (700000Pa x 0.002m<sup>2</sup> x 0.044m)

(17.39) + (57.99  $\chi$ ) = (61.6)

$\chi$  = (61.6-17.39) / (57.99)

$\chi$  = 0.762 m

### 5.3.2 Wet-of-saw boards, dip-treated with SLS and then kiln-dried

Wet, rough sawn 2.4 m x 114 mm x 38 mm *Pinus elliottii* timber was obtained from the Southern Cape. Pieces of about 10 cm were cut from one end of each board and the rest of the board was cut into 700 mm length sample boards. The boards were randomly distributed amongst three sets of four groups (I to IV), each group containing five replicates. Groups varied in SLS treatment. Details of the dip treatments appear in Table 2. The first set was dried using schedule 1 and the next two sets were dried using schedule 2. Group numbers and treatments in bold indicate the groups that were evaluated for possible SLS contamination. Only the bold groups were used because in practice the process would most likely entail dipping the boards in a SLS solution for extended periods of time and spraying the boards would not necessarily treat the boards to a surface depth sufficient to control mould and blue stain.

**Table 2** Dip treatments of sample groups for PVAc and UF (% expressed as mass SLS/mass tap water)

#### **Drying schedule 1**

<b><u>Group number</u></b>	<b><u>Dip treatment</u></b>
<b>I (Control)</b>	<b>None</b>
II	Water only
III	0.5% 10 sec SLS dip
<b>IV</b>	<b>0.5% 3 days SLS dip</b>

**Table 2 cont.  
Drying schedule 2**

<u>Group number</u>	<u>Dip treatment</u>
<b>I (Control)</b>	<b>None</b>
II	1% 10 sec SLS dip
III	0.5% 3 days SLS dip
<b>IV</b>	<b>1% 3 days SLS dip</b>

**Repetition of Drying schedule 2**

<u>Group number</u>	<u>Dip treatment</u>
<b>I (Control)</b>	<b>None</b>
II	5% 10 sec SLS dip
<b>III</b>	<b>2% 3 days SLS dip</b>
<b>IV</b>	<b>5% 3 days SLS dip</b>

**Details of drying schedule 1:**

<b>Drying phase</b>	<b>Dry Bulb (°C)</b>	<b>Wet Bulb (°C)</b>	<b>RH (%)</b>	<b>EMC (%)</b>
<b>Initial MC – 10%</b>	60	50	58	8.9
<b>Initial MC – 20%</b>	62	50	53	7.9
<b>Initial MC – 30%</b>	65	50	44	6.8
<b>Initial MC – 40%</b>	68	50	39	5.8
<b>Initial MC – 50%</b>	72	50	32	4.8
<b>To 11 %</b>	75	45	19.5	3.0
<b>Equalize 300 min</b>	75	68	72.5	10.6

**Details of drying schedule 2:**

<b>Drying phase</b>	<b>Dry Bulb (°C)</b>	<b>Wet Bulb (°C)</b>	<b>RH (%)</b>	<b>EMC (%)</b>
<b>Warm up 240 min</b>	90	87	89	14.3
<b>Green to 12%</b>	90	60	25.4	3.1
<b>Equalize 300 min</b>	90	87	89	14.3

After the wood was dried, the boards were flatsawn in half, 2 mm was planed from the tangential side that was treated (the outside) and the inside planed smoothly to finish with a final thickness of about 18 mm. From five of the boards in each group, ten samples, having the same dimensions as the previous tests, were cut so that each board had 5 and each group had 20 PVAc glued pairs that could be tested for strength. The PVAc gluing procedure was repeated as in section 2.1. The main

treated group within each set (the 0,5%, 1%, 2% and 5%) was compared to the control group of that specific set, because the three experiments were dried using two different drying schedules. In the last experiment both the 2% and 5% treatment had the same control group.

Glue bond tests were also performed using UF provided by a local sawmill. The same UF to hardener ratio (the hardener used was ammonium chloride) as used by the mill was used for these experiments. The UF glue was mixed in the laboratory and applied to the test samples. The samples were prepared and glued duplicating the procedures in section 2.1. The optimal laboratory curing time of the UF received from the mill was not known as curing at the mill was accelerated by heating the laminated beams. It was, therefore, necessary to determine the optimal curing time for the laboratory conditions to ensure reliable and representative results. Different combinations of pressure (closed assembly for curing) and no pressure (conditioning) had to be found for the laboratory conditions that would be used for these experiments. Combinations up to twelve days were tested.

### 5.3.3 Strength tests

The samples were evaluated in an Instron testing machine using a constant loading speed of 0.5 cm/min and the maximum breaking force recorded. It was decided to use a shear test as the method of evaluating the samples. A tension test, was not considered, because the area glued in the tension test (6,25 cm<sup>2</sup>) was too small to really get a result representative of what happens inside a laminated beam or joint. Normally, when given the choice, a designer will seldom use the direct tensile loading mode, because in practice the contact area is markedly increased by overlapping, scarfing, and so on (Pizzi and Mittal, 1994). Of these, the finger joint is the most commonly known method of increasing the contact area.

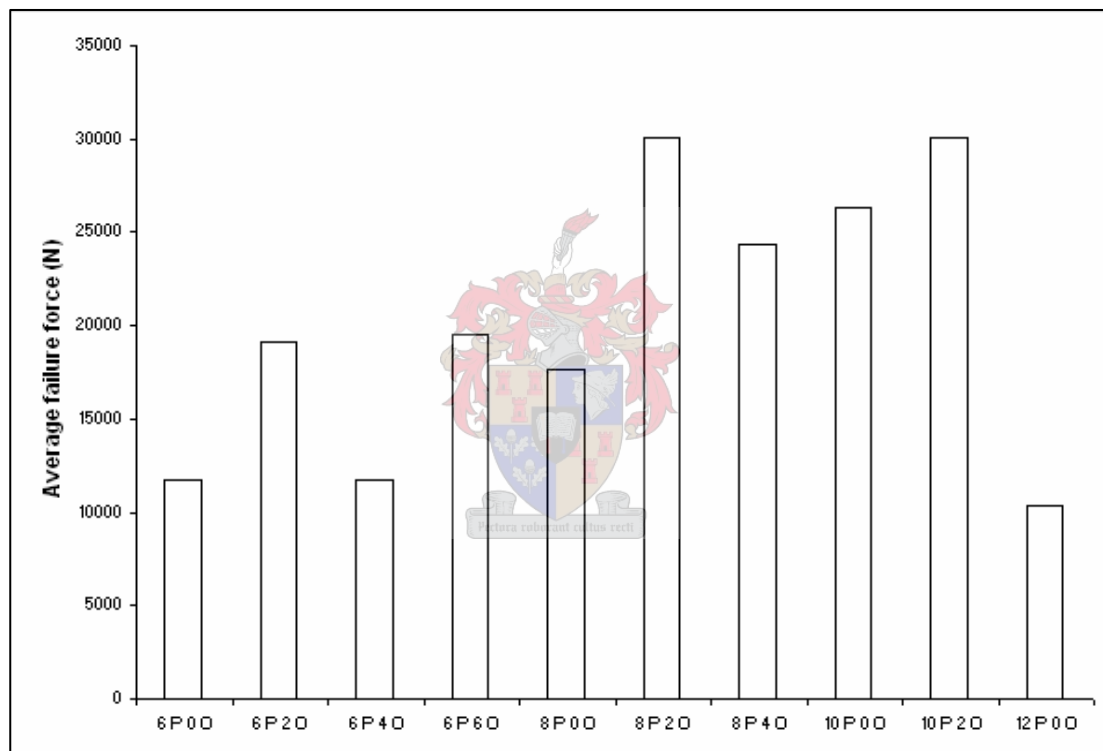
The number, preparation, selection and testing of the samples complied with the SABS methods 1029, 1031, 1033 and 1044. To determine the percentage wood (cohesive) and glue/wood (adhesive) failure a grid, 50 mm x 40 mm (the area glued in the shear test samples) containing 63 points, was used (SABS 987).



## 5.4 Results and Discussion

### 5.4.1 Determination of optimal UF curing time

Under a constant pressure of 700 kPa there was a marked increase in bond strength from 6 to 10 days closed assembly time (Figure 2). The very low strength value obtained after 12 days closed assembly time was due to inferior wood properties (grain direction) present in the samples. The highest strength value was obtained with the 8 days closed assembly (for curing) and 2 days conditioning combination.



