Evaluation of the bonding quality of E. grandis cross-laminated timber made with a one-component polyurethane adhesive

by
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Declaration

By submitting this thesis electronically, I declare that the entirety of the work contained therein is my own, original work, that I am the sole author thereof (save to the extent explicitly otherwise stated), that reproduction and publication thereof by Stellenbosch University will not infringe any third party rights and that I have not previously in its entirety or in part submitted it for obtaining any qualification.

Date: March 2018

Summary

Eucalyptus grandis is South Africa's most important commercial hardwood species. The availability of *E. grandis* and its fast growth rate creates the opportunity to explore its uses further. Cross-laminated timber (CLT) is a prefabricated multilayer engineered panel product made of at least three layers, with the grain direction of some or all of the consecutive layers orthogonally orientated. In order to add value to *E. grandis*, reduce the export of low-cost chips, increase the profit margins of local plantation owners and create jobs, the development of *E. grandis* CLT in South Africa may be an option.

There is concern among some researchers that the bonding quality evaluation tests proposed by CLT standards have been developed for glulam and are too severe for CLT. These researchers proposed that further analysis and possibly even revision of the test methods should be considered. There is also a need to evaluate the mechanical properties of CLT panels made of *E. grandis* to completely understand the structural performance of these panels, including their bond quality and durability, and therefore be able to rely on *E. grandis* CLT as a construction material.

The objectives of this study were:

- To evaluate the face-bonding quality of CLT panels from E. grandis timber bonded with a
 one component polyurethane resin;
- To determine the influence of material and processing parameters on the face-bonding quality of CLT manufactured from *E. grandis* timber bonded with a one component polyurethane resin:
- To analyse different testing methods for evaluating the face-bonding quality of CLT.

The design for this experiment consisted of eight groups with different combinations of parameters for density, grooves and pressure per group. Four different testing methods were used to evaluate the face-bonding quality of CLT panels from E. grandis and to determine the effect of parameters on face-bonding quality: A delamination test on $100 \times 100 \text{ mm}$ block specimens (Test A), a shear test on $40 \times 40 \text{ mm}$ specimens (Test B), a shear test on $40 \times 40 \text{ mm}$ specimens with grain direction 45° to load direction (Test C) and a combined delamination and shear test on $70 \times 70 \text{ mm}$ specimens with grain direction 45° to load direction (Test D).

Results of the statistical analysis indicated that *E. grandis* CLT made with 1C-PUR adhesive can obtain excellent face-bonding quality using a clamping pressure of 0.7 MPa and with no stress relief grooves present. All samples passed the shear test (Test B) which is the reference test method proposed by EN 16351 (2015). It was found that a strength component and durability component will be an advantage when testing the bond quality of CLT. Shear tests at 45° to the load direction did not completely eliminate the rolling shear effect. The combined delamination and shear test (Test D), seems to have potential as a good test for bond quality since it is a combination of a durability and shear strength test. There are still questions about the relative advantages of specific test methods for bond quality, especially on the effect of rolling shear. Further work should focus on this aspect and the use of stress models might be a way of gaining further insights.

Keywords: *Eucalyptus grandis*, CLT, 1C-PUR, density, stress-relief grooves, pressure, shear test, delamination test, WFP, bond quality

Opsomming

Eucalyptus grandis is Suid-Afrika se belangrikste kommersiële loofhoutspesie. Die beskikbaarheid van *E. grandis* en sy vinnige groeitempo skep die geleentheid om sy gebruike verder te ondersoek. Kruis-gelamineerde hout (KGH) is 'n voorafvervaardigde, multilaag verwerkte paneelproduk van ten minste drie lae, met die greinrigting van sommige of al die opeenvolgende lae ortogonaal georiënteer. Ten einde waarde toe te voeg tot *E. grandis*, die uitvoer van lae-koste spaanders te verminder, winsmarges te verbeter van plaaslike plantasie-eienaars en vir die skepping van werksgeleenthede, het die ontwikkeling van *E. grandis* KGH in Suid-Afrika potensiaal.

Daar is kommer onder sommige navorsers dat die lasgehaltetoetse voorgestel deur KGH standaarde ontwikkel is vir gelamineerde balke en te streng is vir KGH. Hierdie navorsers het voorgestel dat verdere analise en moontlik selfs hersiening van die toetsmetodes oorweeg moet word. Daar is ook 'n behoefte om die meganiese eienskappe van KGH panele, gemaak van *E. grandis*, te evalueer en die strukturele vermoë van hierdie panele, insluitend hul laskwaliteit -en duursaamheid, volledig te verstaan en dus te kan staatmaak op *E. grandis* KGH as 'n konstruksiemateriaal.

Die doelwitte van hierdie studie was die volgende:

- Evalueer die laskwaliteit op die platkante van KGH panele van *E. grandis* hout gelym met 'n een-komponent poliuretaan hars;
- Bepaal die invloed van materiaal -en verwerkingsparameters op die laskwaliteit van KGH, vervaardig uit *E. grandis* hout, gelym met 'n een-komponent poliuretaan hars;
- Ontleed verskillende toetsmetodes om die laskwaliteit op die platkante van KGH te evalueer.

Die ontwerp van hierdie eksperiment het uit agt groepe met verskillende kombinasies van parameters vir digtheid, spanningsverligtinggroewe en klampdruk per groep bestaan. Vier verskillende toetsmetodes is gebruik om die laskwaliteit van KGH panele van *E. grandis* te evalueer en om die uitwerking van verskillende parameters te bepaal. Die vier toetse was: 'n delamineringtoets op 100 x 100 mm blokmonsters (toets A), 'n skuiftoets op 40 x 40 mm monsters (toets B), 'n skuiftoets op 40 x 40 mm monsters met greinrigting 45° met lasrigting (toets C) en 'n gesamentlike delaminering -en skuiftoets op 70 x 70 mm monsters met greinrigting 45° met lasrigting (toets D).

Resultate van die statistiese analise het aangedui dat *E. grandis* KGH gemaak met 1C-PUR kleefmiddel uitstekende lasgehalte kan verkry met behulp van 'n klampdruk van 0.7 MPa en met geen spanningsverligtinggroewe teenwoordig nie. Alle monsters slaag die skuiftoets (toets B) wat die verwysingstoetsmetode in EN 16351 (2015). Daar is gevind dat 'n sterktekomponent en duursaamheidskomponent 'n voordeel sal wees wanneer die laskwaliteit van KGH getoets word. Skuiftoetse teen 45° met die lasrigting het nie die rolskuifeffek heeltemal uitgeskakel nie. Die gekombineerde delaminering -en skuiftoets (toets D), het oënskynlik potensiaal as 'n goeie toets vir laskwaliteit omdat dit 'n kombinasie van 'n duursaamheid -en skuiftoets is. Vrae bestaan nog oor die relatiewe voordele van spesifieke toetsmetodes vir laskwaliteit, veral op die effek van rolskuif. Verdere navorsing moet fokus op hierdie aspek en die gebruik van spanningsmodelle sal dalk 'n manier wees om verdere insigte daaroor te verkry.

This thesis is dedicated to GOD

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Table of Contents

Declaration	II
Summary	III
Opsomming	IV
Acknowledgements	VI
Table of Contents	
List of Figures	
List of Tables	xii
Acronyms and Abbreviations	xiii
Chapter 1: Introduction	
1.1 Background	
1.2 Problem	1
1.3 Objectives	1
1.4 This study as a continuation of a previous study	2
1.5 Approach and procedure	2
Chapter 2: Literature review	3
2.1 State of forestry and economy	3
2.2 Wood chips	3
2.3 Eucalyptus grandis	4
2.4 Cross-Laminated Timber	4
2.5 PUR adhesive and specifications	5
2.6 Adhesive penetration and bond quality at different densities	6
2.7 Radial variation in wood density	7
2.8 Stress relief grooves	8
2.9 Clamping pressure	9
2.10 Rolling shear	10
2.11 CLT standardisation for testing of adhesive bond quality	12
2.11.1. European standardisation	12
2.11.2. Shear and delamination tests	12
Chapter 3: Materials and methods	14
3.1 Materials	14
3.1.1 Timber	14
3.1.2 Adhesive	14
3.2 Methods	14
3.2.1 Experimental design	
3.2.2 CLT panel production	15
Primary Lumber selection	16
Lumber planing and cutting to length	16

CLT layer formation	16
Secondary lumber selection, planing and cutting to length	17
CLT panel layup	18
Stress relief grooves	18
Adhesive application	19
CLT pressing	20
CLT finishing	20
3.2.3 CLT test sample production	21
Panel breakdown	22
3.2.4 CLT sample testing	23
Test A: Delamination of 100 x 100 mm samples	23
Test B: Shear strength of 40 x 40 mm samples	
Test C: Shear strength of 40 x 40 mm samples at 45° to the grain direction	26
Test D: Delamination and shear strength of 70 x 70 mm samples at 45° to direction	_
3.2.5 Statistical analysis	28
Chapter 4: Results and discussion	29
4.1 Face bonding quality of <i>E. grandis</i> CLT according to EN 16351 (2015)	31
4.2 Comparison of different test methods	31
4.2.1 Test A: 100 x 100 mm	31
4.2.2 Test B: 40 x 40 mm	33
4.2.3 Test C: 40 x 40 mm 45° to grain	34
4.2.4 Test D: 70 x 70 mm 45° to grain	
4.3 Density, grooves and pressure effect	
4.3.1 Test A: 100 x 100 mm	37
Total delamination (Dtot)	37
Wood failure percentage (WFP)	
4.3.2 Test B - 40 x 40 mm	
Shear strength (fv)	42
Wood failure percentage (WFP)	43
4.3.3 Test C - 40 x 40 mm at 45°	45
Shear strength (fv)	45
Wood failure percentage (WFP)	47
4.3.4 Test D - 70 x 70 mm at 45°	50
Total delamination (Dtot)	50
Shear strength (fv)	52
Total delamination (Dtot) vs Shear strength (fv)	54

References	63
Chapter 5: Conclusion	62
4.5 Comparison of different test methods revisited	61
4.4 Shear strength vs WFP – A comparison and correlation analysis	57
Wood failure percentage (WFP)	55

List of Figures

Figure 2-1: Stress relief groove pattern as performed in the study by Mestek and Winter (2010
Figure 2-2: Adhesive failure vs normal wood failure vs rolling shear failure (left) and rolling shear
failure of cross laminated specimen (right) (Kim et al., 2013)11
Figure 3-1: The sawing pattern used by Merensky Timber for the 20-25 year old E. grandis as
well as the positions of the 3 boards per log supplied for the study. Heartwood and sapwood
are also shown in dark and light (Pröller, 2017)14
Figure 3-2: Production of 300 x 300 mm CLT panels from individual boards
Figure 3-3: OEST Ecopur HK gluing system for 1C-PUR adhesives (left) and Facetac HAV10
application gun (right) (Oest 2016)16
Figure 3-4: Edge laminating press fully loaded with 3 panels. Vertical clamps are applying
pneumatic pressure to flatten the panel, while six clamps are applying 0.75 MPa of pressure
for edge bonding17
Figure 3-5: 2400 x 500 x 38 mm edge laminated panel showing the location of the 10 density
samples taken to achieve a density profile across the width of the panel18
Figure 3-6: Top and bottom lamellae in the main strength direction with grooves on their inne
faces and a middle lamella, which was orientated 90° to the bottom and top lamellae, with
grooves on both faces19
Figure 3-7: Adhesive application pattern
Figure 3-8: CLT press loaded with six CLT panels under pressure
Figure 3-9: Finished CLT panel (300 x 300 x 75 mm) (left) and panel 16 of 40 with the positions
marked (A – I) (right)21
Figure 3-10: Four different sample types for the four proposed testing methods: a) 100 x 100
mm samples for delamination testing, b) 40 x 40 mm samples for shear testing, c) 40 x 40
mm samples at 45° to the grain direction for shear testing and d) 70 x 70 mm samples at 45°
to the grain direction for delamination and shear testing21
Figure 3-11: 300 x 300 mm CLT panel and the positions and sizes of the different samples to be
cut from the panel. The letters (A- I) indicate the respective positions within the panel as a
means of changing the sample position from panel to panel and keeping track of the
sample's origin22
Figure 3-12: Samples in autoclave, weighed down and about to be submerged in water (left
and hammer and chisel used to split the glueline to estimate WFP (right)24
Figure 3-13: 40 x 40 sample showing shear test method (left) and shearing tool with self-
aligning cylindrical bearing (right)25
Figure 3-14: 40 x 40 sample showing shear test method at 45° to the grain direction 26
Figure 3-15: 70 x 70 sample showing shear test method at 45° to the grain direction 27

Figure 3-16: WFP (approximately 60%) of a 70 x70 sample. Blue indicates wood failure 27
Figure 4-1: 100 x100 mm - Graph showing the effect of pressure on total delamination (<i>Dtot</i>). 37
Figure 4-2: 100 x 100 mm - Graph showing the effect of the density and grooves interaction or
total delamination (<i>Dtot</i>)38
Figure 4-3: 100 x 100 mm - Graph showing the effect of pressure on WFP (%)40
Figure 4-4: 100 x 100 mm - Graph showing the effect of the density and grooves interaction or WFP (%)41
Figure 4-5: 40 x 40 mm - Graph showing the effect of the three-way interaction of density
grooves and pressure on shear strength (fv)42
Figure 4-6: 40 x 40 mm - Graph showing the effect of the three-way interaction of density grooves and pressure on WFP
Figure 4-7: 40 x 40 mm at 45° to grain - Graph showing the effect of the density and grooves interaction on shear force (fv)
Figure 4-8: 40 x 40 mm at 45° to grain - Graph showing the effect of the grooves and pressure interaction on shear strength (fv)46
Figure 4-9: 40 x 40 mm at 45° to grain - Graph showing the effect of the density and grooves interaction on WFP48
Figure 4-10: 40 x 40 mm at 45° to grain - Graph showing the effect of the grooves and pressure interaction on WFP49
Figure 4-11: 70 x 70 mm at 45° to grain - Graph showing the effect of the density and grooves interaction on total delamination (<i>Dtot</i>)
Figure 4-12: 70 x 70 mm at 45° to grain - Graph showing the effect of pressure on tota delamination (<i>Dtot</i>)
Figure 4-13: 70 x 70 mm at 45° to grain - Graph showing the effect of the interaction between density and grooves on shear strength (<i>fv</i>)
Figure 4-14: 70 x 70 mm at 45° to grain - Graph showing the effect of the interaction between density and pressure on shear strength (fv)53
Figure 4-15: 70 x 70 mm at 45° to grain - Graph showing the linear model of total delamination (<i>Dtot</i>) vs shear strength (<i>fv</i>)54
Figure 4-16: 70 x 70 mm at 45° to grain - Graph showing the effect of the three-way interaction of density, grooves and pressure on WFP (%)55
Figure 4-17: 40 x 40 mm vs 40 x 40 mm 45° to grain – Graphs of the comparison of the effect o pressure on shear strength (fv) (top) and WFP (bottom)57
Figure 4-18: 40 x 40 mm (top) and 40 x 40 mm at 45° to grain (bottom) - Graphs showing the linear model of WFP (%) vs shear strength (<i>fv</i>)59
Figure 4-19: 70 x 70 mm at 45° to grain - Graph showing the linear model of WFP (%) vs shear strength (fv)60

List of Tables

Table 2-1: Average density and shrinkage of <i>E. grandis</i> (a = 12-15 years old, b = 35 years old)
at 10% MC (Wand, 1990)8
Table 3-1: The 2 x 3 factorial design with three factors at two levels each
Table 3-2: The eight sample groups with the three factors being tested and their upper and
lower levels
Table 4-1: Summary of the results for all four test methods for each of the groups $(1 - 8)$ 29
Table 4-2: Ranking differences between different tests and groups. Rankings used were for
mean Dtot (Test A), mean shear strength (Test B), mean shear strength (Test C) and mean
Dtot and mean shear strength (Test D)
Table 4-3: ANOVA table of the total delamination ($Dtot$) values of 100 x 100 mm samples 37
Table 4-4: ANOVA table of the WFP values of 100 x 100 mm samples39
Table 4-5: ANOVA table of the shear strength <i>(fv)</i> values of 40 x 40 mm samples42
Table 4-6: ANOVA table after Box-Cox tranformation of the WFP values of 40 x 40 mm samples
43
Table 4-7: ANOVA table of the shear strength (fv) values of 40 x 40 mm samples with grain
angle 45° to the load direction45
Table 4-8: ANOVA table after Box-Cox transformation of the WFP values of 40 x 40 mm
samples with grain angle 45° to the load direction
Table 4-9: ANOVA table after Box-Cox transformation of the total delamination (Dtot) values of
70 x 70 mm samples with grain angle 45° to the load direction
Table 4-10: ANOVA table after Box-Cox transformation of the shear strength (fv) values of 70 x
70 mm samples with grain angle 45° to the load direction
Table 4-11: ANOVA table after Box-Cox transformation of the WFP values of 70 x 70 mm
samples with grain angle 45° to the load direction.

Acronyms and Abbreviations

1C-PUR One-Component Polyurethane Resin

ANOVA Analysis Of Variance

ANSI American National Standards Institute

APA American Plywood Association

CLT Cross-Laminated Timber

DWAF Department of Water Affairs and Forestry

LS Least Squares

MC Moisture Content

PUR Polyurethane Resin

RH Relative humidity

WFP Wood Failure Percentage

glulam glued laminated timber

Chapter 1: Introduction

1.1 Background

Eucalyptus grandis is South Africa's most important commercial hardwood species. Due to the extreme deformation, splitting and collapse that occurs when *E. grandis* is processed (cut and dried), it is generally not considered as high value saw timber. It is currently mostly used for pulp and paper products and wood chips, most of which are exported to China and Japan.

The availability of *E. grandis* and its fast growth rate creates the opportunity to explore its uses further. Cross-laminated timber (CLT), is a prefabricated multilayer engineered panel product made of at least three layers, with the grain direction of some or all of the consecutive layers orthogonally orientated. Lower grade wood (such as *E. grandis*) can be utilised in CLT as it is very effective at minimising the effect of wood defects, which usually disqualify *E. grandis* for structural applications. *E. grandis* has short cultivation cycles and relatively good mechanical properties, making it an ideal candidate for CLT applications (Liao et al., 2017).

In order to add value to *E. grandis*, reduce the export of low-cost chips, increase the profit margins of local plantation owners, and create jobs, the development of *Eucalyptus* CLT in South Africa may be an option. With the predicted shortage of saw timber in South Africa, a move towards sustainable building, and the need to stay internationally competitive, adding value to *E. grandis* by using it for CLT would provide a timeous solution to many of the abovementioned problems.

1.2 Problem

Eucalyptus grandis is one of the species with extremely high shrinkage coefficients (Piter et al., 2004). There is concern that this might lead to poor bond durability between orthogonal CLT layers, especially in an environment where there will be moisture fluctuations.

Currently, there is a need to evaluate the mechanical properties of CLT panels made of *E. grandis* to completely understand the structural performance of these panels, including their bond quality and durability, and therefore be able to rely on *E. grandis* CLT as a construction material.

There is also a concern among researchers that the bonding quality evaluation tests proposed by CLT standards have been developed for glulam and are too severe for CLT (Betti et al., 2016 and Knorz et al., 2017). These researchers proposed that further analysis and possibly even revision of the test methods should be considered.

1.3 Objectives

The objectives of this study were:

- To evaluate the face-bonding quality of CLT panels from *Eucalyptus grandis* timber bonded with a one component polyurethane resin;
- To analyse different testing methods for evaluating the face-bonding quality of CLT;
- To determine the influence of material and processing parameters on the face-bonding quality of CLT manufactured from *E. grandis* timber bonded with a one component polyurethane resin.

1.4 This study as a continuation of a previous study

The edge gluing of wet *E. grandis* boards into panels before kiln drying to potentially reduce deformation and "drying defects" was investigated by Pröller (2017). Wet boards are boards that have a moisture content (MC) higher than fibre saturation point (FSP). He proposed that the panels could be ripped into sawn lumber again or maintained as a panel product. Although cup and twist were significantly reduced in individual boards by edge bonding them together before kiln drying, it is doubtful that the slight reduction in these two properties will justify the green edge gluing process if sawn lumber is the intended end-product.

Since the full sized panels successfully inhibited excessive warp it will be better to utilise the panels in another way rather than ripping it into structural lumber. Stellenbosch University proposed the new concept of using green edge-laminated *E. grandis* panels to manufacture three-layered cross-laminated timber (CLT). The theory is that the edge gluing and pressing prior to drying would have a stabilising effect on the wood and serve as to reduce "drying defects" in the panel to stabilise it for use as individual CLT layers.

1.5 Approach and procedure

The approach used standard test procedures, namely, delamination and shear tests, material specifications and construction methods described in EN 16351 (2015), along with two new testing procedures adapted from tests performed by Betti et al. (2016), to evaluate the bonding quality of CLT in a sample of test specimens. Results were analysed to evaluate conformance to current standards and possibly to establish new testing procedures and standards.

Chapter 2: Literature review

2.1 State of forestry and economy

South Africa has approximately 1.3 million hectares of plantations of commercial value, making up only 1.2% of the country's total land area. The *Pinus* species accounts for approximately 51% of the total plantation area, with *Eucalyptus* accounting for about 40% (DAFF, 2012). Afforestation has declined significantly in recent years due to a shortage of suitable forestry land, the difficulty in obtaining the necessary water licenses and the focus on returning previously afforested areas to natural vegetation. It is forecasted that South Africa will experience a major shortage in saw timber in the near future (Crickmay et al., 2004). 74.3% of the newly afforested areas in recent times is *Eucalyptus* (DAFF, 2009). There is also potential for afforestation in the Eastern Cape with as much as 100 000 hectares available. This area can be planted with *Eucalyptus grandis* in an attempt to make use of this fast growing species as well as bringing an income to the rural, poverty-stricken areas of the Eastern Cape (DWAF, 2009).

In the small-scale sawmilling industry, as much as 80% is owned by the previously disadvantaged. A high percentage of the employees are women with almost all of them coming from rural areas. This presents massive potential for increasing the volume of timber provided to this sector, with a view to improving the state of poverty in the rural areas (DWAF, 2009).

According to Forestry South Africa (2008), it is predicted that there will be a 9 million ton deficit in domestic timber supply by 2030. With an ever expanding saw timber market and a move toward sustainable building, it is predicted that South Africa will not be able to keep up with the domestic demand (Crafford, 2013).

The forest and forest products industry is responsible for the creation of 170 000 direct jobs. The potential is there for a large number of new jobs to be created as the demand increases (DWAF, 2005). One of these potential ideas is to set up cross-laminated timber (CLT) plants in South Africa. This will result in value adding to the product as well as increasing the number of job opportunities as CLT plants are far more labour intensive than the pulp sector (DAFF, 2012).

In terms of the economic sustainability of the forestry sector, forestry contributes as much as R22 billion per annum to the Gross Domestic Product (GDP). South Africa's commercial plantations produce approximately 22 million m³ of round wood worth about R 5.1 billion per annum (DWAF, 2009).

As most of the saw timber available in South Africa is from the *Pinus* species (softwood), and most of the plantations that were removed to return to natural vegetation were in the Western Cape, the shortage in saw timber will be most notable in softwood. A possible solution to the timber shortages could be to utilise *E. grandis*, by far South Africa's most important hardwood species, which is currently used for pulp and paper products and wood chips (83.6% in 2013) (DAFF, 2015 and Pröller, 2017). This could be a possible solution to meeting the timber needs that are currently being experienced and that are forecasted to increase in the future.

2.2 Wood chips

Because of the low beneficiation and job creation of the chip industry, the use of *Eucalyptus* for CLT is a great opportunity from a national perspective.

With an increased reliance on electronics and digital reading, there has been a decreased reliance on paper and paper products and this has led to a decline in the pulp and paper industry. This industry makes use of most of the hardwood supply in the country. That which is

not used by the pulp and paper mills is exported in chip form with 98% of exported chips going to Japan. In 2003, 2.1 million bone dry tons (BDT) of *Eucalyptus* chips were exported to Japan (Japanese Import Statistics, 2004 and LHA 2004:13). The chip export market is a somewhat controversial one in South Africa. While it leads to higher income for small plantation owners, it also leads to a loss of beneficiation in terms of value adding that may have taken place (Chamberlain et al., 2005).

As of 2003, woodchip exports create employment for at most 500 people across the four woodchip mills making it a very small employer relative to the log volume processed (DWAF, 2004a).

2.3 Eucalyptus grandis

Eucalyptus grandis was introduced into South Africa approximately 105 years ago mainly as a source of mining timber (Malan, 1995). With more than 269 000 ha of plantation area, *E. grandis* makes up more than half the South African *Eucalyptus* plantation area and is by far the most important hardwood species in South Africa (DWAF, 2009).

E. grandis is one of the fastest growing tree species in the world with a mean annual growth rate of as much as 40-50 m³/ha/annum. More than 14 million hectares of *E. grandis* have been planted in commercial timber plantations worldwide due to its adaptability, high growth rate, good form, good strength properties and excellent fibre properties (Louppe et al., 2008).

Due to the high growth rate of *E. grandis*, the species has been extensively studied for the high growth stresses present during the growing, felling, sawing and drying phases of the timber life cycle (Wand, 1990). These growth stresses are responsible for checking and end splitting. End splitting is as a result of the partial release of growth stresses and is seen as the defect with the biggest negative impact on yield and product dimensions (Malan and Gerischer, 1987). Another defect affecting the quality of the sawn timber, brittle heart, is a condition arising from compression failure in the central part of the stem of the tree, but is more likely to occur in older trees than younger trees (Walker, 2010). The presence of this defect significantly affects the quality and yield of the end product (Malan, 1995).

This leads to a higher proportion of the saw logs being classified as unsuitable for use as structural timber (Zobel and Sprague, 1998 and Nel, 1965).

2.4 Cross-Laminated Timber

Cross-laminated timber (CLT) is a prefabricated multilayer engineered panel product made of at least three layers, with the grain direction of some or all of the consecutive layers orthogonally orientated. These panels are constructed by bonding the surfaces together with an appropriate adhesive under pressure for a certain length of time (Yeh et al., 2013 and Sikora et al., 2016). The orthogonal orientation of successive layers effectively reduces the anisotropic property of wood, leading to a structurally stable CLT product (Nie, 2015).

CLT was first developed in the 1970s in Europe, but has only been in commercial production for approximately the last 20 years, while North America and Canada have only begun the process of CLT structural system design and product qualification within the last 15 years (Kim et al., 2013 and Yeh et al., 2013). The boom in the development of CLT in the 1990's was due to the sawmilling industry finding a higher value return for the sideboards than what they had at the time (Guttmann, 2008).

Global production of CLT was approximately 625,000 m³ in 2014, and was forecasted to increase to approximately 700000 m³ in 2015. In 2014, Europe was responsible for the

production of about 90% (560,000 m³) of global CLT, with this figure estimated to reach 630,000 m³ in 2015.

Typical softwoods, such as spruce (*Picea spp.*), lodgepole pine (*Pinus contorta*) and Douglas fir (*Pseudotsuga menziesii*) are the common species which are used for the construction of CLT in Europe and North America (Liao et al., 2017 and OpenEI, 2010).

As CLT technology and knowledge has developed, so too has the potential for using hardwoods that aren't usually successful as construction grade timber. As these hardwoods cannot be used for structural applications, there is an oversupply of timber. CLT is an ideal alternative, sustainable building material which allows for value adding to the product to take place (Kramer et al., 2014).

Several studies have been performed attempting to use hardwoods for CLT due to the high rolling shear modulus and high strength characteristics of hardwoods. Liao et al. (2017) explored the feasibility of manufacturing three-layer CLT using fast-grown small diameter *Eucalyptus* wood (*E. urophylla* x *E. grandis*).

Mohamadzadeh and Hindman (2015) confirmed that yellow-poplar (*Liriodendron tulipifera*) CLT displayed acceptable mechanical performance and could be a starting point for using hardwoods in CLT structural design.

E. grandis has relatively good mechanical properties and short cultivation cycles making it an ideal candidate for CLT applications (Liao et al., 2017).

2.5 PUR adhesive and specifications

The adhesive that is used must satisfy the structural adhesive requirements of the three main CLT standards for adhesive qualification, namely the American National Standards Institute (ANSI) and American Institute of Timber Construction (AITC) 405, Canadian Standards Association (CSA) O112 and the European Standard EN 302 (2013), Adhesives for load-bearing timber structures - Test methods.

According to EN 16351 (2013), the US CLT Handbooks (Karacabeyli and Douglas, 2013) and the Canadian CLT Handbooks (Gagnon and Pirvu, 2011), there are three types of adhesive systems allowed for CLT production, namely:

- Phenoplast and aminoplast adhesives (melamine-urea-formaldehyde (MUF) and phenol-resorcinol-formaldehyde (PRF) adhesives),
- One-component polyurethane adhesives (1C-PUR) and
- Emulsion–polymer–isocyanate adhesive (EPI).

The two adhesive types dominating the structural timber industry are melamine urea formaldehyde resins (MUR) and polyurethane resins (PUR). PUR is free of solvents and carcinogen- containing formaldehydes and is, for these reasons, more popular than MUR (Crespell and Gagnon, 2010). The first 1C-PUR adhesives entered the engineered wood timber market more than 20 years ago in the form of PURBOND HB 110 (Purbond AG/Switzerland). Since then, 1C-PUR adhesives have captured a large market share as they offer several benefits to the traditional adhesive systems. These benefits include:

- 1. No mixing needed
- 2. Reduced pressing time
- 3. Ductile bondline

- 4. Invisible bondlines
- 5. Fast bonding at room temperature

1C-PUR adhesives are now so widely accepted that they have started replacing the conventional adhesive systems (MUF, PRF and EPI) in finger jointing, glulam and now even CLT applications (Lehringer and Gabriel, 2014). This acceptance has largely been due to the emergence of its own European standard for classification and requirements EN 15425 (2008).

Many tests have been conducted using 1C-PUR adhesives to manufacture CLT with mixed results. Sikora et al. (2016) compared PUR and PRF systems at different clamping pressures and test configurations and found that the adhesive type had little or no effect on structural bonding performance. In a study conducted by Luedtke et al. (2015) to examine the influences of primer treatment and other parameters on some common European hardwood species, a 1C-PUR adhesive (PURBOND HB S109) was used as an alternative to the more common aminoand phenoplast adhesives which were used for bonding these hardwoods. It was concluded that 1C-PUR adhesives, in combination with a primer, provide good alternatives to the more common adhesives used in hardwood gluing. Kim et al. (2013) used PUR in the study on the shear performance of PUR in cross laminating of red Pine (Pinus densiflora) because it very conveniently doesn't require a hardener and is commonly used in Europe for CLT manufacturing. It was found that there was a significant difference between cross-lamination and parallel-to-grain lamination for shear strength, but no significant difference was found for wood failure. Based on these findings, the effect of the laminating direction should be considered as an important factor when correctly evaluating the adhesive performance of CLT. Aicher et al. (2016) used 1C-PUR Loctite HB S139 Purbond with an applicable primer on CLT manufactured from Beech Wood as 1C-PUR is not approved without a primer for European beech wood (Fagus sylvatica) applications. A spread rate of 180g/m², assembly time of 10-12 minutes and clamping pressure of 1.2 MPA for 2.5 hours were employed. No manufacturing or bonding problems were found. Sikora et al. (2014) used a 1C-PUR adhesive (PURBOND HB S309) to manufacture CLT specimens for shear and delamination testing. It was found that wood failure percentage (WFP) for PUR adhesives is very high and the results for shear and delamination tests were in accordance with the requirements of EN 16351 (2013).

2.6 Adhesive penetration and bond quality at different densities

The interaction between the wood and adhesive is essential for the understanding of bonding in laminated wood products and how certain factors can affect the quality of this bond. The absorption of a given adhesive into wood is limited by the porosity of the wood. *E. grandis* and other diffuse porous hardwoods are made up of vessels, tracheids, fibres and parenchyma (Crafford, 2013). Vessels in hardwoods, and specifically *E. grandis*, are the most important elements for the flow of adhesive through the wood. *E. grandis* is characterised by thick cell walls, small cell lumina, small pores and small early wood vessels and consequently a decreased adhesive permeability compared to softwoods and ring porous hardwoods (Kamke and Lee, 2007). Due to the diffuse porous structure, there is a uniform pattern of adhesive distribution along the entire length of the glueline. This can be seen as an even bondline with slight and even squeeze out.

Adhesive penetration into the wood cell structure occurs as a result of both gross penetration and cell wall penetration. Gross penetration is the forcing of adhesive into the cell lumina as a result of compression clamping. Cell wall penetration is the diffusion of adhesive into the cell walls and as a result of charged elements in the adhesive and wood aspiring to reach a state of neutrality (Kamke and Lee, 2007).

However, Lehringer and Gabriel (2014) stated that it is unknown if 1C-PUR adhesives can actually penetrate cell walls in *E. grandis*. For this reason, it can be theorised that most of the adhesive penetration into the wood takes place via vessel elements (Pröller, 2017).

Sterley (2012) theorised that deeper adhesive penetration may increase the bonding strength by improving the stress distribution within the bondline under load. Kamke and Lee (2007) proposed that penetration depth would lead to improved bond strength due to increased interaction between wood and adhesive in the form of intermolecular bonding such as van der Waals forces and hydrogen bonds.

Sikora et al. (2016) recognised the effect that bonding pressure had on bond durability using a PUR adhesive. It was found that higher pressure was directly correlated with deeper penetration and consequently better bond durability. Lower pressures led to shallower penetration and the formation of a thick glueline with a large glue area for exposure to water during delamination testing, resulting in poor results for bonding quality.

Sterley (2012) reported that pressing time had a substantial effect on bonding strength. Increased pressing time allowed time for deeper penetration into the cell structure and increased bonding strength. It was further proposed that increasing the pressing time would lead to improved delamination and shear results of bonded hardwoods.

Two theories exist for the effect that bondline thickness has on delamination behaviour.

- 1. Kamke and Lee, (2007) observed the phenomenon where greater adhesive penetration enhances mechanical adhesion, but at the same time leads to a lack of adhesive at the surface of the bondline causing adhesive starvation at the actual glueline and higher delamination values. Pröller (2017) reported that 1C-PUR adhesives used on dry, high density woods showed increased delamination as a result of poor adhesion quality. The reason for this phenomenon is that PUR adhesives struggle to penetrate high density wood substrates at a low MC, causing excessive adhesive squeeze out and a thin bondline.
- 2. Wetzig (2009) found that thick 1C-PUR gluelines have high ductility and contribute to the absorption of swelling and shrinkage stresses in hardwoods, leading to improved delamination results.