**Figure 2** Effect of duration pressure and conditioning on UF bond strength

Legend: First number = number of days under pressure (P) (closed assembly);

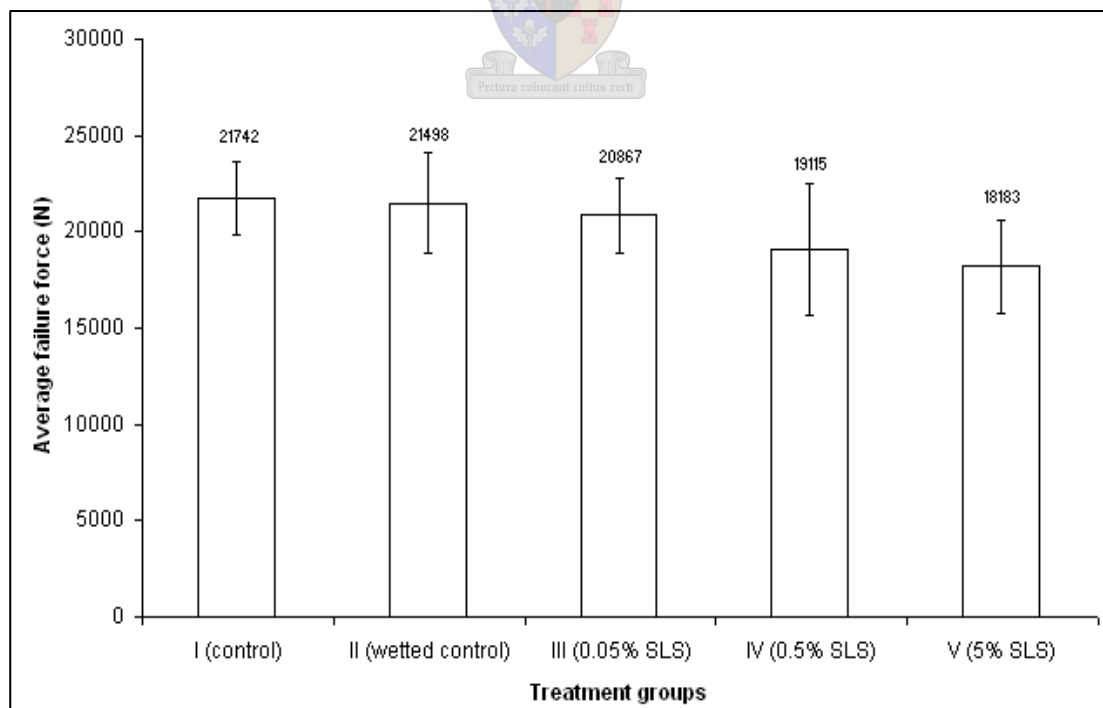
second number = number of days for conditioning (O)

From a structural point of view, the observed increase in UF bond strength, at room temperature, as the number of closed assembly days for the UF increased, is very important in the manufacture and use of UF laminated beams. Under production conditions of glue laminated beams, the minimum time the UF needs to cure at room temperature ( $20 \pm 2^{\circ}\text{C}$ ) to attain adequate bond strength should not be less than 10 days (preferably not less than 8 days closed assembly) before the glued product can

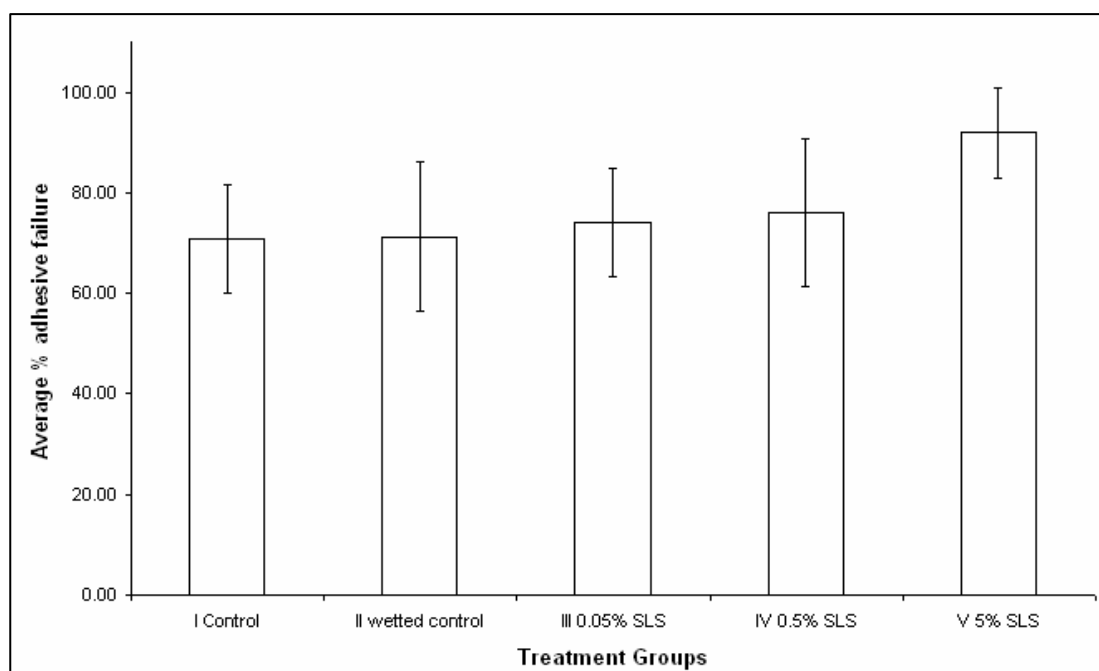
be put under load. The time before the glued product can be loaded all depends on the manufacturing process used at the different plants. For instance, if heating was used to accelerate the curing process during closed assembly, it would take less time to attain adequate bond strength. Due to inadequate facilities and equipment, the effect of temperature on accelerated curing of UF was not tested.

#### 5.4.2 Shear test on kiln-dried, PVAc bonded samples sprayed with different SLS concentrations

From Figure 3 it can be seen that the (failure) force required to break the glued samples decreased as the SLS concentration increased. The shear tests also showed that as the concentration of SLS increased from group III to V, the percentage failure pointed more towards an adhesive type and less towards a cohesive type of failure (Figure 4). These two observations support the view that at these concentration levels SLS solutions induced a negative effect on bond quality, being manifested as a reduction in adhesive strength.



**Figure 3** Effect of different treatments on kiln-dried wood on the average failure force and standard deviation of PVAc bonded wood



**Figure 4** Average % adhesive (wood/glue) failure per group after different treatments

A Bonferroni test was performed on the data to determine whether there were any significant statistical differences between the two control groups and the three treated groups. Working with a 95% confidence level (i.e. a proportion of 0.05), the p-value of 0.0479 for group V (the 5% SLS solution) and the untreated group I (See Table 3) indicated a significant difference. The p-values highlighted in bold are less than 0.05 indicating a 95% certainty that group V significantly differs from group I possibly due to the treatment.

**Table 3** Statistical evaluation of the PVAc bond strength indicating the confidence level, p, as computed with the Bonferroni test. When  $p < 0.05$  (values in bold), it indicates with more than 95% certainty, that the samples significantly differ due to treatment effects.