High density wood species have properties which make bonding with adhesive extremely difficult. They have small cell lumens because of their thick cell walls, making adhesive penetration extremely difficult and severely compromising the depth of mechanical interlock to two cells deep. Higher clamping pressure is required to compress the high stiffness, high density wood with large numbers of growth stresses in order to bring the wood layers and the adhesive into contact with each other. High density woods have larger amounts of extractives which may interfere with adhesive curing and subsequent bond formation (Frihart and Hunt 2010).

2.7 Radial variation in wood density

Malan (2005) reported on the effect that the radial position of the board in relation to the pith has on the properties of that board in *E. grandis*. Wood closer to the pith tends to be of lower strength, fibres are shorter and pith tissue might be present. Nel (1965) and Zobel and Sprague (1998) found that short fibre length and high micro fibril angle near the pith in *Eucalyptus* leads to a low impact strength and brash failures. As the trees get older, the inner zones close to the pith will come under greater compression leading to the formation of brittle heart and poor quality wood. This leads to a higher proportion of the saw logs being classified as unsuitable for use as structural timber.

Hardwoods are made up of four different types of cells, namely parenchyma, tracheids, fibres and vessels. The wood density is then a product of the ratio between cell wall and cell lumen along with the number and size of vessel elements present (Pröller, 2017). Malan (1995) and Malan and Gerischer (1987) reported that the length, diameter and wall thickness of fibres as well as vessel diameter are directly correlated with density in *Eucalyptus*.

Kramer et al. (2014) investigated the viability of using a low density hardwood, hybrid poplar (*Pacific albus*), for manufacturing performance rated CLT. In that study, attention was drawn to the lower limit of lumber density, 350 kg/m³ as stated in ANSI/APA (2012), the standard for performance rated cross-laminated timber. As wood density is generally correlated with enhanced mechanical properties such as strength and stiffness, ANSI/APA (2012) limits the use of low density species to ensure that structural quality products are produced (Pröller, 2017). In Table 2-1, Wand (1990) displays the average density at 10% MC of 12-15 year old and 35 year old *E. grandis* timber in South Africa.

Table 2-1: Average density and shrinkage of *E. grandis* (a = 12-15 years old, b = 35 years old) at 10% MC (Wand, 1990).

Green Moisture Content	Green D	ensity	Density moisture		Percentage	e shrinka to oven	_	shrinka		ercentag nkage fr een to 10 ture cor	ge from o 10%	
%	Average g/cm ³	Range g/cm ³	Average g/cm ³	Range g/cm ³	Shrinkage volumetrica Ily determined	Radial	Tange ntial	Longit udinal	Radial (R)	Tange ntial	$\frac{R+T}{2}$	
a) 88	0.88	0.77- 0.94	0.57	0.5-0.8	14.8	5.48	9.98		3.07	6.49	4.78	
b) 97	1.02	0.54	0.66		18.8	6.32	12.05	0.23	4.24	9.4	6.82	

It has been well documented that density in *E. grandis* increases rapidly from the pith to the bark, especially in the juvenile wood zone (Bhat et al., 1990; Malan, 2005 and Wand, 1990). Research done by Perez del Castillo (2001) also found better strength and stiffness values for Uruguayan *E. grandis* in boards further from the pith than close to it.

2.8 Stress relief grooves

Stress relief grooves are grooves that can be cut along the grain direction by sawing through partial thickness of the lumber. These grooves release stresses, developed in CLT panels by moisture loss, and in turn reduce the chances of warping and the development of cracks. However, caution is needed to ensure that the grooves are not too wide or deep as this will reduce the bonding area and may lead to a reduction in the performance of the CLT panel (Karacabeyli and Douglas, 2013). EN 16351 (2015) specifies that grooves may have a maximum depth of 90% of the thickness of the lamination and a maximum width of 4 mm.

Pröller (2017) found that the presence of stress relief grooves cut in green *E. grandis* boards prior to kiln drying was unable to reduce the effects of the defects, namely check, split, bow, cup or twist in the structural lumber product that he investigated. Due to the excessive deformation and subsequent reduction in strength properties caused by the grooves during drying, it is advised to cut the grooves after drying and immediately prior to laminating and CLT panel manufacturing (Pröller, 2017).

Mestek and Winter (2010) investigated the effect of stress relief grooves parallel to the grain on the rolling shear capacity in CLT panels. They found that tension perpendicular to the grain direction appears in the corners of the relief grooves because of shear deformation. These corner areas have reduced rolling shear capacity and failure is thus expected in these regions. The arrangement of the grooves is shown in Figure 2-1.

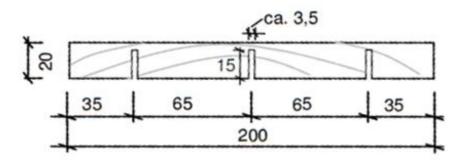


Figure 2-1: Stress relief groove pattern as performed in the study by Mestek and Winter (2010).

Gereke et al. (2010) investigated the use of stress relief grooves in the middle composite layer of spruce cross-laminates and composite laminates. It was found that the slits had no significant influence on moisture-induced stresses but did increase the thermal insulation.

1C-PUR adhesives react chemically with water in the wood and air which leads to slight foaming upon curing. This leads to the formation of CO₂ gas cavities within the bondline and consequently an increased bondline thickness. For this reason, there is a strict tolerance on the glueline thickness for PUR in CLT, namely (0.1 to 0.3) mm (Brandner, 2013; Brandner et al., 2016 and Sikora et al., 2016). Stress relief grooves may create space for adhesive absorption to ensure a thin glueline within the thickness tolerance.

2.9 Clamping pressure

There are two main types of presses available for CLT manufacturing, namely a hydraulic press which uses rigid plates and a vacuum press which uses a flexible membrane for pressing. A vacuum press can generate a pressure of up to 0.1 MPa, while a rigid hydraulic press has a huge range and can generate pressures ranging from 0.1 MPa to 1 MPa and even more (Karacabeyli and Douglas, 2013).

The 0.1 MPa of pressure generated by the vacuum press may not be sufficient to suppress the potential warping of layers and nullify surface irregularities so as to create intimate bonding contact. Another consequence of lower manufacturing pressures is shallower adhesive penetration resulting in a thick bondline and subsequent greater chance of delamination as there is a larger adhesive surface exposed to water during testing (Sikora et al., 2016).

The 1 MPa of pressure generated by the rigid hydraulic press may be too high and subsequently cause damage to the surface of the wood by crushing the cell structure. Another consequence of very high manufacturing pressures is a reduction in adhesive penetration due to the phenomenon known as "squeeze out" where the high pressure forces the glue to be squeezed out the side of the lamellas. This may lead to reduced shear resistance as well as insufficient bonding as a result of a glueline that is too thin (Brandner, 2013 and Sterley, 2012). Sikora et al. (2016) found, however, that higher pressures may be associated with deeper glue penetration on the bonded surfaces and thus better bonding results.

In a study performed on Irish Sitka spruce (*Picea sitchensis*), Sikora et al. (2016) reported that the manufacturer of the PUR adhesive recommended a pressing pressure of 0.6 to 1 MPa

for softwoods. A lower pressure of 0.4 MPa was also tested with low minimum wood fibre failure results obtained, suggesting poor durability. It was further observed that a pressure of 1 MPa, the highest pressing pressure tested, provided the most durable bonds. However, the 0.4 MPa pressure was sufficient to fulfil the EN 16351 (2013) shear strength requirements for Irish Sitka spruce.

Knorz et al. (2017) investigated the use of a hydraulic press (0.8 MPa) and a vacuum press (0.08-0.09 MPa) to manufacture spruce cross laminates with a 1C-PUR adhesive. It was found that the pressing pressure had a negligible effect on both delamination and wood failure. This is in stark contrast to the results achieved by Sikora et al. (2016) who found that increasing bonding pressure in the range (0.4-1 MPa) had a positive effect on bond durability and wood failure percentage (WFP) when using a 1C-PUR adhesive. Knorz et al. (2017) concluded that further research on pressing pressure is needed.

The reference pressure for softwood glulam is 0.6 MPa and this value is generally acceptable for CLT from softwoods such as spruce. However, regulations for an ideal surface bonding pressure for CLT still need to be established (Brandner, 2013).

Liao et al. (2017) used pressing pressures of 0.6 MPa, 0.8 MPa and 1 MPa to determine the effect of pressing pressure on the mechanical properties of CLT manufactured from a hardwood, namely fast-grown small diameter *Eucalyptus* wood (*E. urophylla x E. grandis*), using a 1C-PUR adhesive. The conclusion was made that higher pressing pressure and longer pressing time duration allowed for increased adhesive penetration in the wood substrate and subsequently improved the bonding strength.

Kramer et al. (2014) used a pressing pressure of approximately 0.98 MPa to laminate a hardwood, hybrid poplar, with a density of 300-350 kg/m³ using a phenol-resorcinol-formaldehyde (PRF) resin.

2.10 Rolling shear

Despite the undisputed advantages of CLT as a structural material, the weak rolling shear property of CLT is a major concern (Nie, 2015). Aicher et al. (2016) reported on the low strength and stiffness properties of softwoods in rolling shear and found that it led to extensive shear deformation as a result of the shear lag between the layers parallel to the main span direction and the cross-layers.

Li (2014) defined rolling shear stress as the shear stress in the radial-tangential plane of wood which was perpendicular to the longitudinal grain direction and Fellmoser and Blaß (2004) defined rolling shear in CLT as shear stresses leading to shear strain in the radial-tangential plane. Booth and Reece (1967) stated that rolling shear stresses result in shear strain perpendicular to the grain.

Mestek et al. (2008) found that because of the anisotropic nature of wood, the strength and stiffness properties of shear in the radial-tangential plane is substantially lower than the shear capacity in the parallel to grain direction. Mestek and Winter (2010) found that shear fracture occurs in the cross layers of CLT because rolling shear capacity is substantially lower than shear capacity which is parallel to the grain. Kim et al. (2013) reported the same findings while Blass and Gorlacher (2000) went even further by stating that rolling shear strength is as little as 10% of the strength of normal shear strength. Based on limited results from testing on solid-sawn wood, The Wood Handbook (FPL, 2010) concluded that the rolling shear capacity is between 18 and 28% of the value of the shear capacity parallel to the grain. A visual representation of rolling shear failure is displayed in Figure 2-2.



Figure 2-2: Adhesive failure vs normal wood failure vs rolling shear failure (left) and rolling shear failure of cross laminated specimen (right) (Kim et al., 2013).

EN 16351 (2015) prescribes shear tests on face bonds as a method of testing bond quality. In this test, the load is applied such that it is parallel to the wood grain of one adherend and perpendicular to the grain of the other adherend. According to the definition for rolling shear, these tests cause rolling shear stress (shear stress perpendicular to the grain) in one adherend and parallel-to-grain shear stress in the other adherend (Betti et al., 2016). This may be problematic as the test would then evaluate the wood property, known as rolling shear, rather than the intended bonding quality.

As CLT standards are developed from the glulam standards, the same test for evaluating adhesive bond quality is prescribed by CLT standards. However, Blass and Gorlacher (2000) stated that rolling shear properties limit the structural behaviour of CLT and rolling shear failure takes place only in cross-laminating practices and not laminations bonded exclusively along the grain direction. This feature of CLT results in much lower load capacity when compared to glulam where all the plies are laminated with a parallel orientation to the strength axis of the member (Hindman and Bouldin, 2013; Kim et al., 2013 and Aicher et al., 2016).

Since the rolling shear strength varies for different species and classes of wood, European Eurocode 5 (EN 1995-1-1, 2004) has given a rolling shear strength of 1.0 MPa as a general value for all wood species and classes (Nie, 2015). For CLT panels where edge bonding of the individual lamellas takes place, that value is increased to 1.1 MPa (EN 16351, 2015). Liao et al. (2017) found that normal rolling shear failure was somewhat limited when the middle lamella in CLT made of fast-grown *Eucalyptus* lumber was edge glued. Edge gluing the boards prior to panel lay-up restrained shear deformation more than a middle layer with gaps between boards.

There are various factors that influence rolling shear such as wood species, clamping pressure, sawing pattern, annual ring width, type of adhesive and type of loading (Kim et al., 2013 and Zhou, 2014). Brandner et al. (2016) discovered a significant relationship between rolling shear modulus and the annual ring pattern, while Zhou (2014) concluded that a direct relationship exists between manufacturing pressure or cross-layer lumber quality and rolling shear strength.

Buck et al. (2016) tested CLT panels manufactured with alternating layers of 90° and ±45° to the grain direction. The purpose of doing this was to test the load bearing capacity based on the theory that alternating layers at ±45° minimises the risk of rolling shear. Standard EN 13354 (EN 13354, 2008) proposes a similar method of preventing rolling shear in samples. For shear testing, lamellas are perpendicular to each other but orientated at 45° to the load axis. This causes both the lamellas that are being tested to have the same grain direction with respect to the load being applied ensuring that there is not only one weak surface out of the two that make up the glued joint (Betti et al., 2016).

2.11 CLT standardisation for testing of adhesive bond quality

2.11.1. European standardisation

Production of engineered wood products is usually controlled by standards for the certification of the product. Glulam is a laminated wood product, similar to CLT, with several layers bonded together with parallel grains. It is used as a reference product for CLT panels as glulam has been successfully used in industry for a long period. The standard used for the certification of glulam is EN 14080 (2013). This standard provides shear and delamination tests as a means of verifying the bonding quality and bondline integrity of the product. Standardized Initial Type Testing is done in the form of delamination, where the blocks are subjected to extreme climatic conditions by undergoing a pressure-vacuum-dry cycle in an autoclave. The glueline integrity is determined by analysing the glueline for openings caused by the moisture cycles and recording them. Factory Production Controls require the performing of a shear test where the shear strength is used to determine glueline integrity (Betti et al., 2016 and Knorz et al., 2017).

In recent years, however, extensive work has been performed on the European CLT standard EN 16351 with the most recent publication in 2015 (EN 16351, 2015). The standard prescribes shear tests on face bonds (block shear tests) where load is applied parallel to one adherent and perpendicular to the other adherent. This layup includes rolling shear stress (perpendicular to grain) in one adherent and shear stress parallel to grain in the other one. Delamination testing is done to evaluate bond integrity with a very similar method to the one described in EN 14080 (2013), but it has been found that the delamination test method obtained from the EN 14080 (2013) standard is too severe for CLT. Where the delamination values exceed the allowable lengths allowed, or surface defects such as knots are present, EN 16351 (2015) then recommends the splitting of the gluelines to estimate WFP and use this as a means of determining bond integrity (Betti et al., 2016 and Knorz et al., 2017).

2.11.2. Shear and delamination tests

Steiger et al. (2010) concluded that the block shear test method is objective, easy to perform and provides easily readable results and should be used for determining quality of gluelines in glulam.

However, weaknesses to this method include positioning of the samples in the test equipment during loading and shear stress distribution which is not uniform. When this testing method is introduced to CLT, the occurrence of rolling shear is a further weakness to consider.

Delamination tests have also been extensively used in glulam. This test has the advantage of using the stresses in the wood created by swelling and shrinkage under moisture change to determine bond integrity. However, the measurement of delamination in specimens can be subjective and achieving accuracy difficult (Ohnesorge et al., 2010). When EN 16351 (2015) applied this delamination method to CLT, weaknesses in the method were encountered. Due to the large specimen size (100 x 100 mm), excessively high stresses formed by shrinkage and swelling are created, leading to increased delamination. This effects a high non-conformance rate for delamination results, prompting the subsequent process of splitting the glueline with hammer and chisel to determine WFP. This process of determining WFP is criticised as inaccurate and subjective by some researchers (Betti et al., 2016 and Knorz et al., 2017).

Because of the weaknesses described in the two methods prescribed in the standard for determining adhesive bonding quality, it is clear that neither test method on their own can definitively provide the necessary information to make a decision on bonding quality (Schmidt et al., 2010). For this reason, both shear and delamination testing procedures require WFP to be estimated. WFP is very effective as it clearly shows whether the failure occurs in the wood or adhesive, but is ineffective at communicating the failure behaviour (Betti et al., 2016 and Steiger

et al., 2014). However, the high ductility of the 1C-PUR adhesives may lead to increased bonding strength (shear strength) and decreased WFP (Xiao et al., 2007 and Lehringer and Gabriel, 2014). Pröller (2017) indicated that the use of WFP, for the assessment of bonding quality using 1C-PUR adhesives, should be considered questionable.

These two methods (delamination and shear), have only had limited studies performed on them to study their suitability for use in qualifying bond integrity in CLT. Most of the tests have been performed on softwoods, with some recent tests being performed on hardwoods, especially poplar.

Sikora et al. (2016) conducted both delamination and shear tests to determine the performance of PUR and PRF adhesives used to manufacture CLT panels from Sitka spruce. Hindman and Bouldin (2013) used resistance to shear by compression loading and resistance to delamination tests on CLT from southern pine lumber to compare the results with the values given in the product standards.

Yellow-poplar CLT was tested by performing resistance to shear by compression loading and resistance to delamination tests in conformance with ANSI/APA (2012), the standard for performance rated cross-laminated timber (Mohamadzadeh and Hindman, 2015). Liao et al. (2017) evaluated CLT manufactured using fast-grown small diameter *Eucalyptus* wood (*E. urophylla x E. grandis*). The effects of pressure, pressing time and adhesive spread rate on WFP and rate of delamination were determined using block shear tests and cyclic delamination tests.