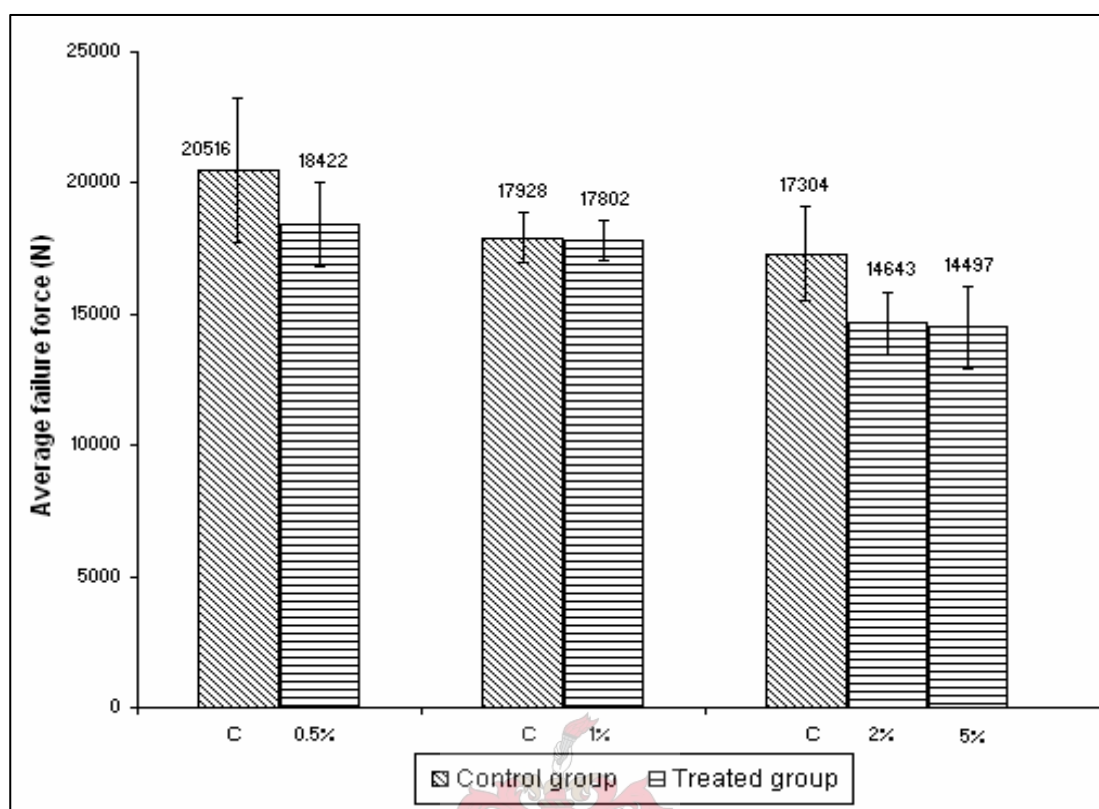
	I	II	III	IV	V
I		1.000	1.000	0.294	<b>0.048</b>
II	1.000		1.000	0.416	0.068
III	1.000	1.000		1.000	0.262
IV	0.294	0.416	1.000		1.000
V	<b>0.048</b>	0.068	0.262	1.000	

Therefore, these statistical results indicated that only a SLS solution as high as 5% did in fact negatively influence the PVAc glue bond when the SLS was sprayed onto

a dry wooden surface. As mentioned in section 1, the higher the SLS concentration the more available bindings sites are occupied by the SLS molecules (Van Der Waals type bonds) causing a reduction in the bond quality when gluing. Effective control of mould and bluestain was achieved by dipping green wood in SLS concentrations of less than 5% (Perold *et al.*, 2006). It is for that reason that the worst case scenario was used to evaluate the SLS treatments and only dip treatments of extended periods using high SLS concentrations were evaluated in this study when green wood were dip treated, dried and glued.

#### 5.4.3 PVAc shear tests of wet boards treated with SLS prior to drying

Figure 5 shows the average maximum failure force recorded for the PVAc samples as well as the standard deviation of each treatment. The treated groups were compared with the control group in each drying run to make provision for the difference in the drying conditions between schedule 1 and 2 and the possible slight differences between the repetition of schedule 2. A qualitative assessment of the results depicted in Figure 5 obtained for the control groups indicated that drying schedule 1 and 2 had different effects and that the repetition of schedule 2 almost had identical effects on the results. As far as the SLS treatment is concerned, the height of the bars in Figure 5 suggests that only the 2% and 5% treatments appear to have influenced the bond strength.



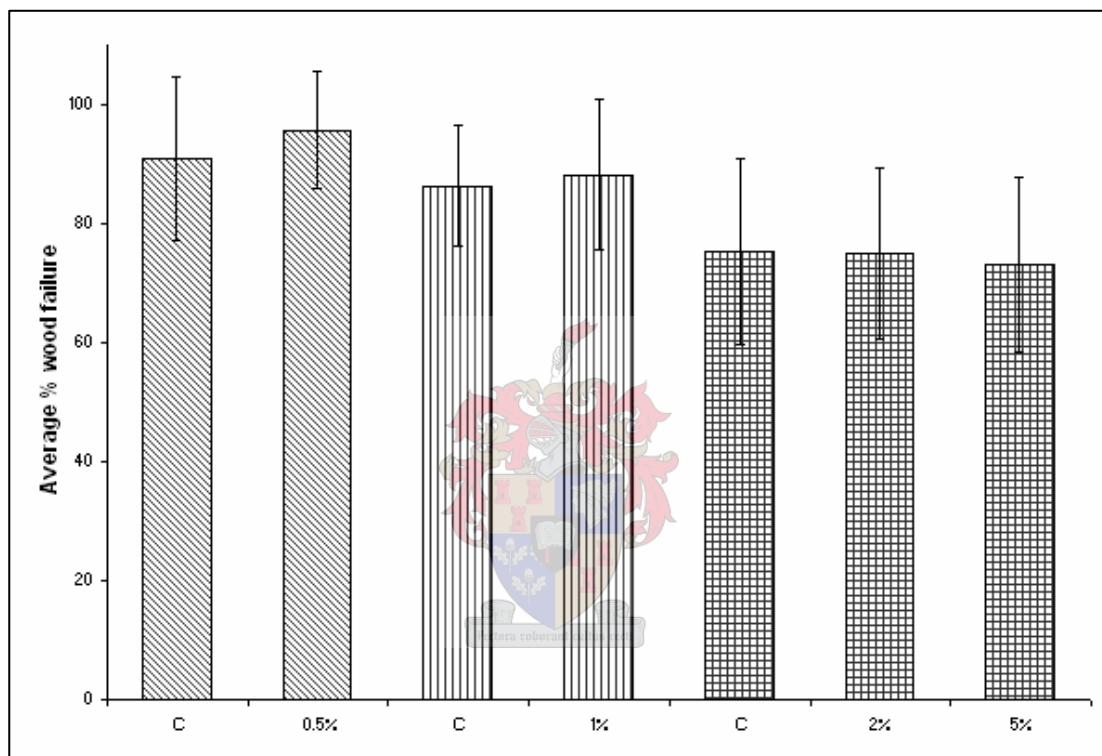
**Figure 5** Effect of different treatments of wet wood before kiln-drying on average failure force and standard deviation of PVAc bonded wood

As shown in Table 4, a Bonferroni test of significance on the data confirmed that the 2% and 5% treatments (indicated by the bold values in the treatment vs the control group column) had significantly negatively influenced the strength of the glue bond.

**Table 4** The group p-values for the statistical evaluation of the PVAc tests using a 95% confidence level and the p-values for the treated group (T) vs the control group (C) for each experiment (Values in **bold** indicate statistical significant difference)

Treatment	PVAc	
	Group value	T vs C
0.5%	0.0975	0.1647
1%	<b>0.0000</b>	1.0000
2%	<b>0.0000</b>	<b>0.0080</b>
5%	<b>0.0000</b>	<b>0.0043</b>

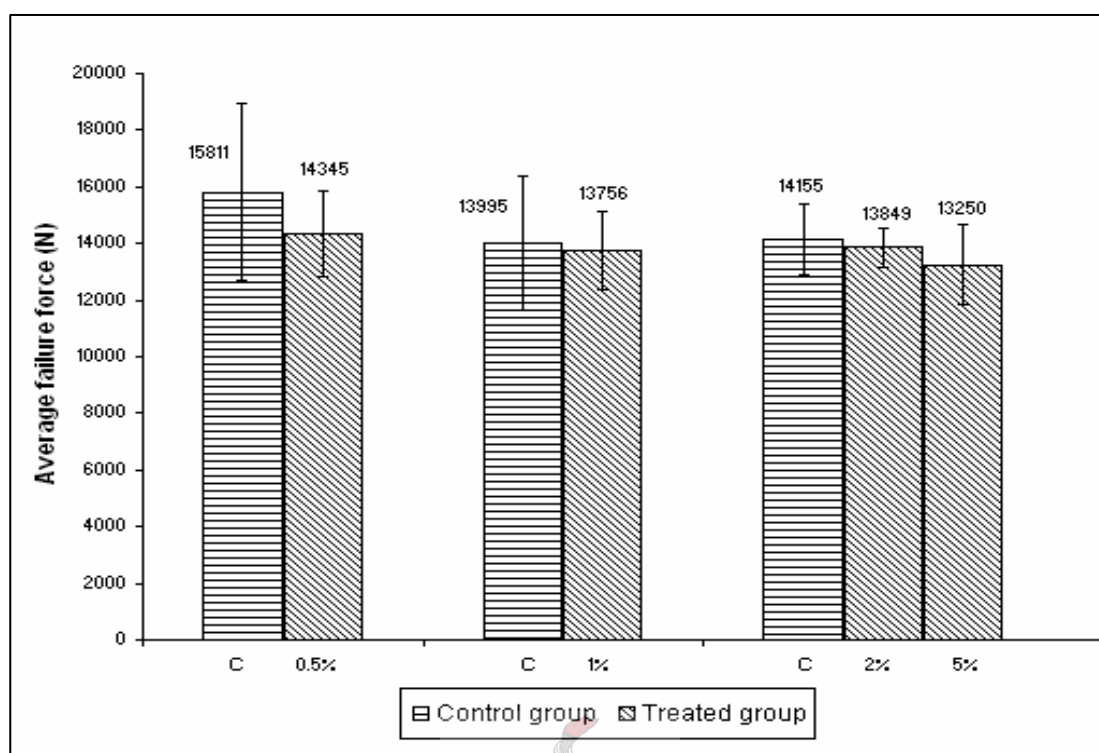
However, after surface evaluation in terms of the percentage of cohesive (wood) and adhesive (wood/glue) failure, it is clear in Figure 6 that the control group and the treated groups, 2% and 5% of the repetition of schedule 2, generated a similar percentage cohesive failure. Therefore, the statistical results in Table 4 indicated that the difference between the control group and the treated groups was not due to the SLS treatment, but is an indication of the difference in wood properties between the control group and the treated groups.