Castro and Paganini (2003) determined the bonding reliability of mechanically graded laminations of poplar (*Populus* x euramericana, 'Neva' clone) and *Eucalyptus grandis* (*E. grandis*, '7', '329', '330' and '358' clones) by performing delamination and shear tests in the gluelines. Block shear tests and delamination tests were used to compare the bond integrity performance with standard CLT performance criteria in an optimized hybrid poplar CLT panel by Weidman (2015).

Luedtke et al. (2015) found that block shear tests were successful in accessing the quality of adhesive bonds in hardwoods. Shear strength values were found to be higher than softwood values due to the higher density of the hardwoods, but WFP was found to be slightly lower than the WFP values of softwoods. To allow the use of hardwood engineered products, which to date have been very limited, current softwood standards must be adapted to fulfil the requirements of the hardwood species being investigated (Luedtke et al., 2015).

The delamination tests proposed by the CLT standards have been found to be too severe, while the shear test values are comparatively low and have been found to be inconsistent due to the rolling shear element. The CLT standard allows the choice between delamination and shear tests within factory production control. However, this does not address the fact that these tests are not ideally suited for determining the bond integrity of CLT. Betti et al. (2016) and Knorz et al. (2017) proposed that further analysis and possibly even revision of the test methods should be considered.

Chapter 3: Materials and methods

3.1 Materials

3.1.1 Timber

The timber used in this study was obtained from Merensky Timber's *Eucalyptus* plantations located near Tzaneen in the Limpopo Province. The area is characterised by a sub-tropical climate with an average annual rainfall of around 1200 mm (Pröller, 2017).

Over 300 Eucalyptus grandis boards in the wet state (green) from twenty to twenty five year rotation plantations were supplied. All the boards that were supplied came from one of the three positions in and surrounding the pith. This is represented as either the P0 or P1 position in the heartwood of the log (Figure 3-1). The P0 boards had continuous pith, while the P1 boards were largely without pith although in some cases pith flowed in and out of the board. The boards all had similar dimensions of about 2400 x 114 x 38 mm (length x width x thickness) (Pröller, 2017).

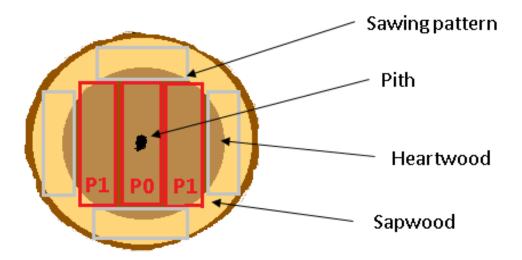


Figure 3-1: The sawing pattern used by Merensky Timber for the 20-25 year old *E. grandis* as well as the positions of the 3 boards per log supplied for the study. Heartwood and sapwood are also shown in dark and light (Pröller, 2017).

3.1.2 Adhesive

The glue used for this study was a one-component polyurethane adhesive. This adhesive, "LOCTITE HB S159 PURBOND", was manufactured and certified for structural load bearing applications by Henkel (ATG 2888, 2016). According to the specifications for "HB S159" provided by the manufacturer, the product has an assembly time of 15 minutes and a curing time of 45 minutes when applied on wood that has a moisture content (MC) between 8 and 18% at standard climatic conditions of 20°C and 65% relative humidity (RH). An adhesive spread rate of 120 to 160 g/m² is recommended for use at these standard conditions (Henkel, 2015 and Pröller, 2017).

3.2 Methods

3.2.1 Experimental design

The effect of the three factors, namely density (kg/m³), stress relief grooves and clamping pressure (MPa) on the performance of the CLT panels after conducting face-bonding quality

tests was investigated. The design of the experiment was such that each factor had an upper level and a lower level. The upper and lower levels for each of the three factors are displayed in Table 3-1.

Table 3-1: The 2 x 3 factorial design with three factors at two levels each.

Factors	Lower Level	Upper Level
Wood density (kg/m³)	<540	>540
Stress relief grooves	Y (Yes)	N (No)
Clamping pressure (MPa)	0.1	0.7

These three factors, along with the two levels per factor, led to the creation of a 3×2 factorial design for this experiment. A 3×2 factorial design has eight different permutations or possible combinations of factors. These eight combinations of different levels and factors are displayed as eight groups in Table 3-2.

Table 3-2: The eight sample groups with the three factors being tested and their upper and lower levels.

Group	1	2	3	4	5	6	7	8
Density (kg/m³)	<540	<540	<540	<540	>540	>540	>540	>540
Stress relief grooves	Y	Y	N	N	Y	Y	N	N
Clamping pressure (MPa)	0.1	0.7	0.1	0.7	0.1	0.7	0.1	0.7

3.2.2 CLT panel production

It is important to note that single layer edge-bonded panels were manufactured from wet (green) boards in this process. Face-bonding occurred after drying of the single layer panels.

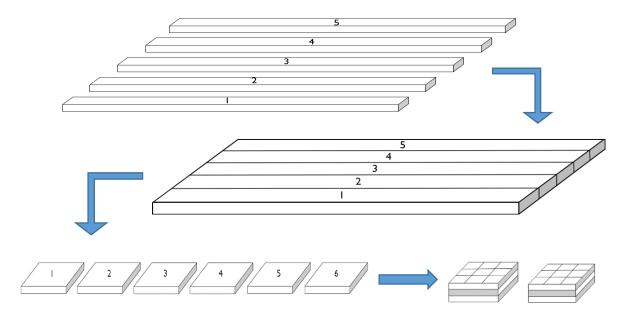


Figure 3-2: Production of 300 x 300 mm CLT panels from individual boards.

Primary Lumber selection

Each panel was made up of five boards selected at random from the material supplied by Merensky Timber. Boards that contained excessive crook, wane, end-splitting or checking were not selected to ensure that the raw material defect did not have an effect on the panel quality. Boards that contained significant amounts of bow "were arranged alternately within the panel with respect to their bow direction in order to even each other out when glued together" (Pröller, 2017).

Lumber planing and cutting to length

All the boards were cut to 2.4 m lengths in order to fit in the press and planed to 102 mm widths. The reason for planing was to ensure a smooth, clean surface for edge gluing and pressing which was performed within 24 hours of planing. Planing conditions the edges of the boards for better bonding by "activating" the wood surface and reducing oxidation (Yeh et al., 2013).

CLT layer formation

According to Brandner (2013), CLT layer formation can be formed in two different ways, namely the edge bonding of individual boards to limit gaps in individual lamellae and the formation of CLT panels from loose boards without first edge bonding in individual lamellae.

The advantage of edge bonding is the equalisation of lamellae and the reduction of gaps between boards as well as the reduction in pressure needed to clamp the CLT panel during manufacturing due to lamella surface uniformity. The disadvantage of edge bonding is the formation of surface checks and checks within layers due to swelling and shrinkage stresses within the CLT element (Brandner, 2013).

The single layer panels were obtained from a previous study by Pröller (2017). The panels were edge glued in the wet state (Figure 3-2) for the purpose of that study. According to Pröller (2017), the production of panels by edge gluing green *E. grandis* boards together took place as follows:

1. Four of the five boards that make up a panel were stacked edge up and glue was applied to all four edges simultaneously using the glue applicator ("OEST Ecopur HK" with "Facetac HAV10", see Figure 3-3) at a spread rate of approximately 180 g/m².

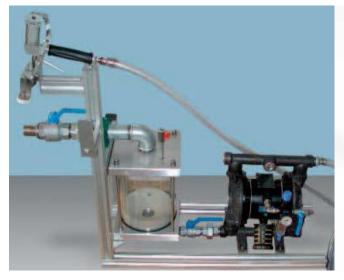




Figure 3-3: OEST Ecopur HK gluing system for 1C-PUR adhesives (left) and Facetac HAV10 application gun (right) (Oest 2016).

2. The four boards with adhesive, together with another side board without adhesive, were immediately placed in the edge laminating press (Figure 3-4) and clamped together, applying a pressure of approximately 0.75 MPa. Vertical pneumatic clamps provided force from the top to flatten the boards and ensure a uniform panel surface.

The edge laminating press was built specifically for the purpose of manufacturing panels for use in this project and the study by Pröller (2017). The press was a combination of manual tightening clamps using a torque wrench and button operated pneumatic clamps.



Figure 3-4: Edge laminating press fully loaded with 3 panels. Vertical clamps are applying pneumatic pressure to flatten the panel, while six clamps are applying 0.75 MPa of pressure for edge bonding.

- 3. Each panel was allowed to cure for 45 minutes under pressure before being removed from the press and stacked in four piles of 30 panels each for a total of 120 panels.
- 4. After air drying for three days, the panels were kiln dried in six-zone progressive kilns according to the standard conditions (approximately 24 days at a medium temperature drying schedule to a target MC of below 12%) employed by Merensky Timber for their *E. grandis* boards.

The entire edge laminating production process as well as kiln drying was performed at Merensky Timber in Tzaneen. The panels were stored for four weeks at dry conditions at Merensky Timber before being transported to Stellenbosch and then stored in similarly dry conditions at the Department for Forest and Wood Science at the University of Stellenbosch, South Africa.

Secondary lumber selection, planing and cutting to length

Secondary lumber selection took place in the form of selecting 20 edge laminated panels for the production of CLT. The panels were selected for their straightness, lack of checks, end splits and warp and based on density, with high and low density material being needed to test the density factor. The 20 selected panels were stored in a conditioning chamber ($65\% \pm 5\%$ RH, $20^{\circ} \pm 2^{\circ}$ C) for two weeks before CLT panel preparation. This was necessary to ensure a uniform moisture content of 12% in the specimens (Sikora et al., 2016).

The process of selecting for density was performed as follows:

- 1. The approximately 2400 x 500 x 38 mm edge laminated panels were weighed to get a general idea of density and 10 "heavy panels" with similar mass and 10 "light panels" with similar mass were selected.
- 2. Five density samples (one per board) were taken on each end of the selected panels to get the average density profile of each panel (Figure 3-5).

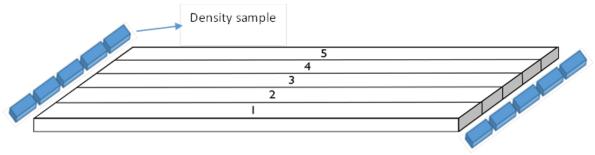


Figure 3-5: 2400 x 500 x 38 mm edge laminated panel showing the location of the 10 density samples taken to achieve a density profile across the width of the panel.

- 3. From the density results, three consecutive boards (1, 2, 3 or 3, 4, 5 or 2, 3, 4) per panel were selected that had a similar density distribution.
- 4. The 2400 x 500 x 38 mm panels were planed with a Paoloni SP 515 thicknesser down to 25 mm thickness to remove surface unevenness caused by drying defects. All face bonding surfaces must be planed within 24 hours of adhesive application and subsequent face bonding according to Weidman (2015). This prevents surface oxidation, dimensional instability and as a result improves bonding effectiveness (Yeh et al., 2013).
- 5. These panels were then ripped and cross cut into 1980 x 330 x 25 mm panels based on the density distribution in Step 3. The cross cutting process ensured that six 330 x 330 x 25 mm lamellae were cut from each of the panels.
- 6. These 120 lamellae were weighed to determine final density according to the calculation p = m / V (density equals mass divided by volume) and sorted into two groups, 60 high density (>540kg/m³) lamellae and 60 low density (<540kg/m³) lamellae.

CLT panel layup

Each of the sixty lamellae per density group was given a number and three lamellae were randomly assigned to each of the 20 CLT panels per density group. The position of each layer within the panel was also assigned with the two outer lamellae being in the major strength direction of the panel and the single middle lamella being orientated perpendicularly to the major strength direction (also known as the minor strength direction).

Stress relief grooves

Stress relief grooves were included in half of the high density lamellae and half of the low density lamellae. Grooves were cut in the direction of the grain in each lamella. The two outer lamellae in the major strength direction were cut with grooves on only the inward-facing contact surfaces of the lamella, while the single middle lamella was cut with grooves on both of its faces (Figure 3-6). Four grooves were cut across the face of the lamella, with a gap of 58 mm between each groove, while the thickness of the grooves was 4 mm and the depth of the grooves was

8mm. Both these values were within the maximum allowable limits of 4 mm for maximum groove width and 90% of the lamella thickness for groove depth as stipulated in EN 16351 (2015).

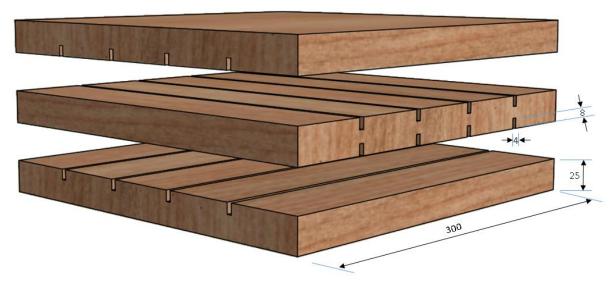


Figure 3-6: Top and bottom lamellae in the main strength direction with grooves on their inner faces and a middle lamella, which was orientated 90° to the bottom and top lamellae, with grooves on both faces.

Adhesive application

Each lamella was first wiped with a clean cloth to remove dust particles and any oiliness according to a method developed by Yeh et al. (2013). The adhesive was applied to the upward face of the middle and bottom lamella in each panel using a squeeze bottle with a small 5 mm nozzle. The application was done in the form of parallel lines/threads of adhesive approximately 40 mm from each other as well as a line all along the circumference of the panel approximately 10 mm from the edge (Figure 3-7). The adhesive amount was based on the spread rate recommended by the manufacturer of 120 - 160 g/m². To calculate the amount of adhesive needed to satisfy the spread rate, the adhesive was applied to a piece of cling wrap with an area (A) of 330 x 330 mm which had been pre weighed. The adhesive and cling wrap were then weighed and reweighed until the required mass (m) was achieved by trial and error. The required mass was calculated to be between 15 and 17 grams by transforming the formula:

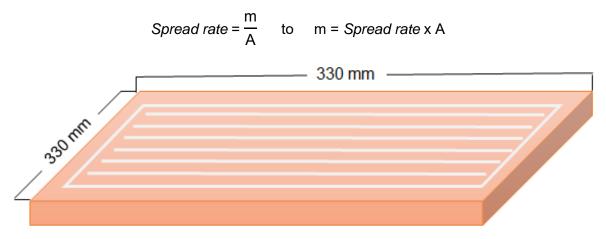


Figure 3-7: Adhesive application pattern.

Note: A more consistent surface coverage could possibly have been achieved by using a roller for the application of the adhesive. This should be considered for subsequent studies.

CLT pressing

A pneumatic press system was specially designed and built for use in this study focussing on a simple design and construction as well as ease of use. It is also a system widely used by large scale CLT producers in Europe and provides up to 1 MPa of pressure (Brandner, 2013).

The pneumatic press was adapted from the design used for a glulam press. It pushed compressed air into hoses to force two rigid steel plates towards each other where the CLT panels were inserted between the steel plates. This achieved the clamping of the CLT panels (Figure 3-8).



Figure 3-8: CLT press loaded with six CLT panels under pressure.

Half of the panels were pressed at 0.1 MPa, while the other half was pressed at 0.7 MPa. Both these values fall within the range of pressures chosen in previous studies on hard and softwoods where PUR adhesive was used (Sikora et al., 2016; Knorz et al., 2017 and Liao et al., 2017).

The panels were stacked in the press, as many as eight at a time fitted in the press, with the adhesive application time (assembly time) being the limiting factor in the number of panels able to be pressed at a time.

PUR is a swelling adhesive (foams and expands upon curing) and it was therefore important to adhere to the specification of a maximum of 0.3 mm for bondline thickness of PUR adhesives in EN 16351 (2015). To ensure conformance to this specification, the open/assembly time limit of 15 minutes was strictly adhered to. All panels were pressed for at least an hour which is more than the recommended 45 minutes of curing time for the adhesive. Both adhesive application and pressing were done at room temperature (approximately 25°C).

CLT finishing

The panels were stored at room temperature for two days. After two days, the 330 x 330 x 75 mm panels were edged on all four edges using a table saw. The edged panels (Figure 3-9), with dimensions $300 \times 300 \times 75$ mm, were the final CLT product before samples were to be cut for the shear and delamination tests.



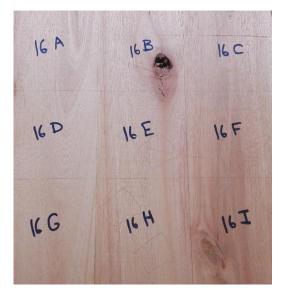


Figure 3-9: Finished CLT panel (300 x 300 x 75 mm) (left) and panel 16 of 40 with the positions marked (A - I) (right)

3.2.3 CLT test sample production

Four test procedures were conducted to determine the face bonding quality of the *E. grandis* CLT samples (Figure 3-10). The shear and delamination tests were the two methods prescribed by the CLT standards as the means of determining the face bonding quality of CLT. The delamination test according to Annex C of EN 16351 (2015) required a 100 x 100 mm sample ("a" in Figure 3-10) while the block shear test according to Annex D of EN 16351 (2015) required a 40 x 40 mm block ("b" in Figure 3-10). In addition to the two standard test procedures, two further test methods were trialled based on literature and theories from previous studies. The aim of these two tests was to provide different test procedures that addressed the shortcomings of the test procedures prescribed in the CLT standards and more specifically, for this study, EN 16351 (2015). 40 x 40 mm samples, angled at 45° to the grain direction ("c" in Figure 3-10), were required for the shear tests to limit rolling shear and 70 x 70 mm samples, angled at 45° to the grain direction ("d" in Figure 3-10), were required to perform the combined delamination and shear tests according to the proposed test procedure in Betti et al. (2016).