**Figure 6** The average % wood failure and standard deviation for each group in the PVAc shear tests

#### 5.4.4 UF shear tests of wet boards treated with SLS prior to drying

Figure 7 shows the average maximum breaking force results obtained for UF bonded wood samples when the procedures of the PVAc experiments were duplicated. It is clear from Figure 7 that no significant difference existed between the 0.5%, 1%, 2% and 5% groups treated with SLS.



**Figure 7** Average maximum breaking force and standard deviation for the UF shear tests

Statistical evaluation indicated no significant difference between the different control groups and their corresponding treated groups (see Table 5).

**Table 5** The group p-values for the statistical evaluation of the UF tests using a 95% confidence level and the p-values for the treated group (T) vs the control group (C) for each experiment

Treatment	UF	
	Group value	T vs C
0.5%	0.6367	1.0000
1%	0.9039	1.0000
2%	0.9039	1.0000
5%	0.9039	1.0000

Results from the surface evaluation in terms of the percentage adhesive or cohesive failure indicated that cohesive (wood) failure was the main type of failure and that the SLS treatment did not influence the glue bond (see Figure 8).

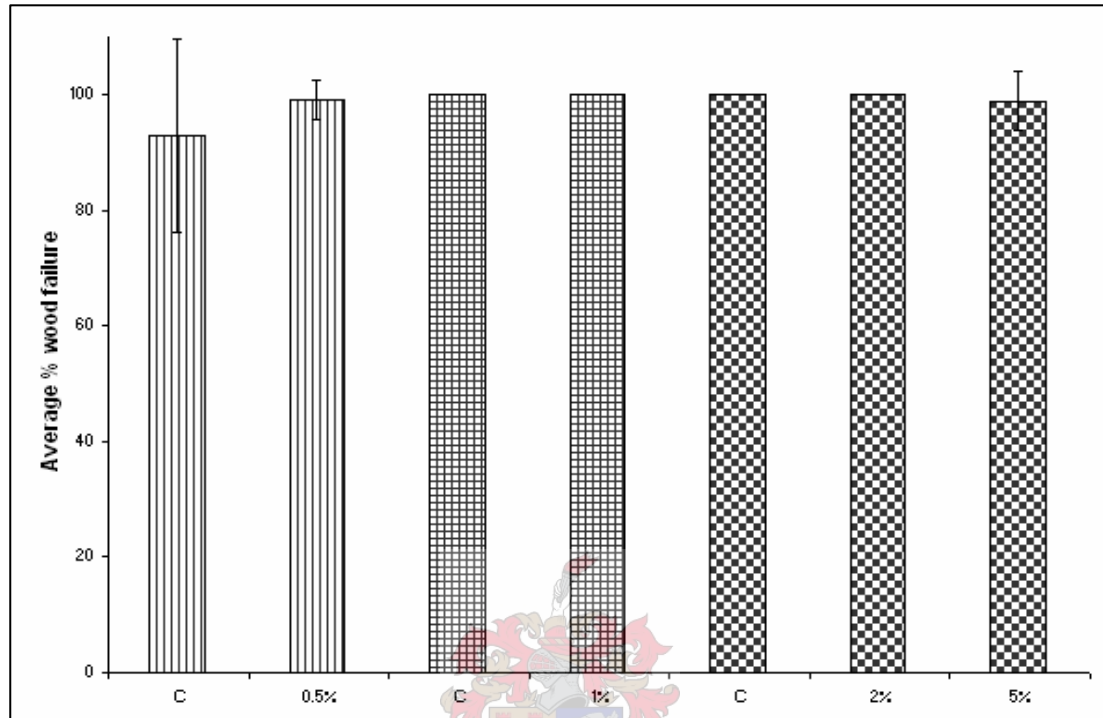


Figure 8 The average % wood failure for each group in the UF shear tests

## 5.5 Conclusions

### SLS applied on dry, PVAc bonded softwood

When SLS was applied on dry, PVAc bonded softwood it only acted as a surface contaminant when the concentration was as high as 5%, which is outside the range of application that was effective for controlling mould on open-stacked boards.

### Wet-off-saw softwood dip-treated with SLS prior to kiln drying and PVAc bonded

The treatment of green lumber with 2% and 5% SLS concentrations had no appreciable influence on the bond quality of PVAc bonded, kiln dried wood.



On surface analysis it was found that a similar average percentage cohesive failure within the control group and treated groups existed and, therefore, the average maximum breaking force recorded was an indication of the difference in wood properties of the test samples used for the experiments. Therefore, SLS did not contaminate the surface when test samples from wood treated with SLS prior to drying were glued using PVAc.

*Wet-off-saw softwood dip-treated with SLS prior to kiln drying and UF bonded*

SLS treatment of green softwood did not influence the UF bond quality of the kiln dried test samples and, therefore, did not contaminate the surface when test samples from wood treated with SLS prior to drying were glued using UF.

Treating green lumber before drying to control mould and bluestain will not influence the gluing properties of softwood when gluing with PVAc or UF.



## 5.6 References

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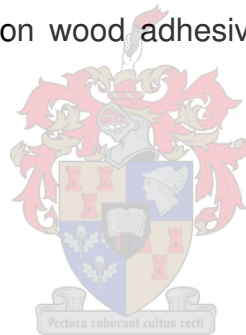
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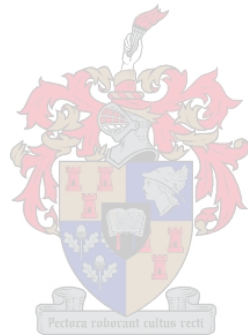
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## Chapter 6: General discussion and conclusions



## General discussion and conclusions

An SLS concentration as low as 0.1% effectively controlled surface mould and blue stain development for 3 weeks on open-stacked boards. When close-staked, a 0.2% SLS solution effectively controlled blue stain development over a two week period.

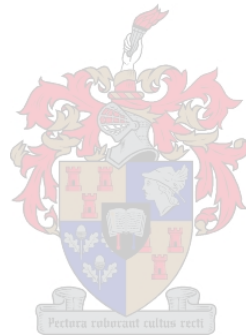
Treating green lumber with SLS prior to kiln drying did not affect the number of surface checks found in the lumber after drying. Consequently, SLS treatment of green lumber to control mould and blue stain would not result in a greater number of surface checks during drying.