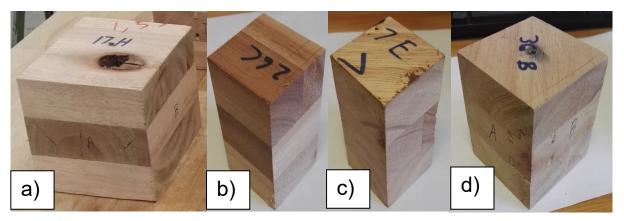


Figure 3-10: Four different sample types for the four proposed testing methods: a) $100 \times 100 \text{ mm}$ samples for delamination testing, b) $40 \times 40 \text{ mm}$ samples for shear testing, c) $40 \times 40 \text{ mm}$ samples at 45° to the grain direction for shear testing and d) $70 \times 70 \text{ mm}$ samples at 45° to the grain direction for delamination and shear testing.

Panel breakdown

The 40 CLT panels, consisting of 5 panels for each of the 8 groups, were cut into samples for shear and delamination testing procedures. These 5 panels per group were cut into samples according to the dimensions specified by their respective tests (Figure 3-11), resulting in the following sample numbers per group:

- 15 (100x100x75mm) samples for delamination testing per group = 120 total
- 20 (40x40x75mm) samples for shear testing per group = 160 total
- 20 (40x40x75mm) samples at 45° to the grain direction for shear testing per group = 160 total
- 15 (70x70x75mm) samples at 45° to the grain direction for delamination and shear testing per group = 120 total

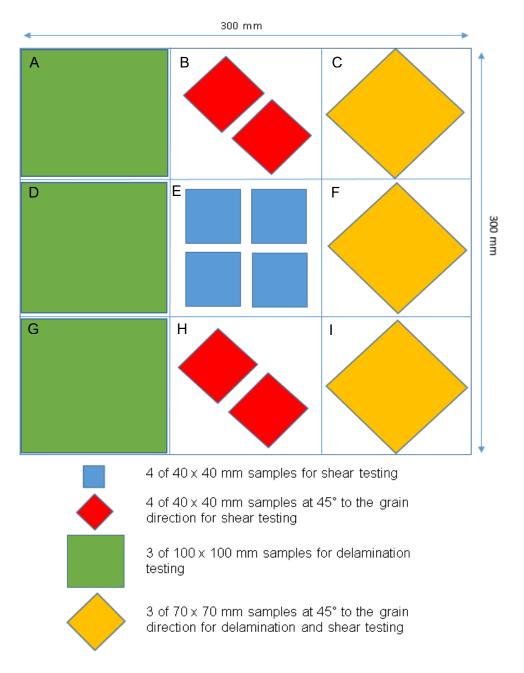


Figure 3-11: 300×300 mm CLT panel and the positions and sizes of the different samples to be cut from the panel. The letters (A- I) indicate the respective positions within the panel as a means of changing the sample position from panel to panel and keeping track of the sample's origin.

Samples were labelled by their panel number and the location (A - I) in the panel from which they were taken. For example, the delamination block taken from the top left corner of CLT panel sixteen would be labelled "16 A" (Figure 3-9).

The positions of the samples within the panel were changed for each panel so as to ensure that the samples for a certain test did not all originate in the same position in their respective panels. This change was done by moving each of the blocks (A - I) in Figure 3-11 one position on (to the right). For example, the 100 x 100 mm sample at position A will move to position B in the next panel and the 70 x 70 mm block at position I will move to position A in the next panel.

The samples were stored in a conditioning chamber (65% \pm 5% RH, 20° \pm 2°C) for 2 weeks, as required by EN 16351 (2015), before any testing took place.

3.2.4 CLT sample testing

Test A: Delamination of 100 x 100 mm samples

The delamination test is used to assess the moisture durability and quality of the bondline. A quadratic cut out is impregnated with water and subsequently dried to determine whether the bondline is able to resist the swelling and shrinkage stresses present in the wood as a result of the moisture gradient (Yeh et al., 2013).

The delamination testing process was performed at CNR - IVALSA (Trees and Timber Institute), Firenze, Italy who have extensive experience with these types of tests. The delamination test of gluelines between layers was conducted according to Annex C of EN 16351 (2015). The 100 x 100 mm samples were removed from the conditioning room, weighed to the nearest 5g and the exact dimensions of all the sides of the block to the nearest mm determined using a Vernier calliper. The sample size conformed to the EN 16351 (2015) standard requirement of "approximately quadratic cut outs having minimum lateral lengths of (100 \pm 5) mm and a top view area of at least 10 000 mm²". The samples were then placed in a pressure vessel (autoclave), submerged in water (15 \pm 5) °C with the end grain surfaces of the blocks exposed to water and weighted down to ensure the blocks remained submerged. A vacuum of (75 \pm 5) kPa was drawn for 30 minutes followed by a pressure of 550 kPa for 2 hours.

The samples were removed from the pressure vessel and immediately placed in the drying oven at a temperature of 70 ± 5 °C, air speed of 2 - 3 m/s and a relative humidity of 8 - 10%. They were placed in the oven and spaced 50 mm apart with the end-grain surfaces parallel to the stream of air. The samples were left in the oven until their mass returned to within 100 - 110 % of the original mass and the drying time was recorded.

Samples were removed from the drying oven when they had reached their required mass and were visually inspected within the first hour.

A 10x magnifying glass was used to inspect the length of both the total and maximum delamination in the bondlines and the values were recorded as percentages. Delamination was considered to be failure in the adhesive layer or within the first two cell layers of the wood next to the glueline.

Openings in the glueline that were present before the delamination test, delamination resulting from wood defects (resin pockets and knots), and hidden defects that were only visible after splitting of the gluelines, were not considered to be delamination instances.

The total delamination ($Delam_{tot}$) is the total delamination length (in mm) across both gluelines divided by the sum of the perimeter of both gluelines in the sample (in mm) as seen in Eq. (1).

$$Delam_{tot} = 100 \frac{l_{tot,delam}}{l_{tot,glue\ line}}$$
 (%)

Where:

 $I_{tot. delam}$ is the total delamination length (in mm),

 $I_{tot, glue line}$ is the sum of the perimeters of all glue lines in a delamination specimen (in mm).

The maximum delamination (Delam_{max}) is the singular maximum delamination length (in mm) divided by the perimeter of a single glueline as seen in Eq. (2).

$$Delam_{max} = 100 \frac{l_{max,delam}}{l_{glue\,line}}$$
 (%)

Where:

I max. delam is the maximum delamination length (in mm),

I glue line is the perimeter of one glue line in a delamination specimen (in mm).

After assessing delamination, wood failure percentage (WFP) was determined by splitting both gluelines using a hammer and chisel and visually inspecting the amount of wood failure versus adhesive failure for each glueline (Figure 3-12). WFP was determined to the nearest 5% for each glueline and then averaged to determine average WFP for the sample. Wood defect areas were subtracted from the total bonding surface area and not considered for WFP.



Figure 3-12: Samples in autoclave, weighed down and about to be submerged in water (left) and hammer and chisel used to split the glueline to estimate WFP (right).

Test B: Shear strength of 40 x 40 mm samples

The block shear test effectively tests the strength of the bond between layers as it isolates the glueline for exposure to shear (Weidman, 2015).

The shear test to determine bondline strength was conducted according to Annex D of EN 16351 (2015).

The 40 x 40 mm samples were removed from the conditioning room and the exact dimensions of all the sides of the block to the nearest 0.5 mm determined using a Vernier calliper. The sample size conformed to the EN 16351 standard requirement of "a square test bar with a shear area of 40 mm x 40 mm". The samples were weighed prior to testing, oven dried for 24 hours after testing and weighed again to determine the MC of the samples. The average MC was 9.5% with a standard deviation of 0.34%.

The samples were placed in the shearing tool so that the vertical load that is applied is in the direction of the grain for the timber on one side of the glueline and perpendicular to the grain for the timber on the other side of the glueline (Figure 3-13). The shearing tool has a self-aligning cylindrical bearing (Figure 3-13) to ensure uniform stress distribution in the width of the CLT element. The samples were positioned in the shearing tool so that the distance between the vertically applied load and the glueline was always 1 mm or less. The vertical load was applied by an INSTRON load cell, which had been calibrated prior to testing, at a constant rate of 0.7 mm/minute to ensure that failure occurred after roughly 20 seconds.

The shear test was performed twice per sample to get the pure shear strength per glueline. In order to determine the shear strength (fv) (in N/mm²), pure shear strength / ultimate shear strength ($F\mu$) (in N) was divided by sheared area (A) (in mm²) as seen in Eq. (3).

$$f_{v} = \frac{F_{u}}{A} \tag{3}$$

After each glueline was sheared, the wood failure was visually estimated and expressed as a percentage (\pm 5%) of the sheared area.

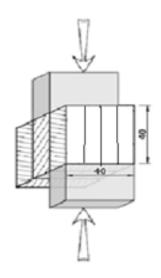




Figure 3-13: 40 x 40 sample showing shear test method (left) and shearing tool with self-aligning cylindrical bearing (right).

Test C: Shear strength of 40 x 40 mm samples at 45° to the grain direction

In the 40 x 40 mm samples for Test C, the grain direction of every panel lamella forms an angle of 45° with respect to the sides of the panel (Figure 3-14) and an angle of 90° with respect to the previous lamella. The average MC was 9.4° with a standard deviation of 0.41° .

The testing procedure is exactly the same as the one described in Test B.

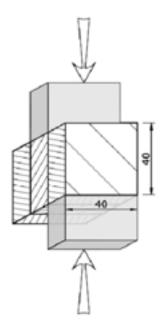


Figure 3-14: 40 x 40 sample showing shear test method at 45° to the grain direction.

Test D: Delamination and shear strength of 70 x 70 mm samples at 45° to the grain direction

The delamination process was conducted according to Annex C of EN 16351 (2015) and as described in Test A, with the size of the samples being 70×70 mm instead of 100×100 mm and the grain direction of every panel lamella forming an angle of 45° with respect to the sides of the panel, instead of an angle of 0 or 90° with respect to the sides of the panel. The average MC was 10% with a standard deviation of 0.48%.

After determining total and maximum delamination, however, the samples were not split to determine WFP but were subjected to a shear test (see Figure 3-15) and subsequent WFP determination (Figure 3-16) as described in Test B.

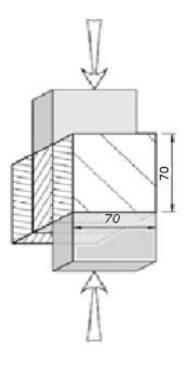


Figure 3-15: 70 x 70 sample showing shear test method at 45° to the grain direction.



Figure 3-16: WFP (approximately 60%) of a 70 x70 sample. Blue indicates wood failure.

3.2.5 Statistical analysis

The Statistica 13 software was used to conduct statistical analysis on the results of the tests. Three-way factorial analyses of variances (ANOVA) were performed with the input factors Density (L, H), Pressure (L, H) and Grooves (Y, N), to show the effect of the variables on the response variables total delamination (*Dtot*), shear strength (*fv*) and WFP as well as their interactions with each other. Highest order significant interactions were displayed in graph format to represent the findings.

The residuals were checked for normality, using the Shapiro-Wilk normality test, while at each level of interaction between factors and for main effects, Levene tests were done to check for non-homogeneity of variances involved. Data that did not conform to the normality requirement was transformed toward normality by performing Box-Cox transformations on the response variables and reanalysed to determine if the transformed data provided alternative conclusions to the original data. Where identical significances were found between the non-normal data and Box-Cox transformed data, it was deemed possible to use the non-normal data as the transformed data backed up the conclusions of the non-normal data. Where highly significant non-homogeneity was discovered, Games-Howell multiple comparisons were done to detect differences among interaction or main effect means instead of the Least Significant Difference (LSD) multiple comparisons.

- A pass / fail evaluation was conducted for both the delamination and shear test results (Test A and B) according to the bonding strength of glue lines between layers requirements in EN 16351 (2015). These test methods are known as "Pass Delam" and "Pass Shear" with the shear test given as the reference test method.
- The results from method B and C were compared to determine the effect of grain angle to load direction on rolling shear.
- The relationship between the mean delamination and shear results for Test D were analysed to determine the method's ability to determine bondline quality. This is a qualitative test that gives the actual strength after delamination testing as a better representation of what might happen in a real life weathering and load bearing situation.
- Mean comparisons of the results of the 4 different testing methods was done to determine relationships between them, and highlight differing results if any, between the different methods.
- The effect of density, pressure and grooves on the face bonding performance of *Eucalyptus grandis* cross-laminated timber (CLT) was determined in order to propose the best combination of factors (group 1 − 8) for use in manufacturing *E. grandis* CLT. For each of the graphs, Y is yes, N is no, L is low and H is high.

28

Chapter 4: Results and discussion

Table 4-1: Summary of the results for all four test methods for each of the groups (1 - 8). Note: Rankings from 1 - 8 are displayed encircled in red for each of the groups (1 - 8). The rankings were done to display the performance of each group (1 being best and 8 being worst) in order to compare the results within single test methods and between different test methods.

Group		1	2	3	4	5	6	7	8	All
oles	Density (kg/m³)	<540	<540	<540	<540	>540	>540	>540	>540	
Variat	Stress relief grooves	Y	Y	N	N	Y	Y	N	N	
Input Variables	Clamping pressure (MPa)	0.1	0.7	0.1	0.7	0.1	0.7	0.1	0.7	
Mean de	ensity (kg/m³)	522.1	517.8	525	525	582.3	568.9	584.8	568.6	549.3
Density	Stdev (kg/m³)	4.32	13.24	9.53	13.66	13.63	13.34	19.29	8.16	
Test A:	100 x 100 mm									
n		15	15	15	15	15	15	15	15	120
Pass De	lam	9	14	9	15	1	8	8	15	79
Mean D _t	ot (%)	19.64	14.72	6 26.34	9.67	8 42.76	28.56	20.40	10.57	21.58
D _{tot} (%) {	5 th percentile	41.79	34.9	8 65.19	28.99	57.17	6 52.38	38.71	24.75	
WFP _{mean}	(%)	70.17	80.17	7 58.17	81.33	39.67	61.00	5 65.83	1 82.17	67.31
WFP _{mean}	5 th percentile	48.76	64.81	6.03	67.3	7.97	6 23.44	38.28	64.77	
Test B:	40 x 40 mm									
n		20	20	20	20	20	20	20	20	160
Pass Sh	ear	20	20	20	20	20	20	20	20	160
Mean sh	ear strength (fv)	3.78	3.65	3.49	4.62	3.76	3.81	4.89	4.96	4.12
Characte	eristic shear strength (fv,k)	2.13	2.08	1.57	2.82	1.30	1.90	2.87	3.24	
WFP _{mean}	(%)	65.5	72.75	40.5	74.38	49.5	61.25	62.75	79.88	63.31
WFP _{mean}	,5 th percentile	45.51	3 52.81	8	2 54.88	7	6 32.54	5 39.28	57.07	00.01

Test C: 40 x 40 mm 45° to grain									
n	20	20	20	20	20	20	20	20	160
Mean shear strength (fv)	7	4	8	3	6	5	2	1	
Wedit Stieright (W)	5.09	5.36	5.08	6.08	5.1	5.25	6.23	6.79	5.62
Characteristic shear strength (fv,k)	6	4	7	2	8	5	3	1	
The state of the s	2.85	3.32	2.43	4.16	2.20	3.10	3.87	4.90	
WFP _{mean} (%)	6	3	7	1	8	4	(5)	2	
· · ·	59	74.5	46.25	85.25	40.75	70.25	69.88	76.13	65.25
WFP _{mean} 5 th percentile	6	(3)	8	(1)	(7)	4	(5)	2	
	29.52	44.31	0	70.94	5.74	42.22	39.28	59.57	
Test D: 70 x 70 mm 45° to grain									
n	15	15	15	15	15	15	15	15	120
Mean D _{tot} (%)	7	4	(5)	1	8	6	3	2	
	30.17	22.97	24.65	12.11	58.45	30.14	20.99	14.28	26.72
D _{tot} (%) 5 th percentile	6	3	7	2	8	5	4	1	
	55.97	34.31	59.46	30.49	77.03	49.64	39.63	27.96	
Mean shear strength (fv)	7	(5)	4	2	8	6	3	(1)	
	1.54	1.65	1.9	2.18	0.67	1.58	2.03	2.33	1.74
Characteristic shear strength (fv,k)	7	(5)	4	1	8	6	3	2	
	0.13	0.44	0.72	0.89	0.04	0.31	0.72	0.87	
WFP _{mean} (%)	(5)	(3)	7	(1)	8	6	4	2	
	40.67	48.83	37.33	65.67	11	39.67	45.17	63.33	43.96
WFP _{mean} 5 th percentile	6	3	8	(1)	7	(5)	4	2	
	0.65	26.43	0.01	48.65	0.1	6.97	25.49	31.41	

Table 4-2: Ranking differences between different tests and groups. Rankings used were for mean Dtot (Test A), mean shear strength (Test B), mean shear strength (Test C) and mean Dtot and mean shear strength (Test D).

	Group	Sum							
	1	2	3	4	5	6	7	8	
Tests A-B	1	4	2	2	2	3	3	1	18
Tests A-C	3	1	2	2	2	2	3	1	16
Tests A-D	3	2	2	1	0	1	2	1	12
Tests B-C	2	3	0	0	0	1	0	0	6
Tests B-D	2	2	4	1	2	2	1	0	14
Tests C-D	0	1	4	1	2	1	1	0	10

4.1 Face bonding quality of *E. grandis* CLT according to EN 16351 (2015)

According to EN 16351 (2015) the face bonding quality of CLT is acceptable in terms of factory production control if less than 5 out of 100 specimens fail the shear test (Test B) results. According to that, all the groups would have acceptable face bonding quality since no tests failed the shear requirements (Table 4-1). There are no requirements listed in EN 16351 (2015) for factory production control in terms of delamination results (Test A) – probably since the shear test (Test B) is defined as the reference method.

For two groups (groups 4 and 8) all the specimens passed the delamination test. This indicated very good bonding quality since this test was acknowledged as very severe (Betti et al., 2016 and Knorz et al., 2017). Groups 4 (low density) and 8 (high density) were those which did not have stress relief grooves and were clamped at a high pressure of 0.7 MPa. According to these results it is possible, with the right processing variables, to obtain very good face bonding quality when using a 1C-PUR adhesive to manufacture *Eucalyptus grandis* CLT.

4.2 Comparison of different test methods

4.2.1 Test A: 100 x 100 mm

A pass / fail evaluation was conducted for Test A: delamination of 100 x 100 mm samples according to the requirements in Annex C of EN 16351 (2015) and declared as test method "Pass Delam". Bond strength was considered sufficient if:

- 1. Maximum delamination (*Dmax*) length did not exceed 40% of the total length of each individual glue line.
- 2. Total delamination (*Dtot*) length did not exceed 10% of the sum of both glue lines.

Where the maximum delamination length or the total delamination length exceeded the limits given above or if the delamination lengths could not be estimated due to inadequate surface quality, each glue line was split and the sample only passed if:

3. Minimum WFP (*WFPmin*) of each split glued area was not less than 50%, while minimum WFP of the sum of both split glued areas (*WFPmean*) was not less than 70%.

A detailed inspection of individual results was performed. In all the results, all the failures according to the maximum delamination requirement also failed according to the total delamination requirement with more failures as a result of total delamination. This indicated that total delamination was a more severe test and was the more critical criterion for determining delamination. For this reason, only total delamination results were displayed in Table 4-1. For the relevance of the analysis in this study, total delamination is a representation of all the samples that failed the delamination requirement.

The same was observed for the WFP. All the WFPmin failures also failed according to the WFPmean requirement. This indicated that WFPmean was the more severe test and for this reason WFPmean was used as the criterion for determining WFP. For this study, where WFP is used it is a reference to the WFPmean as this value represents WFPmin and WFPmean.

It was, therefore, deemed unnecessary to analyse maximum delamination and *WFPmin* further to avoid duplication of results.

The number of samples that failed the delamination test was extremely high. This could be due to the fact that 1C-PUR adhesive is relatively untested on hardwoods and especially *Eucalyptus* and it is well documented that the hydrogen bonds between the wood and adhesive are susceptible to disruption under water immersion (Clauß, 2011). The high swelling and

shrinkage stresses that occur in hardwoods, especially *Eucalyptus*, under water immersion may cause the high delamination values, as found for hardwood bonds in glulam (Knorz et al., 2014). In addition, the cross-laminated property of CLT, where there is highly different shrinkage and swelling behaviour between consecutive layers, results in even higher stresses developed in the panel than in glulam where the fibre direction is uniform. This was confirmed by Betti et al. (2016) who found the delamination test to be extremely severe for CLT. This was also reported in findings by Sikora et al. (2016), with delamination failure for all their specimens, and Knorz et al. (2017) who reported delamination failure for 46% of their specimens. Glulam forms the basis for adhesive bonding suitability tests, which possibly explains the high number of samples failing the pass/fail test.

The question of test method suitability needs to be addressed. The delamination test method was originally designed to determine the bond line durability of glulam exposed to outdoor conditions. It was adapted for use in CLT as it was found to be extremely successful in spruce glulam assessment. However, CLT is mostly used in indoor applications with limited exposure to direct moisture. According to Knorz et al. (2017) there is no scientific evidence for the adoption of this method for CLT and the CLT and glulam reaction to moisture exposure are completely different as already stated above. It can, therefore, be concluded that delamination test specifications need to be adjusted or alternative test methods developed / employed to better evaluate CLT bond durability, which is of course one of the objectives of this study.

- Group 1: Five samples passed based on total delamination fulfilling the requirements. The 10 samples that failed were split to determine WFP. Four of these samples passed based on WFPmean fulfilling the requirements bringing the total to 9 samples that passed.
- <u>Group 2</u>: Eight samples passed based on total delamination fulfilling the requirements. The seven samples that failed were split to determine WFP. Six of these samples passed based on WFPmean fulfilling the requirements bringing the total to 14 samples that passed.
- Group 3: Five samples passed based on total delamination fulfilling the requirements. The 10 samples that failed were split to determine WFP. Four of these samples passed based on WFPmean fulfilling the requirements. Two whole panels 29 and 38 (6 samples) failed completely bringing the total to 9 samples that passed.
- <u>Group 4</u>: 11 samples passed based on total delamination fulfilling the requirements. The four samples that failed were split to determine WFP. All four of these samples passed based on WFPmean fulfilling the requirements bringing the total to 15 samples that passed.
- <u>Group 5</u>: Zero samples passed based on total delamination fulfilling the requirements. The 15 samples that failed were split to determine WFP. One of these samples passed based on WFPmean fulfilling the requirements.
- <u>Group 6</u>: Four samples passed based on total delamination fulfilling the requirements. The 11 samples that failed were split to determine WFP. Four of these samples passed based on WFPmean fulfilling the requirements bringing the total to 8 samples that passed.
- Group 7: Five samples passed based on total delamination fulfilling the requirements. The 10 samples that failed were split to determine WFP. Three of these samples passed based on WFPmean fulfilling the requirements bringing the total to 8 samples that passed.
- <u>Group 8</u>: Nine samples passed based on total delamination fulfilling the requirements. The six samples that failed were split to determine WFP. All Six of these samples passed based on WFPmean fulfilling the requirements bringing the total to 15 samples that passed.
- Group 5 clearly showed the worst performance for delamination testing. The results were drastically lower than any of the other groups, possibly pointing to manufacturing error (such as

low glue spread rate or poor pressing) rather than the effect of factors. Without the skewed effect of group 5, the rest of the groups had more samples that passed than failed. However, the high failure rate of 34% supports the theory that the delamination test might be too severe.

Three of the four groups that ranked best for mean total delamination (4, 8 and 2) were pressed at high pressure. Group 4 and 8 both had no stress relief grooves. It was expected that a higher pressing pressure would lead to significantly better results. However, the behaviour of group 6 was surprising, as it performed far worse than even some of the groups pressed at low pressure. These results seem to indicate the trend that the combination of high pressure without grooves was the most important for creating a good quality, durable bond. The effect of density was less visible.

Groups (4, 8 and 2) also performed the best in terms of WFP. Groups 5, 6 and 3 were the lowest ranked in terms of WFP (and mean total delamination). This seems to indicate that WFP supports the results determined by total delamination as the rankings corresponded in most cases.

The 100% conformance rate (Pass Delam) for group 4 and group 8 showed that despite the severity of the delamination test, these *E. grandis* CLT specimens, bonded at 0.7 MPa, without grooves and irrespective of density were good enough to pass.

The 5th percentile values for total delamination and WFP were determined. The rankings followed very similar trends to their respective mean values. The largest difference in ranking was 2 (WFP in group 8) where the WFP 5th percentile is ranked 3rd and the WFP mean is ranked 1st. However upon closer inspection it is seen that the difference between 1st and 3rd is negligible, which explains the difference in rankings. The 5th percentile values for group 3 tell their own story, with a 65.19% total delamination 5th percentile value (ranked 8th) far higher than the 26.34% total delamination mean value (ranked 6th). This seems to indicate that one or two samples had extremely poor bond quality, dragging the mean down while the rest of the samples in the group had relatively good bond quality. For group 5, the mean and 5th percentile values are quite close indicating that multiple samples had poor bond quality as can be seen by the 14 failures in group 5.

4.2.2 Test B: 40 x 40 mm

A pass / fail evaluation was conducted for shear according to the requirements in Annex D of EN 16351 (2015). The requirements for "Pass Shear" are:

- 1. The characteristic shear strength (fv,k) derived from tests is fv, $k \ge 1,25$ N/mm² and
- 2. The shear strength (fv) of each glue line must be at least 1 N/mm².

As displayed in Table 4-1, the "Pass Shear" evaluation showed that the requirements for shear strength were always met (all 160 samples passed), while a value of 2.04 N/mm² was calculated for characteristic shear strength (fv,k) according to the requirements in EN 14358 (2006), fulfilling the fv, $k \ge 1,25$ N/mm² requirement. This indicates that all the samples fulfilled the requirements for the "Pass Shear" test.

The characteristic shear strength (fv,k) was calculated for each individual group (Table 4-1) with all groups fulfilling the requirement of fv, $k \ge 1,25$ N/mm². Group 3 and group 5 showed the lowest fv,k value by some margin, indicating that one or more panels in these groups displayed poor strength characteristics. Upon closer investigation, it was found that three of the four samples from panel 29 (group 3) had shear strength values in the 5th percentile (fv,k = 1.52 N/mm²), while all 4 samples (fv,k = 0.9 N/mm²) in panel 39 (group 5) were below the required characteristic shear strength value. This could indicate manufacturing error or the presence of defects in panels 29 and 39. It was, however, deemed necessary to analyse the other test

methods to determine the similarities in results between the samples from the corresponding panels, before a judgement on panel properties was made.

Group 4 and group 8 once again were among the top three performers in terms of shear strength. This indicates good consistency between the two methods, Test A and Test B. However, group 7 had the second best mean shear strength results, but only the fifth best mean total delamination results.

Annex D EN 16351 (2015) requires that WFP of the sheared area be estimated to the nearest 5% for each glueline after undergoing the shearing test. However, no requirements are mentioned for WFP, and for this reason, WFP cannot be used to determine the pass / fail rate, but only to support / oppose the findings from the shear tests.

When the WFP results were compared to the corresponding shear results, there were sometimes discrepancies in results (i.e. group 7 and group 2), indicating that WFP estimation after shear testing is ineffectual and unnecessary. It is theorised that, for this reason, no pass / fail requirements were stipulated for WFP in Annex D of EN 16351 (2015).

The EN 16351 (2015) standard allows the option to choose between the delamination and shear test. However, this is scientifically questionable as it is well documented that there are differences between the delamination and shear test results (Betti et al., 2016). The standard (EN 16351 (2015)), therefore, states that the shear test is the reference test method for evaluating the bonding strength of glue lines between layers for CLT. However, the two tests essentially test different bond properties:

- The delamination test determines the durability of the bond after moisture gradients were introduced in a sample.
- The shear test determines the bond strength at testing and is an indication of the pure strength of the bondline before any weathering takes place.

Previous studies by Ohnesorge et al. (2010), Steiger et al. (2014) and Knorz et al. (2014) found that the shear test requirements are not strict enough to effectively evaluate bondline quality and samples will only fail in severe bonding failures. No pre-treatment is needed to perform the shear tests. Only samples with severe bonding deficiencies will fail the shear test requirements. This indicates that the shear test requirements possibly need to be revised.

The delamination test (Test A) and shear test (Test B) showed some similarity in terms of group rankings, but also important differences. Group 2, for instance, was the 7th ranked in terms of shear strength results, but 3rd ranked for the delamination test in terms of mean Dtot. In total, Tests A and B showed the largest combined difference in rankings (18) of all the tests (Table 4-2). This is not unusual since the two tests essentially measure different aspects of bonding quality. The EN16351 (2015) standard refers to the shear test as the "reference" method, probably indicating that if shear test results pass that the product is acceptable even though delamination test results do not. The comparison of our results shows that this can be problematic since high shear test results do not necessarily equate to durable bonds. It will be preferable if both the shear strength and durability aspects of bonding are included in bond evaluation of CLT, especially as CLT might be exposed to moisture conditions, e.g. in the case of water leaks inside a building.

4.2.3 Test C: 40 x 40 mm 45° to grain

It has been well documented that the shear strength achieved from block shear tests, conducted according to Annex D of EN 16351 (2015), is not an accurate representation of "true shear" due to possible presence of rolling shear (Blass and Gorlacher, 2000; Kim et al., 2013; Zhou, 2014 and Buck et al., 2016). The rolling shear failure can be seen as wood failure in the layers

orientated perpendicularly to the main grain direction. In the case of three layered CLT, as with the samples used in this study, only wood failure in the middle layer can be characterised as rolling shear failure.

The mean shear strength (fv) values for the 40x40 mm 45° to grain samples were, as expected, far higher than the shear strength (fv) values of Test B. The mean shear strength (fv) value of Test C (5.62 N/mm²) was 31% higher than the mean shear strength (fv) value of the 40 x 40 mm samples of Test B (Table 4-1). These higher values were likely related to the sample configuration having a grain angle of 45° to the side of the sample and with the load having been applied 45° to the grain direction for all the layers. As there were no layers perpendicular to the load direction, it is likely that this test configuration would have omitted most of the rolling shear element.

The WFP of 65.25% was only marginally higher than the 63.31% of Test B, which seems to indicate that despite the shear values being far higher due to the absence of rolling shear, WFP is not dependent on the presence or absence of rolling shear for its results.

As was done for Test B, a 5^{th} percentile analysis was performed on each group (1-8). The results indicated that group 3 and group 5 had characteristic shear strength values far below the fv, $k = 3.12 \text{ N/mm}^2$ for the entire sample set. Upon closer analysis, it was found that panel 29 from group 3 and panel 39 from group 5 accounted for most of the samples with shear strength values below the 5^{th} percentile value. These findings support the findings from Test B.

Highly similar rankings were found for both Test B and Test C, but the overall picture of groups (4, 7 and 8) having the highest shear strength values remains consistent. Tests B and C showed the most ranking similarity of all tests with a combined ranking difference of 6 (Table 4-2). That is not surprising since the two tests only differ in terms of grain orientation to load direction. However, the lowest ranked groups for mean shear in Test C (3, 1 and 5) were different to that from Test B (3, 2 and 5). The reasons for the difference are not clear from these results. In fact, the opposite reaction was expected, where group 2 (high pressure) should have displayed better results than group 1 (low pressure) for Test B as rolling shear does not have that much of an effect on shear strength at high pressure.

The lowest ranked groups for WFP in Test C (5, 3 and 1) were, as for shear strength, different to that from Test B (3, 5 and 6). The exact reasons for these results are not known.

4.2.4 Test D: 70 x 70 mm 45° to grain

In an attempt to test both the shear strength and the durability of bonds while minimising the possible effect of rolling shear, a new test method was developed (Test D). This method incorporates bond strength determination after pre-treatment or "weathering" which also tests bond durability and tests at a 45° angle to the grain. The aim of this test was to verify its applicability as an effective evaluation of bonding quality in CLT panels, with the future goal of replacing both shear and delamination tests currently provided in EN 16351 (2015).

From studies conducted on test methods similar to Test D, Betti et al. (2016) concluded that side lengths of closer to 75 mm were more appropriate for samples to be used in the delamination test, while shear tests coupled with a pre-treatment (in this case vacuum, impregnation and drying cycles) were feasible, but with larger sample sizes than the $40 \times 40 \text{ mm}$ samples used. For this reason, the $70 \times 70 \text{ mm}$ size was selected for samples.

In contrast to previous findings in Betti et al. (2016) and Knorz et al. (2017) where the larger sample sizes experienced greater amounts of delamination, the delamination percentages of the smaller 70 x 70 mm samples (Table 4-1) were much higher than the larger (100 x 100 mm) samples (Table 4-1). It is generally understood that larger samples induce greater stresses in the bondline during the vacuum, impregnation and drying cycles, leading to greater levels of

delamination. However, a possible explanation to the unexpected poor performance of the smaller samples could be due to the smaller sample size (70 x 70 mm) allowing complete saturation with water, leading to greater swelling and shrinkage and resulting in more delamination.

The mean shear strength results resemble, very closely, the ranking pattern found in the delamination results – all rankings were the same or within one ranking difference. This likely indicates that shear strength was very dependent on the delamination results. This is important as the bond durability will affect the bond strength after a number of years. For this reason, it seems necessary to have tests for both bond durability and bond strength or a test that combines the two. Also, if the shear strength (after delamination) is considered the only result to evaluate, it provides a very objective and quantifiable measure for evaluation.

Test D, which combined the effect of moisture degradation with a shear test, cannot be directly compared with any of the other 3 test methods. Comparing the rankings of this test, one can observe that the mean shear strength rank is in all cases close to the results obtained by Test C, except for the case of group 3 (group 3 ranked 4^{th} in Test D and 8^{th} in Test C). The difference could possibly be accounted for by the delamination behaviour (group 3 ranked 5^{th} for D_{tot}) strongly influencing the shear strength results. Ranking results for mean shear strength of Test D are also somewhat similar to Test A except for the case of group 1 (group 1 ranked 7^{th} in mean shear strength in Test D and 4^{th} in mean D_{tot} in Test A). One can see, however, that the mean D_{tot} of group 1 in Test D was also ranked 7^{th} which indicated that the difference in delamination behaviour between the groups was responsible for the lower mean shear strength ranking.