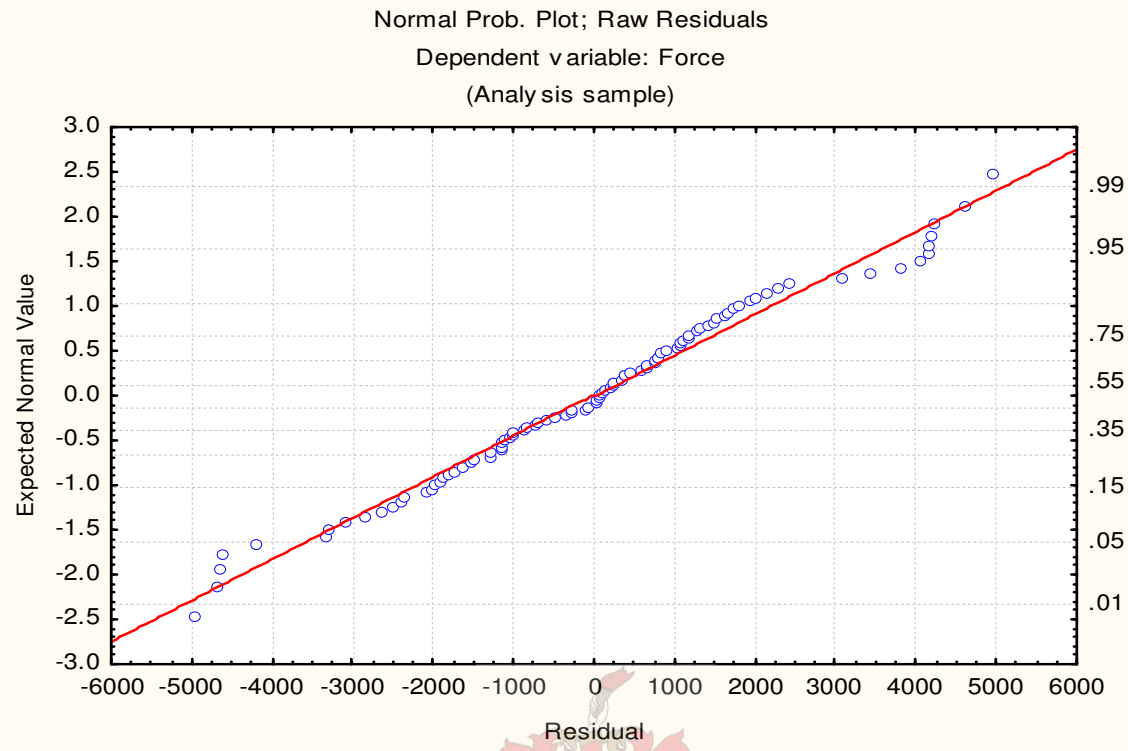
When SLS was applied on air-dry, PVAc bonded softwood it only acted as a surface contaminant when the concentration was as high as 5%, which is outside the range of application that was effective for controlling mould on open-stacked boards. SLS treatment of green softwood did not influence the UF bond quality between the kiln dried test samples and, therefore, did not contaminate the surface when test samples from wood treated with SLS prior to drying were glued using UF. Consequently, treating green lumber before drying to control mould and bluestain will not influence the gluing properties of softwood when gluing with PVAc or UF.

Thus, the SLS results on the control of mould and bluestain development, its effect on surface checking and adhesive bonding quality, indicated that SLS may be a viable, non-toxic alternative for the short-term control of mould and blue stain that would not adversely affect the surface quality of the lumber. Possible application areas could include log and green lumber storage, and air-drying.

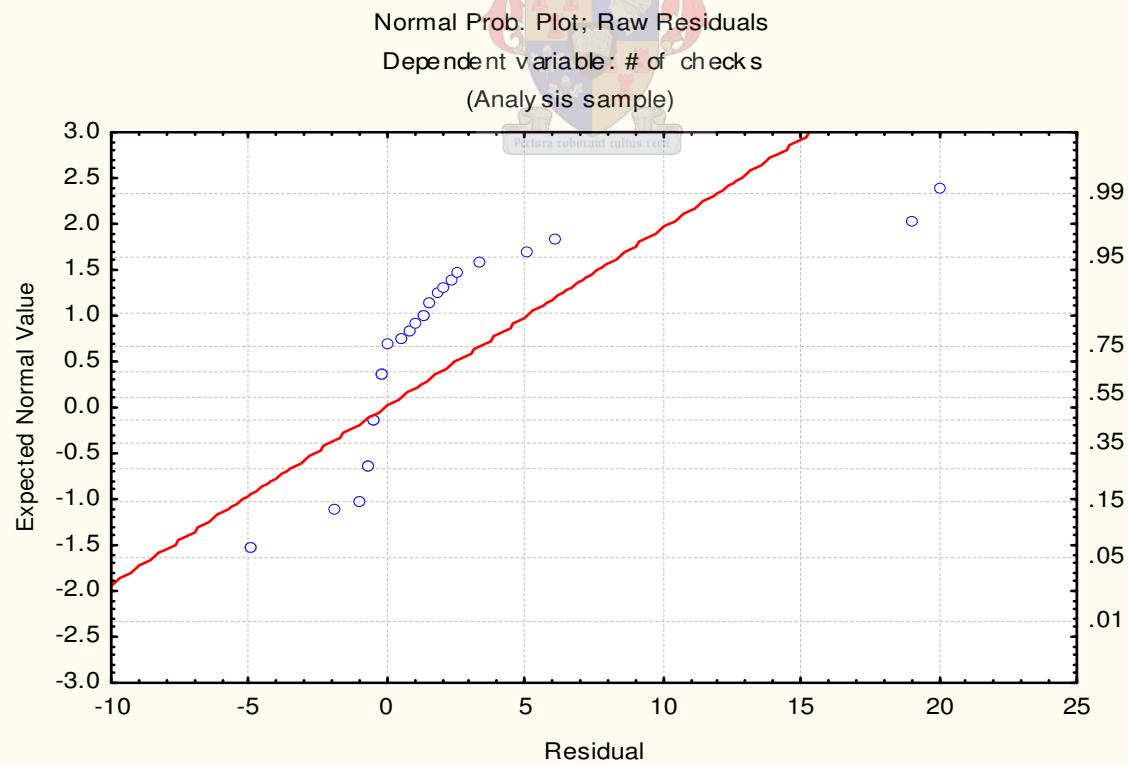
The results also showed that there was not any statistically significant difference in the number of surface checks produced, at a 95% confidence level, between three different kiln schedules. In addition, the results showed that a significantly lower number of surface checks occurred in *P. elliotii* compared to *P. radiata* when dried with the same kiln schedule.

## **Appendix A: Difference between normally and non-normally distributed data**





**Figure I** Example of normally distributed data



**Figure II** Example of data not distributed normally

## Appendix B: Description of bootstrap procedure



### Description of bootstrap procedure:

The data from all the experiments was not normally distributed and, therefore, to determine whether any statistical difference existed between the experimental groups the Kruskal-Wallis anova was the most suitable analysis. Further analysis to determine which groups differed from each other was necessary and a bootstrap analysis was used to produce multiple p-values.

The bootstrap analysis is a resampling technique applied to an existing set of data when the existing set of data is non-uniformly distributed (Appendix A). The bootstrap procedure repeatedly draws random samples from the original data set with replacement. With reasonably large sample sizes, the number of times the data set can be sampled is effectively infinite and usually a thousand or more analyses are performed to arrive at stable average values for the parameter estimates and their standard errors.





**Appendix C: Number of checks per length group for each  
of the surface checks experiments**



**Number of checks per length group for each of the surface checks experiments:**

**Table 1** Number of surface checks for each length group for the *P. radiata* experiment

Group #	I (control)	II (0.05% SLS)	III (0.5% SLS)	IV (5% SLS)
0-19mm	15	25	7	8
20-39mm	36	74	42	76
40-59mm	23	29	23	50
60-79mm	10	4	11	20
80-99mm	7	6	6	6
100mm+	8	5	6	7
<b>Total</b>	<b>99</b>	<b>143</b>	<b>95</b>	<b>167</b>

**Table 2** Number of surface checks for each length group for the first *P. elliotii* (Ell 1) experiment

Group #	I (0.5% 10 sec dip)	II (control)	III (0.5% 3 days)	IV (water 3 days)
0-19mm	0	5	4	0
20-39mm	7	4	14	0
40-59mm	4	1	2	0
60-79mm	0	2	1	1
80-99mm	0	0	6	0
100mm+	0	1	1	0
<b>Total</b>	<b>11</b>	<b>12</b>	<b>28</b>	<b>1</b>

**Table 3** Number of surface checks for each length group for the second *P. elliotii* (Ell 2) experiment

Group #	I (control)	II (water 3 days)	III (0.5% 10 sec dip)	IV (0.5% 3 days)
0-19mm	5	13	17	8
20-39mm	8	12	19	21
40-59mm	5	9	11	8
60-79mm	2	4	4	4
80-99mm	1	1	4	4
100mm+	0	3	3	0
<b>Total</b>	<b>21</b>	<b>42</b>	<b>58</b>	<b>45</b>

**Table 4** Number of surface checks for each length group for the third *P. elliotii* (Ell 3) experiment

Group #	I (control)	II (1% 10 sec dip)	III (0.5% 3 days)	IV (1% 3 days)
0-19mm	1	1	1	2
20-39mm	7	2	1	2
40-59mm	1	0	1	0
60-79mm	0	1	0	0
80-99mm	0	0	0	0
100mm+	0	0	0	0
<b>Total</b>	<b>9</b>	<b>4</b>	<b>3</b>	<b>4</b>

**Table 5** Number of surface checks for each length group for the fourth *P. elliotii* (Ell 4) experiment

Group #	I (control)	II (5% 10 sec dip)	III (2% 3 days)	IV (5% 3 days)
0-19mm	0	1	4	4
20-39mm	2	0	8	8
40-59mm	1	3	4	2
60-79mm	1	2	2	1
80-99mm	0	2	1	0
100mm+	0	0	1	0
<b>Total</b>	<b>4</b>	<b>8</b>	<b>20</b>	<b>15</b>

**Table 6** Number of surface checks for each length group for the fifth *P. elliotii* (Ell 5) experiment

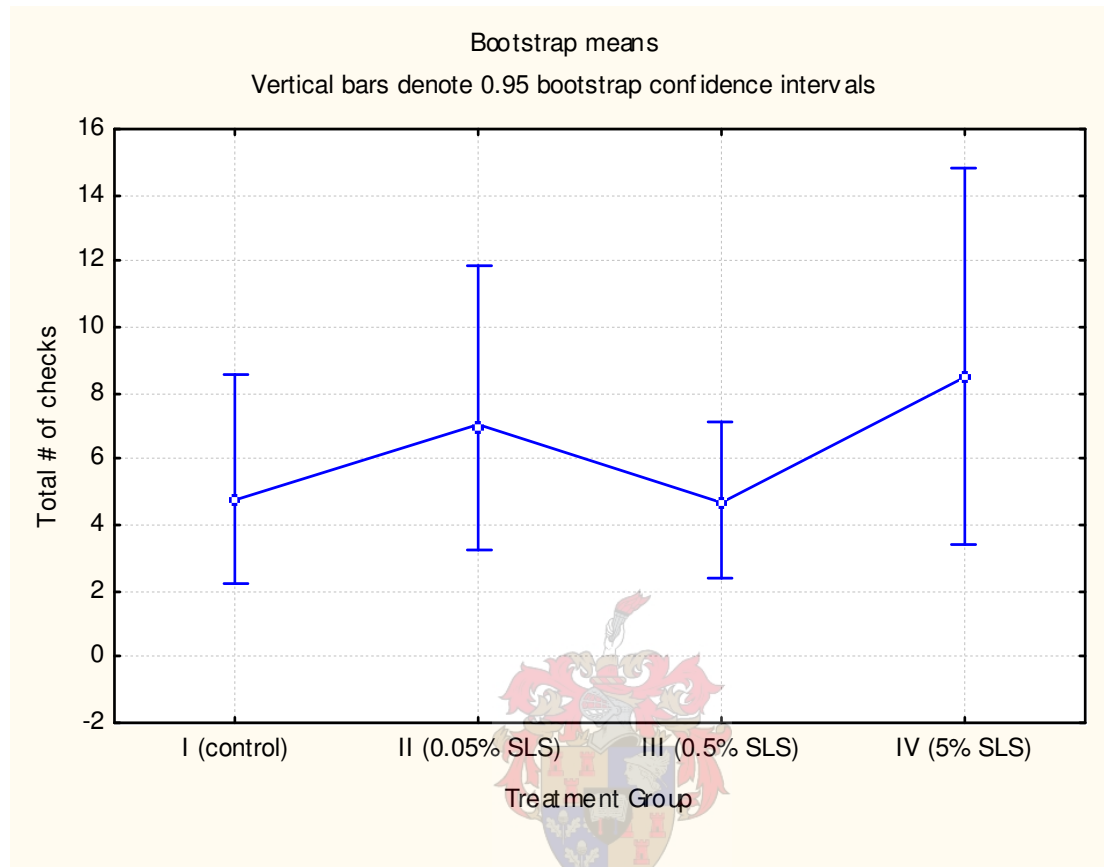
Group #	I (water control)	II (water control)	III (2% 3 days)	IV (5% 3 days)
0-19mm	1	0	3	5
20-39mm	1	0	3	3
40-59mm	1	0	0	1
60-79mm	0	0	0	2
80-99mm	0	0	0	0
100mm+	0	0	1	0
<b>Total</b>	<b>3</b>	<b>0</b>	<b>7</b>	<b>11</b>

## **Appendix D: Bootstrap and Bonferroni results of the surface checks experiments**



## Bootstrap and Bonferroni results of the surface checks experiments

### *The P. radiata experiment*

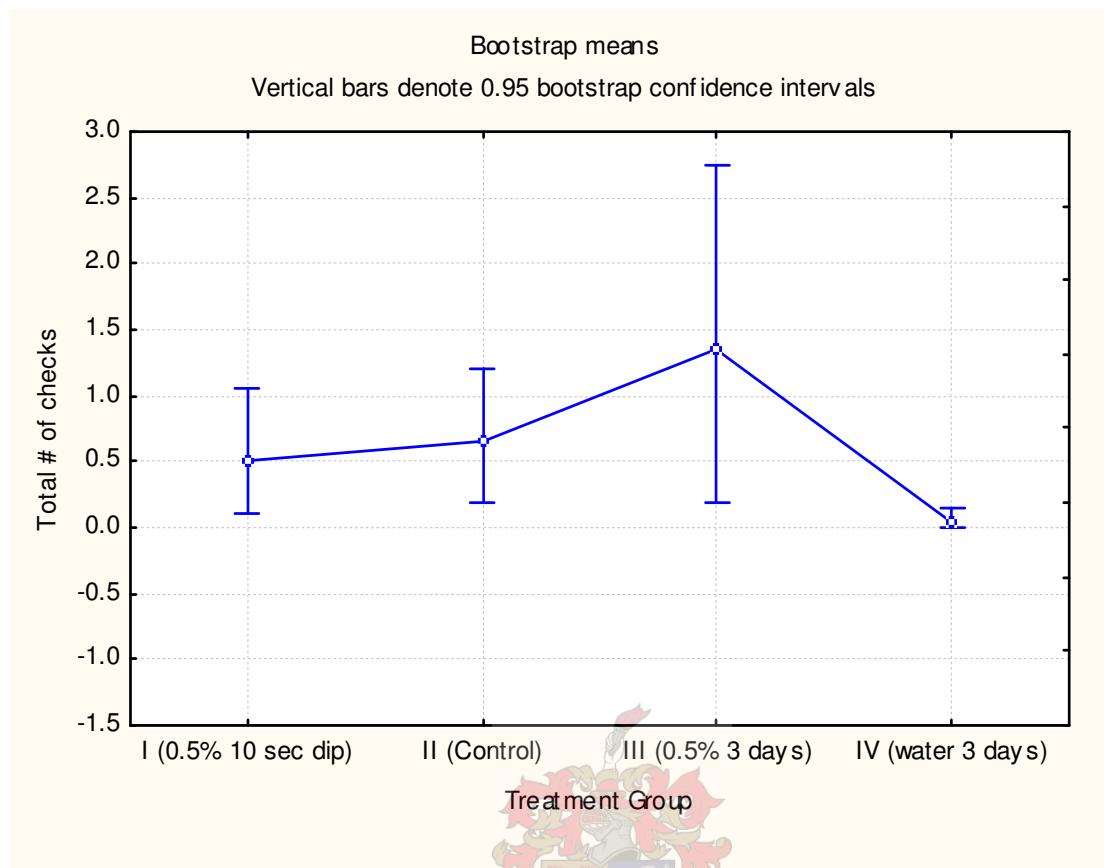


**Figure 1** Bootstrap results of the *P. radiata* experiment using a 95% confidence level to determine significant differences

### Bonferroni results of the *P. radiata* experiment

Group #	Group 1	Group 2	Group 3	Group 4
Group 1		1.0000	1.0000	1.0000
Group 2	1.0000		1.0000	1.0000
Group 3	1.0000	1.0000		1.0000
Group 4	1.0000	1.0000	1.0000	

*P. elliotii* experiment 1(Ell 1)