In summary, one could observe that in most cases all four tests gave relatively similar, comparative results in terms of rankings of the groups even though different aspects of bonding quality were tested. In a few cases there were larger differences. Although it was not always possible to determine the exact cause of these differences, it is possible to make a few general observations. Firstly, the combined differences between Tests A and B (18) and Tests A and C (16) were the largest between all tests which could be expected since Test A was essentially a bond durability test and Tests B and C shear tests (Table 4-2). Similarly, Tests B and C gave the most similar ranking results since both of these were shear tests without any component of degradation. Test D's ranking differences, compared to the other tests, were between the two extremes described above, since it is a combination of these tests. This was of course what was expected. Secondly, Test D can be evaluated in terms of shear strength which is very objective since the subjectivity of eyeball-tests such as WFP is removed. Considering only the results from Table 4-1 and Table 4-2, one can certainly conclude that Test D has potential as a replacement for Tests A and B.

4.3 Density, grooves and pressure effect

4.3.1 Test A: 100 x 100 mm

Total delamination (Dtot)

Table 4-3: ANOVA table of the total delamination (*Dtot*) values of 100 x 100 mm samples.

Effect	SS	Degr. of Freedom	MS	F	р
	55005.70	1 16600111	55005.70	050 5700	0.000000
Intercept	55885.73	1	55885.73	256,5786	0.000000
Density	1910.65	1	1910.65	8.7720	0.003736
Grooves	2806.91	1	2806.91	12.8869	0.000492
Pressure	3903.25	1	3903.25	17.9203	0.000047
Density*Grooves	3304.88	1	3304.88	15.1731	0.000167
Density*Pressure	11.12	1	11.12	0.0511	0.821650
Grooves*Pressure	102.18	1	102.18	0.4691	0.494815
Density*Grooves*Pressure	487.43	1	487.43	2.2378	0.137481
Error	24394.87	112	217.81		

The data was not normally distributed and was transformed using the Box-Cox transformation. However, the ANOVA with the Box-Cox data showed the same significance of pressure and the density with groove interaction than the ANOVA with non-normal data. This meant that the non-normal analysis could be used as it reached the same conclusion as the analysis of the data transformed toward normality.

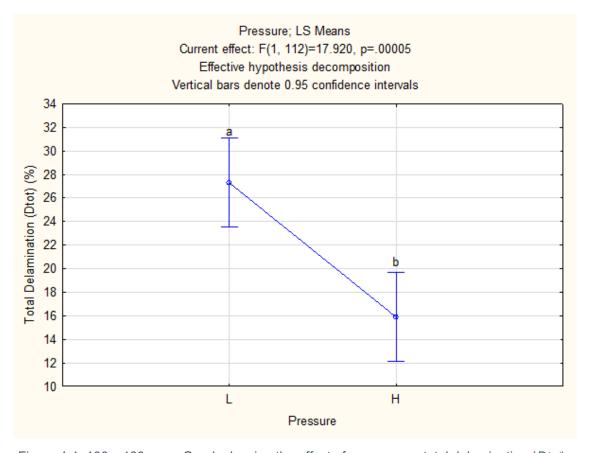


Figure 4-1: 100 x 100 mm - Graph showing the effect of pressure on total delamination (*Dtot*).

The requirements for total delamination (*Dtot*) were taken from Annex C of EN 16351 (2015). Bond strength was considered sufficient if:

- Total delamination (*Dtot*) length did not exceed 10% of the sum of both glue lines.

Figure 4-1 shows a negative relationship between pressure and total delamination (*Dtot*). As pressure increased, total delamination decreased.

Sikora et al. (2016) and Liao et al. (2017) concluded that higher pressing pressure allowed for increased adhesive penetration in the wood substrate and subsequently improved the bonding strength. Kamke and Lee (2007) explained the importance of penetration depth, concluding that deeper penetration would lead to improved bond strength due to increased interaction between wood and adhesive in the form of intermolecular bonding such as van der Waals forces and hydrogen bonds.

Sikora et al. (2016) explained that higher delamination at lower manufacturing pressures came as a result of shallower adhesive penetration causing a thick bondline, exposing a larger adhesive surface to water during delamination testing.

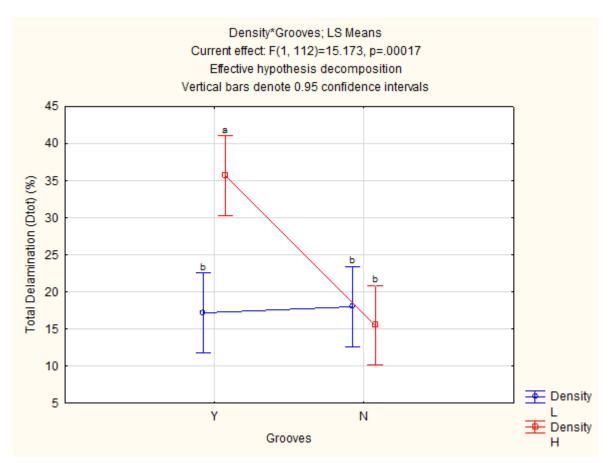


Figure 4-2: 100 x 100 mm - Graph showing the effect of the density and grooves interaction on total delamination (*Dtot*).

Figure 4-2 shows that the difference between high and low density is insignificant in the absence of grooves and highly significant in samples where grooves were present.

The reason for this interaction can be explained by the visual inspection of samples that failed delamination tests. It was noticed that delamination appeared in close proximity to the groove in almost every sample. This lead to the assumption that the groove area, which was filled with glue, created a larger surface area for water penetration resulting in increased delamination.

Thomas et al. (2009) and Frihart and Hunt (2010) explained that high density material results in more exaggerated swelling and shrinkage behaviour. The greater amount of water penetrating the wood substrate through the grooves could have led to greater swelling and shrinkage behaviour in the high density material, causing increased delamination.

Another possible, but less likely explanation could be that the presence of grooves allowed space for the glue to expand upon curing, instead of the adhesive penetrating the wood tissue layer. Coupled with this process, Frihart and Hunt, (2010) explained that high density material has thicker cell walls making adhesive penetration extremely difficult and severely compromising the depth of mechanical interlock to two cells deep; while adding that the high extractive content in higher density material may interfere with adhesive curing and subsequent bond formation. This leads to excessive squeeze out of adhesive and greater delamination. This same squeeze out effect was reported by Pröller, (2017) who found that 1C-PUR adhesives used on dry, high density woods showed increased delamination as a result of poor penetration into the high density wood substrates at a low MC, causing excessive adhesive squeeze out and a thin bond line.

Wood failure percentage (WFP)

Table 4-4: ANOVA table of the WFP values of 100 x 100 mm samples.

Effect	SS	Degr. of Freedom	MS	F	р
Intercept	543716.7	1	543716.7	1571.280	0.000000
Density	3177.6	1	3177.6	9.183	0.003034
Grooves	2498.0	1	2498.0	7.219	0.008313
Pressure	9407.6	1	9407.6	27.187	0.000001
Density*Grooves	6343.8	1	6343.8	18.333	0.000039
Density*Pressure	38.0	1	38.0	0.110	0.741076
Grooves*Pressure	125.1	1	125.1	0.361	0.548952
Density*Grooves*Pressure	618.8	1	618.8	1.788	0.183847
Error	38755.8	112	346.0		

The data was not normally distributed and was transformed using the Box-Cox transformation. However, the same significance of pressure and the density with groove interaction was found for both the non-normal and transformed data. This meant that the non-normal analysis could be used as it reached the same conclusion as the analysis of the data transformed toward normality.

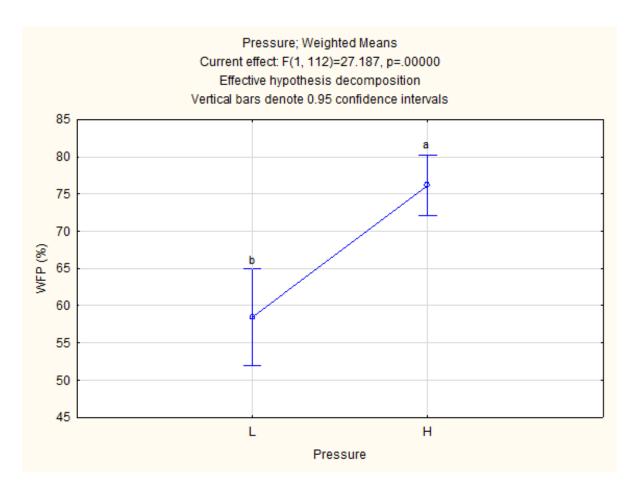


Figure 4-3: 100 x 100 mm - Graph showing the effect of pressure on WFP (%).

The requirements for total delamination (*Dtot*) were taken from Annex C of EN 16351 (2015). Bond strength was considered sufficient if:

- Minimum WFP of the sum of both split glued areas (WFPmean) was not less than 70%.

Figure 4-3 clearly indicates the need for high pressing pressure in order to meet the wood failure requirements according to EN 16351 (2015).

Figure 4-3 shows that an increase in bonding pressure generally leads to increased WFP.

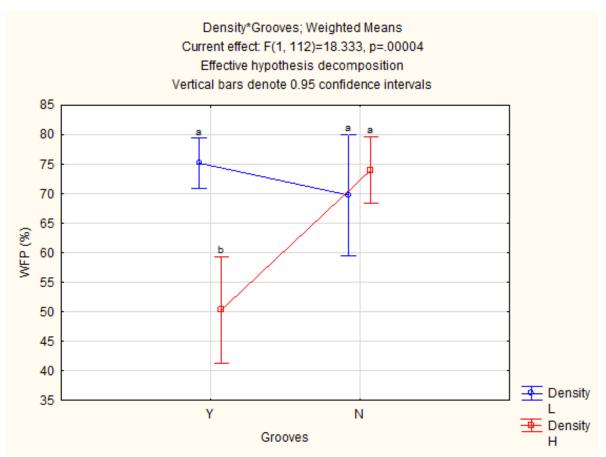


Figure 4-4: 100 x 100 mm - Graph showing the effect of the density and grooves interaction on WFP (%).

Figure 4-4 shows that the difference between high and low density was insignificant in the absence of grooves and highly significant in samples where grooves were present. This is in support of the results in Figure 4-2 for total delamination.

The high density material with grooves had a significantly lower WFP than the low density material. This corresponds well with the findings in Figure 4-2 where high density with grooves led to increased delamination. This indicated that low WFP (high adhesive failure) was the mechanism for the high amounts of delamination in 100 x 100 mm samples tested according to the delamination test method in Annex C of EN 16351 (2015).

4.3.2 Test B - 40 x 40 mm

Shear strength (fv)

Table 4-5: ANOVA table of the shear strength (fv) values of 40 x 40 mm samples.

	SS	Degr. of	MS	F	р
Effect		Freedom			
Intercept	5429.211	1	5429.211	4028.818	0.000000
Density	17.753	1	17.753	13.174	0.000332
Grooves	44.174	1	44.174	32.780	0.000000
Pressure	6.273	1	6.273	4.655	0.031729
Density*Grooves	13.056	1	13.056	9.688	0.002026
Density*Pressure	3.860	1	3.860	2.864	0.091576
Grooves*Pressure	8.112	1	8.112	6.020	0.014692
Density*Grooves*Pressure	7.601	1	7.601	5.641	0.018152
Error	420.449	312	1.348		

The data was not normally distributed and was transformed using the Box-Cox transformation. However, the same three-way significance was found for both the non-normal and transformed data. This meant that the non-normal analysis could be used as it reached the same conclusion as the analysis of the data transformed toward normality.

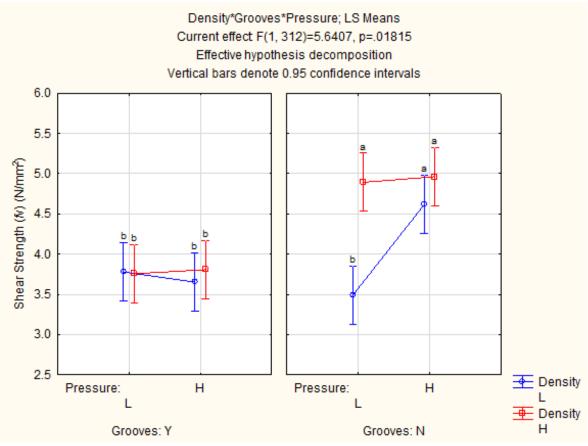


Figure 4-5: $40 \times 40 \text{ mm}$ - Graph showing the effect of the three-way interaction of density, grooves and pressure on shear strength (fv).

The requirements for shear strength (fv) were taken from Annex D of EN 16351 (2015). Bond strength was considered sufficient if:

- 1. The characteristic shear strength (fv,k) derived from tests is fv, $k \ge 1,25$ N/mm² and
- 2. The shear strength (fv) of each glue line must be at least 1 N/mm².

Figure 4-5 displayed a different relationship to what was expected in the absence of grooves. In general, it was expected that high pressure samples would display better bonds and were more likely to display differences in shear strength between low and high density wood, since wood failure would occur rather than bond failure. However, this was not the case and it can be assumed that a good bond formed at high pressure, giving high shear strength (Figure 4-5).

The reason for the significant difference in density at low pressure in the absence of grooves could possibly be explained by the greater strength of the high density samples.

Where stress relief grooves were present, there were no significant differences between factors. It is probable that failure where grooves were present was mainly due to the effect of rolling shear. This implies that bond strength did not play a role in failure and it was rather a wood property limiting the shear strength viz. rolling shear strength. In that case it will make sense that pressure will not affect shear strength. It also implies that wood density did not affect rolling shear strength of the samples. No literature could be found confirming whether rolling shear strength has a relationship with wood density. Further research is required to confirm these assumptions.

Wood failure percentage (WFP)

Table 4-6: ANOVA table after Box-Cox transformation of the WFP values of 40 x 40 mm samples.

	SS	Degr. of	MS	F	р
Effect		Freedom			
Intercept	6669149	1	6669149	1925.827	0.000000
Density	53	1	53	0.015	0.902061
Grooves	4178	1	4178	1.206	0.272903
Pressure	149709	1	149709	43.231	0.000000
Density*Grooves	84119	1	84119	24.291	0.000001
Density*Pressure	3215	1	3215	0.928	0.336062
Grooves*Pressure	31978	1	31978	9.234	0.002576
Density*Grooves*Pressure	8273	1	8273	2.389	0.123214
Error	1080458	312	3463		

The data was not normally distributed and was transformed using the Box-Cox transformation. It is important to note that the values have been transformed and, therefore, actual values have no meaning. The ANOVA table identified the interactions between density and grooves and between grooves and pressure to be significant. However, the three-way interaction graph was displayed for analysis purposes as it corresponds well with the shear strength graph. The WFP after shear failure values were used to determine the type of failure that occurred.

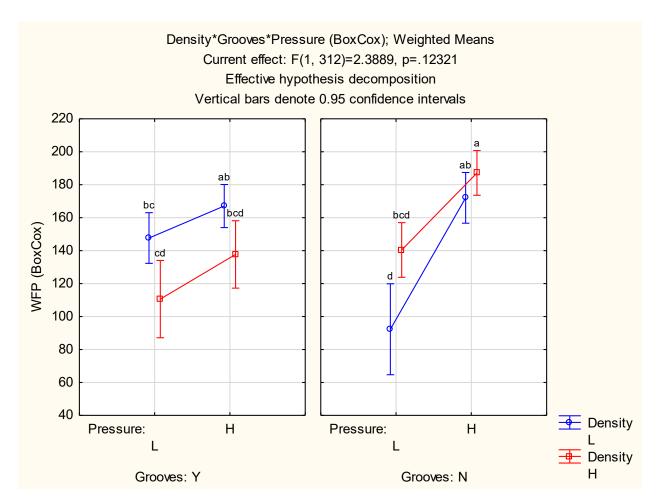


Figure 4-6: 40 x 40 mm - Graph showing the effect of the three-way interaction of density, grooves and pressure on WFP.

The same effect in the presence of grooves was observed as in Figure 4-5. Although the mean wood failure between low and high density samples seemed to be larger, differences were not significant. This confirms the theory that rolling shear appeared to nullify the effects of density and pressure.

The analysis of Figure 4-6 appears to confirm the findings in Figure 4-5. Where grooves were absent, high pressure gave a better bond than in Figure 4-5. The reason for the higher WFP in high density samples was unexplained due to the effect being contrary to expectations. General expectations are that higher density wood is inherently stronger and more likely to fail in the bondline. Added to the increased wood strength, high density wood could possibly have less absorbtion of adhesive, leading to a weaker bond and therefore failure in the bondline.

4.3.3 Test C - 40 x 40 mm at 45°

Shear strength (fv)

Table 4-7: ANOVA table of the shear strength (*fv*) values of 40 x 40 mm samples with grain angle 45° to the load direction.

	SS	Degr. of	MS	F	р
Effect		Freedom			
Intercept	10116.48	1	10116.48	6372.430	0.000000
Density	15.45	1	15.45	9.735	0.001977
Grooves	57.06	1	57.06	35.940	0.000000
Pressure	19.56	1	19.56	12.320	0.000514
Density*Grooves	19.12	1	19.12	12.046	0.000592
Density*Pressure	1.61	1	1.61	1.017	0.314017
Grooves*Pressure	6.73	1	6.73	4.238	0.040365
Density*Grooves*Pressure	0.54	1	0.54	0.340	0.560343
Error	495.31	312	1.59		

The data was found to be normal and the ANOVA table identified the interactions between density and grooves (p = 0.00059) and between grooves and pressure (p = 0.04) to be of statistical significance at a 95% confidence level.