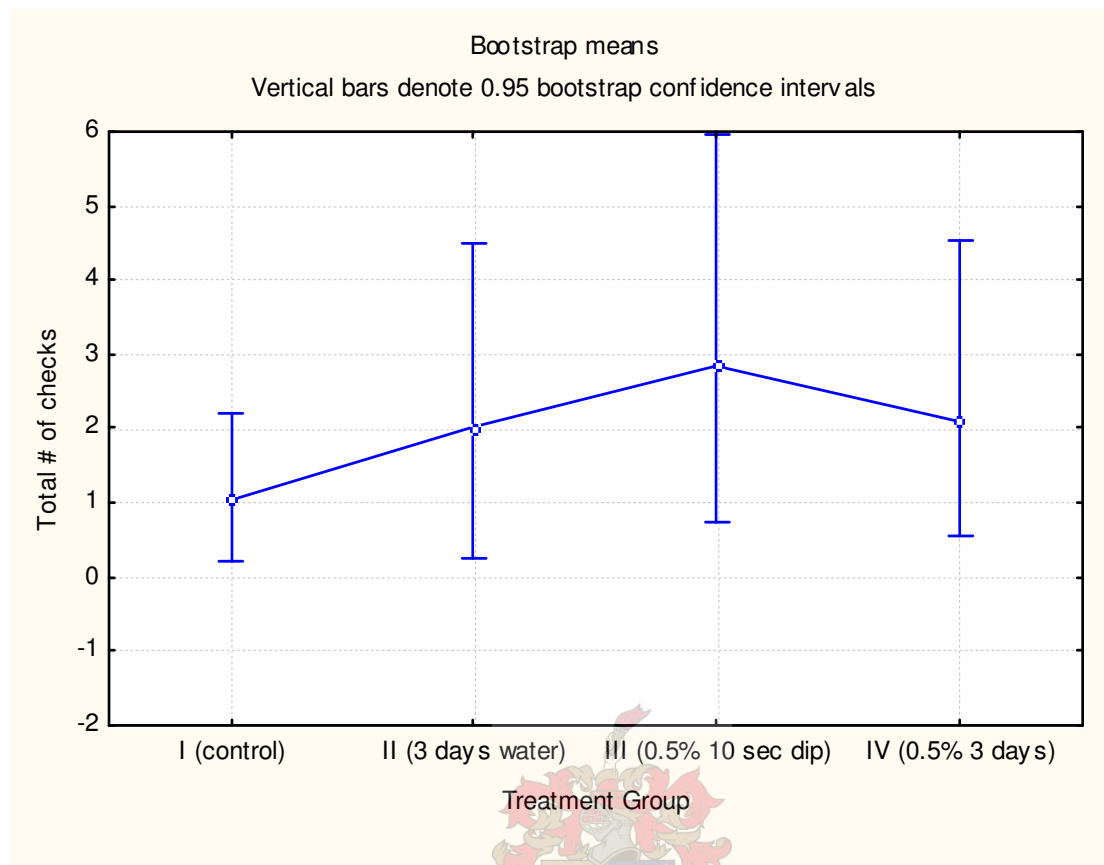


**Figure 2** Bootstrap results of the first *P. elliotii* (Ell 1) experiment using a 95% confidence level to determine significant differences

Bonferroni results of the *P. elliotii* experiment 1 (Ell 1)

Group #	Group 1	Group 2	Group 3	Group 4
Group 1		1.0000	0.8132	1.0000
Group 2	1.0000		1.0000	1.0000
Group 3	0.8132	1.0000		0.1142
Group 4	1.0000	1.0000	0.1142	

*P. elliotii* experiment 2 (EII 2)

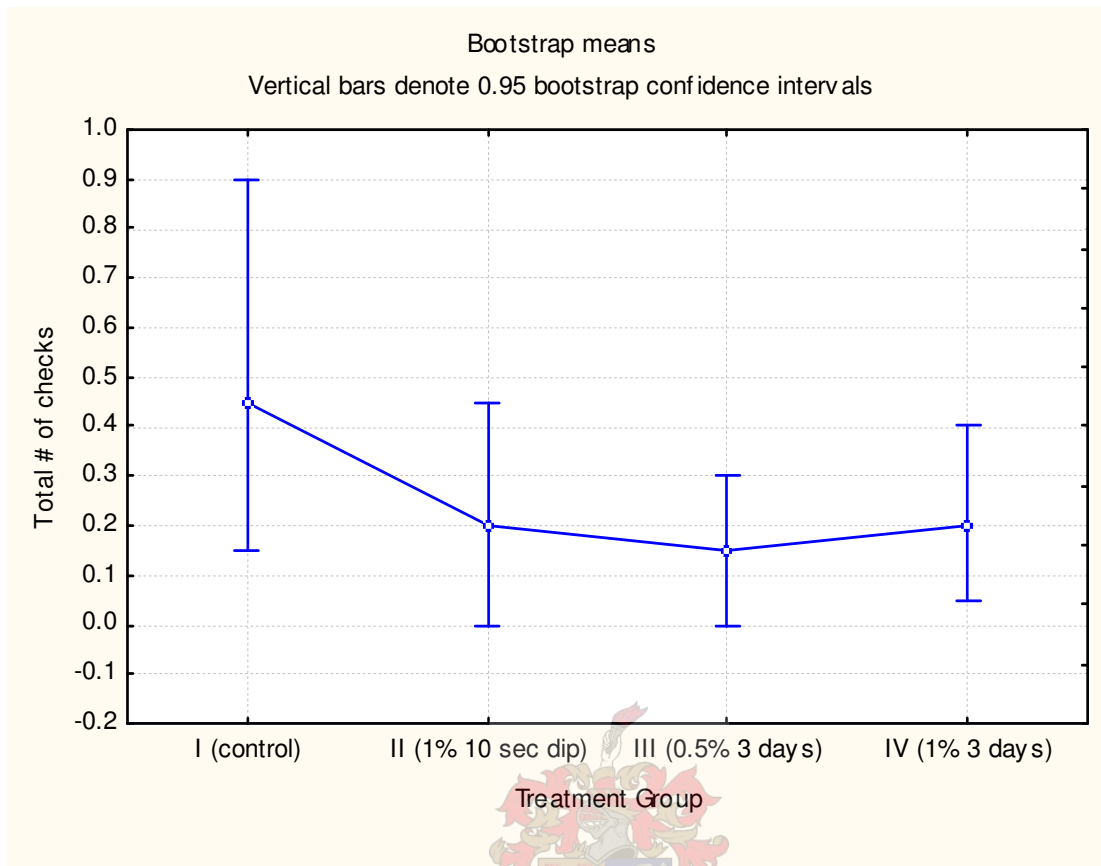


**Figure 3** Bootstrap results of the second *P. elliotii* (EII 2) experiment using a 95% confidence level to determine significant differences

Bonferroni results of the *P. elliotii* experiment 2 (EII 2)

Group #	Group 1	Group 2	Group 3	Group 4
Group 1		1.0000	1.0000	1.0000
Group 2	1.0000		1.0000	1.0000
Group 3	1.0000	1.0000		1.0000
Group 4	1.0000	1.0000	1.0000	

*P. elliotii* experiment 3 (EII 3)



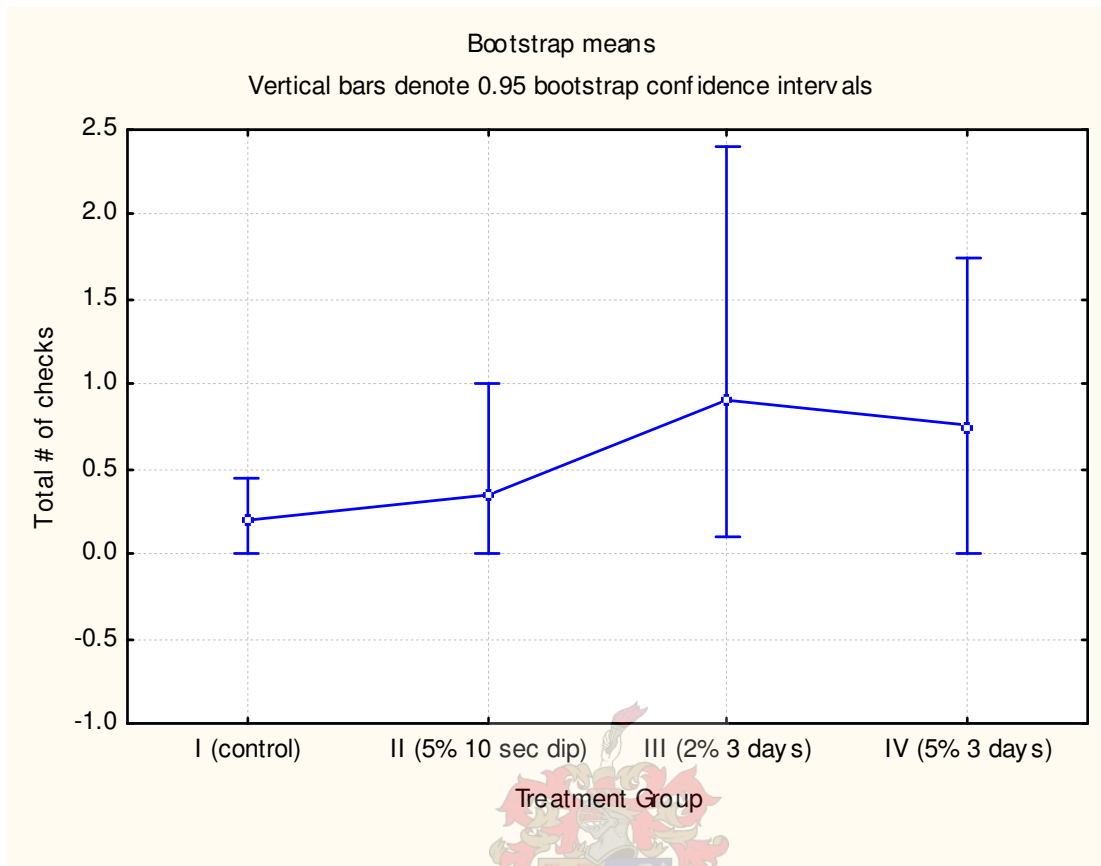
**Figure 4** Bootstrap results of the third *P. elliotii* (EII 3) experiment using a 95% confidence level to determine significant differences

Bonferroni results of the *P. elliotii* experiment 3 (EII 3)

Group #	Group 1	Group 2	Group 3	Group 4
Group 1		1.0000	0.6497	1.0000
Group 2	1.0000		1.0000	1.0000
Group 3	0.6497	1.0000		1.0000
Group 4	1.0000	1.0000	1.0000	



*P. elliotii* experiment 4 (EII 4)

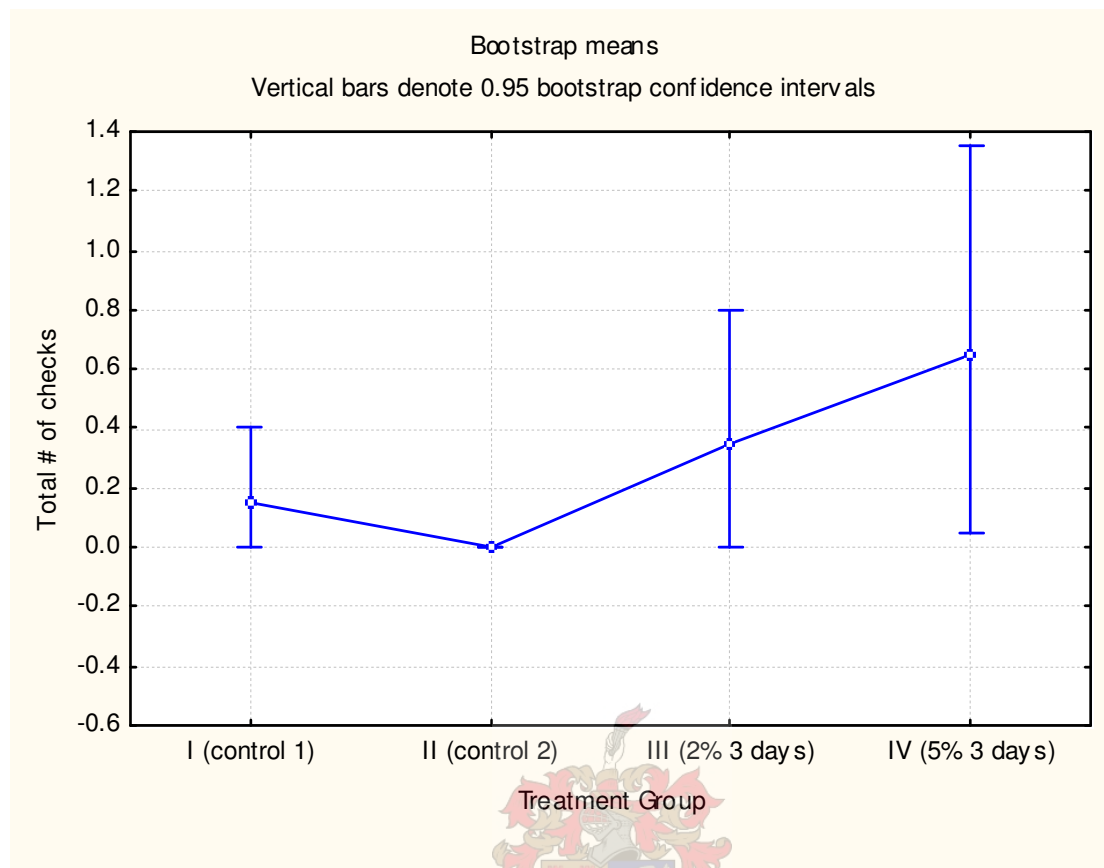


**Figure 5** Bootstrap results of the fourth *P. elliotii* (EII 4) experiment using a 95% confidence level to determine significant differences

Bonferroni results of the *P. elliotii* experiment 4 (EII 4)

Group #	Group 1	Group 2	Group 3	Group 4
Group 1		1.0000	1.0000	1.0000
Group 2	1.0000		1.0000	1.0000
Group 3	1.0000	1.0000		1.0000
Group 4	1.0000	1.0000	1.0000	

*P. elliotii* experiment 5(Ell 5)



**Figure 6** Bootstrap results of the fifth *P. elliotii* (Ell 5) experiment using a 95% confidence level to determine significant differences

Bonferroni results of the *P. elliotii* experiment 5 (Ell 5)

Group #	Group 1	Group 2	Group 3	Group 4
Group 1		1.0000	1.0000	0.5579
Group 2	1.0000		1.0000	0.1800
Group 3	1.0000	1.0000		1.0000
Group 4	0.5579	0.1800	1.0000	

## Appendix E: ALCOLIN's Permoseal technical data sheet



## **DESCRIPTION:**

ALCOLIN COLD GLUE is the premium multi-purpose wood and paper adhesive. It is a polyvinyl acetate (PVA) which dries by evaporation of water, without heat to form a tough, sturdy, non-visible bond which is stronger than wood.

## **APPLICATIONS:**

ALCOLIN COLD GLUE has excellent adhesion to:

Soft woods e.g. Pine, Meranti

Hard woods e.g. Imbuia, Oak

Processed board e.g. Hardboard, Chipboard

Paper and Cardboard

ALCOLIN COLD GLUE can also be used to bond felt, cloth, leather, stone, cork, etc. Ideal for the DIYer, professional, handicraft enthusiast and for school projects.

## **DIRECTIONS FOR USE:**

Surface Preparation:

Ensure surfaces are clean, dry and free of dust and grease.

Application:

The open time assembly of ALCOLIN COLD GLUE is approx. 10 minutes. It is therefore recommended that only the amount that can be used within 10 minutes, be applied at any one time.

Apply ALCOLIN COLD GLUE to one or both surfaces.

Join the surfaces together immediately and clamp for up to 1 hour.

Remove any excess ALCOLIN COLD GLUE immediately with a damp cloth.

Leave for a minimum of approx. 2 hours before handling normally.

Highest bond strengths are obtained at high clamping pressure and extended clamping times (up to 8 hours).

Cleaning:

Applicators can be clean tools with water, immediately after use.

Storage:

Seal container tightly after use and store in a cool, dry place.



**NOTE:**

Hardwoods should be lightly sanded before applying adhesive.

If ALCOLIN COLD GLUE is to be used outdoors or if it will be exposed to the elements; the joints should be covered with paint or varnish once the adhesive has cured. Weather conditions and moisture content of the wood, may affect the drying time. In cold weather, or if wood is slightly moist, clamp for a longer period of time.

**CAUTION:**

ALCOLIN COLD GLUE is non-toxic and non-flammable.

**TECHNICAL INFORMATION**

Appearance :	Viscous white liquid
Density :	Approximately 1.06 g/cm <sup>3</sup>
Dried film :	Tough, flexible, hazy
Solids :	Approx. 38 %
Viscosity :	Approx. 15 000 cps
Open time assembly :	Approx. 10 minutes
Clamping time :	Between 1 and 8 hours
Curing time :	Approx. 24 hours
Water resistance :	Poor, unless painted or varnished
Coverage :	Approximately 4 m <sup>2</sup> / liter
Shelf life :	Approx. 12 months in a sealed container

Information from **[www.alcolin.com](http://www.alcolin.com)**