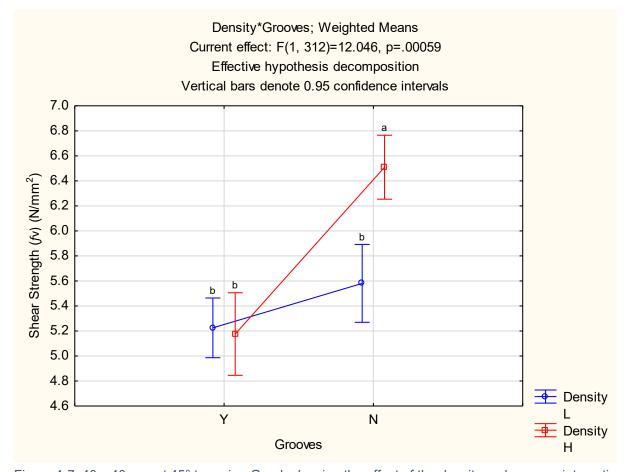


Figure 4-7: $40 \times 40 \text{ mm}$ at 45° to grain - Graph showing the effect of the density and grooves interaction on shear force (fv).

Figure 4-7 showed that density did not have an effect on shear strength when grooves were present. However, in the absence of grooves, high density samples had significantly higher shear strength values than the low density samples. This indicated that the presence of grooves appeared to "nullify" the effect of density on shear strength. The same effect was observed in Figure 4-5 and one can assume the same underlying factors caused this behaviour.

Despite the 45° grain direction, orientated to prevent rolling shear, the failure effect of the grooves seemed to indicate that rolling shear could still be a factor. The grooves "weakened" the wood allowing the wood to "roll over" and fail in itself rather than failing in the glueline. It was expected that the diminished surface area for adhesion, where grooves were present, would lower shear strength results somewhat, however, this seems unlikely as the groove area only made up 3% of the surface area, which was considered to be negligible.

Mestek and Winter (2010) found that tension perpendicular to the grain direction appears in the corners of stress relief grooves as a result of shear deformation. These corner areas have reduced rolling shear capacity and could lead to shear failure at lower strengths.

In addition, low density material allowed for more effective adhesive penetration due to the larger cell lumens and the stress relief grooves allowed for the dispersion of excess adhesive. The combination of these factors could have led to a starved glue line and decreased bonding strength as visible in the low density and grooved material.

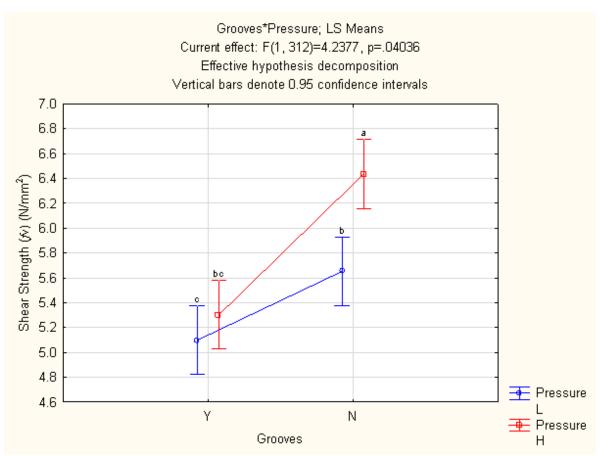


Figure 4-8: 40 x 40 mm at 45° to grain - Graph showing the effect of the grooves and pressure interaction on shear strength (*fv*).

Figure 4-8 showed that pressure had a minimal effect on shear strength when grooves were present in the sample, while it had a significant effect when the sample had no grooves. The reason for the better bonding strength under higher pressure was explained by Sikora et al. (2016) who recognised the effect that bonding pressure had on bond strength using a PUR

adhesive. It was found that better bond strength was achieved at higher clamping pressures due to the deeper penetration of the adhesive.

Knorz et al. (2017) observed that low bonding pressure may be unable to overcome the effect of warping or cupping of the lamellae. The presence of grooves rendered the bonding pressure insignificant. There could be two possible reasons for this.

- The presence of grooves decreased the stiffness in the lamella allowing the low and high
 pressure to overcome the warping or cupping in the lamella. However when grooves
 were absent, only high pressure was able to overcome the higher stiffness in the panel
 and compress it enough to provide sufficient bonding quality.
- 2. Similar to the Density * Grooves interaction behaviour (Figure 4-7), rolling shear may be introduced by the grooves despite the 45° grain orientation. In that case, the bondline is not tested, but rather a wood property. This means that bonding pressure no longer has an effect.

Wood failure percentage (WFP)

Table 4-8: ANOVA table after Box-Cox transformation of the WFP values of 40 x 40 mm samples with grain angle 45° to the load direction.

	SS	Degr. of	MS	F	р
Effect		Freedom			
Intercept	6535611	1	6535611	2072.467	0.000000
Density	3419	1	3419	1.084	0.298584
Grooves	40444	1	40444	12.825	0.000397
Pressure	231936	1	231936	73.548	0.000000
Density*Grooves	33547	1	33547	10.638	0.001231
Density*Pressure	10303	1	10303	3.267	0.071639
Grooves*Pressure	52	1	52	0.016	0.898122
Density*Grooves*Pressure	57467	1	57467	18.223	0.000026
Error	983905	312	3154		

The data was not normally distributed and was transformed using the Box-Cox transformation. It is important to note that the values have been transformed and, therefore, the values will not be WFP anymore. The ANOVA table identified the three way interaction between density, grooves and pressure (p = 0.000026) to be highly significant at a 95% confidence level. However, the interactions between density and grooves and between grooves and pressure were displayed for analysis purposes as they correspond well with the shear strength graphs. The WFP after shear failure values were used to determine the type of failure that occurred.

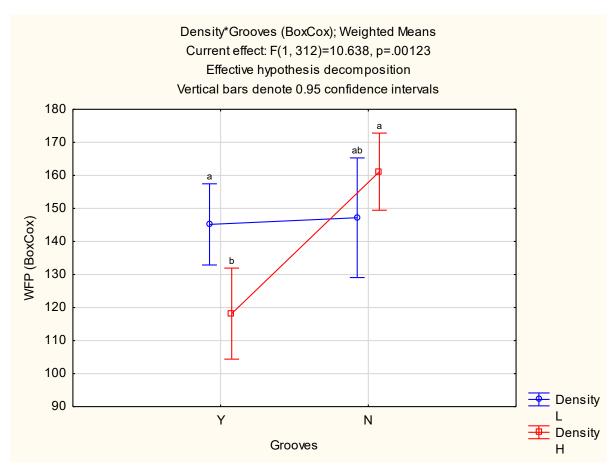


Figure 4-9: $40 \times 40 \text{ mm}$ at 45° to grain - Graph showing the effect of the density and grooves interaction on WFP.

It is important to note that WFP results should be interpreted together with the shear strength results in Figure 4-7 and Figure 4-8.

A significant difference was observed in Figure 4-9 between high and low density where grooves were present. This is in contrast to Figure 4-7 where shear strength values were very similar where grooves were present. The reason for this difference could be that:

- More wood failure was expected in low density wood as it is inherently weaker and more likely to fail in the wood than along the bondline.
- The weaker bond formed in high density wood due to lack of absorption led to bondline failure and thus lower WFP.

If rolling shear played a role, then more wood failure would be expected where grooves were present in the low density material as the wood is weaker and more likely to fail in itself than in the bondline.

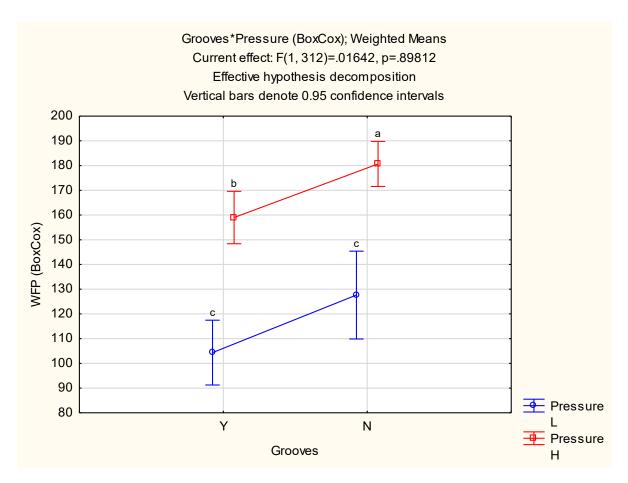


Figure 4-10: 40 x 40 mm at 45° to grain - Graph showing the effect of the grooves and pressure interaction on WFP.

A significant difference was observed between low and high pressure both with grooves and without grooves in Figure 4-10. This was probably largely due to the good bond quality achieved at high bonding pressure. The bond was strong enough to avoid adhesive failure and rather encourage wood failure in both the low density and stronger, high density material. The poorer WFP results observed at low pressure were possibly as a result of the shallow adhesive penetration leading to the lack of quality bond formation in the wood-adhesive interface. This is supported by Kamke and Lee (2007) who proposed that deeper adhesive penetration at higher clamping pressures would lead to improved bond strength in the form of intermolecular bonding.

The difference between high pressure where grooves were present and absent is significant. This could possibly be explained by the adhesive squeeze-out that takes place when the grooves absorb the adhesive leading to a starved bondline and poorer adhesion.

It was observed in Figure 4-8 that the presence of grooves rendered the bonding pressure insignificant. This observation was not supported in Figure 4-10 as a significant difference exists between high and low pressure where grooves are present. The reason for this was not clear.

4.3.4 Test D - 70 x 70 mm at 45°

Total delamination (Dtot)

Table 4-9: ANOVA table after Box-Cox transformation of the total delamination (*Dtot*) values of 70 x 70 mm samples with grain angle 45° to the load direction.

	SS	Degr. of	MS	F	р
Effect		Freedom			·
Intercept	4504.346	1	4504.346	1285.615	0.000000
Density	35.814	1	35.814	10.222	0.001804
Grooves	181.588	1	181.588	51.828	0.000000
Pressure	85.737	1	85.737	24.471	0.000003
Density*Grooves	18.871	1	18.871	5.386	0.022109
Density*Pressure	3.161	1	3.161	0.902	0.344220
Grooves*Pressure	0.422	1	0.422	0.121	0.729134
Density*Grooves*Pressure	13.531	1	13.531	3.862	0.051869
Error	392.409	112	3.504		

The data was not normally distributed and was transformed using the Box-Cox transformation. It is important to note that the values have been transformed and, therefore, are not percentage delamination anymore. The ANOVA table identified pressure and the interaction between density and grooves to be significant.

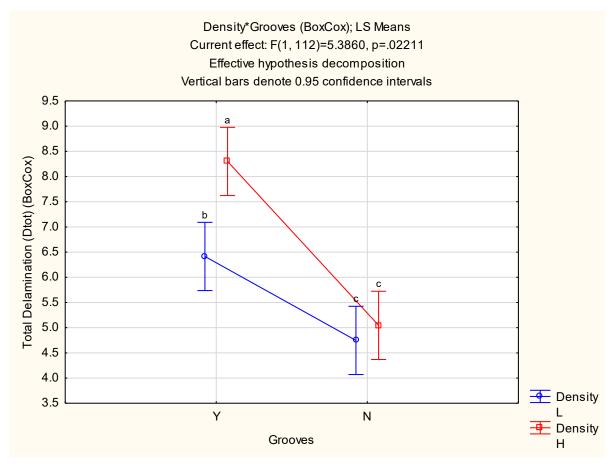


Figure 4-11: 70 x 70 mm at 45° to grain - Graph showing the effect of the density and grooves interaction on total delamination (*Dtot*).

Figure 4-11 shows that the difference between high and low density was insignificant in the absence of grooves and highly significant in samples where grooves were present. This is

similar to total delamination in Figure 4-2. Different behaviour could be observed in Figure 4-7 on shear strength where the opposite interaction was observed.

Somewhat similar interactions were observed, as seen in Figure 4-2, leading to the conclusion that the delamination behaviour in the 70×70 mm samples follows nearly the same trends as in the 100×100 mm samples. A significant difference was observed in the low density samples in the absence and presence of grooves in Figure 4-11. This effect was different to the one observed in Figure 4-2 where no significant difference was observed at low density.

Low density delamination in the presence of grooves is probably as a result of the grooves allowing space for the adhesive to expand upon curing, instead of the adhesive penetrating the wood tissue layer. In addition, for low density where greater adhesive penetration is observed due to thinner cell walls (Frihart and Hunt, 2010). Kamke and Lee (2007) observed the phenomenon where greater adhesive penetration enhances mechanical adhesion, but at the same time leads to a lack of adhesive at the surface of the bond line causing adhesive starvation at the actual glue line and higher delamination values. The combination of these two effects likely led to the increased low density delamination in samples with grooves.

Both high and low density show significantly higher delamination with grooves than their respective delamination values without grooves. This is different to Figure 4-2, with the difference possibly due to the smaller sample size ($70 \times 70 \text{ mm}$) allowing complete saturation with water, leading to greater swelling and shrinkage and resulting in more similar values for high and low density.

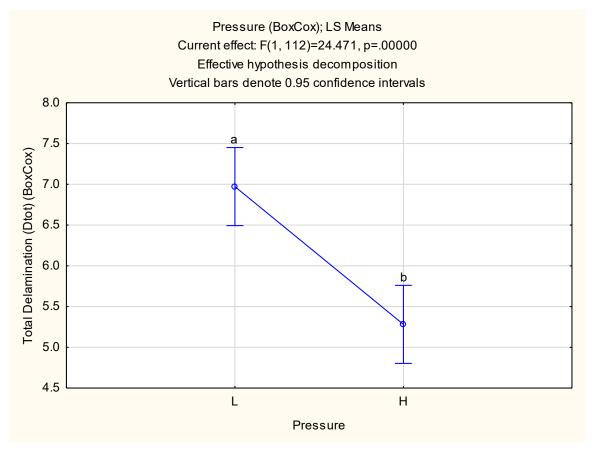


Figure 4-12: 70 x 70 mm at 45° to grain - Graph showing the effect of pressure on total delamination (*Dtot*).

Figure 4-12 shows a negative relationship between pressure and total delamination (*Dtot*). As pressure increases, total delamination decreases. This confirms the exact findings from

Figure 4-1. We can, therefore, conclude that the delamination behaviour in the 70 x 70 mm samples was similar to the delamination behaviour in the 100 x 100 mm samples.

Shear strength (fv)

Table 4-10: ANOVA table after Box-Cox transformation of the shear strength (*fv*) values of 70 x 70 mm samples with grain angle 45° to the load direction.

	SS	Degr. of	MS	F	р
Effect		Freedom			
Intercept	804.7442	1	804.7442	1195.994	0.000000
Density	1.7980	1	1.7980	2.672	0.103477
Grooves	39.2661	1	39.2661	58.356	0.000000
Pressure	11.0518	1	11.0518	16.425	0.000069
Density*Grooves	6.3666	1	6.3666	9.462	0.002350
Density*Pressure	2.9860	1	2.9860	4.438	0.036227
Grooves*Pressure	0.7765	1	0.7765	1.154	0.283820
Density*Grooves*Pressure	2.6107	1	2.6107	3.880	0.050055
Error	156.1050	232	0.6729		

The data was not normally distributed and was transformed using the Box-Cox transformation. It is important to note that the values have been transformed and, therefore, only the interactions can be analysed. The ANOVA table identified the interactions between density and grooves and between density and pressure to be significant.

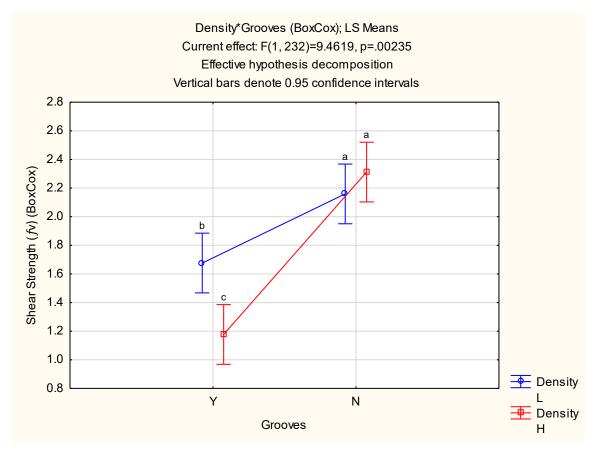


Figure 4-13: 70 x 70 mm at 45° to grain - Graph showing the effect of the interaction between density and grooves on shear strength (fv).

Figure 4-13 shows that the difference between high and low density was insignificant in the absence of grooves and highly significant in samples where grooves were present. This was similar to the delamination behaviour seen in Figure 4-11 - which was not surprising seeing that

the shear tests were done on the same specimens. However, it was quite different to the analysis made in Figure 4-7 on shear strength of 40x40 mm blocks at 45° grain direction where the opposite interaction was observed.

Highly similar interactions were observed, as seen in Figure 4-11, leading to the conclusion that the shear behaviour in the 70×70 mm samples followed similar trends to delamination behaviour in the same samples. This shows that the shear results are greatly affected by the delamination results, indicating that shear testing performed after delamination will give an indication of the true strength values.

The significantly higher shear strength values for low density with grooves, was likely due to the low density material not swelling and shrinking as much in the delamination phase, leaving a stronger bond for shear testing.

Both high and low density show significantly lower shear strength values with grooves than their respective shear strength values without grooves. This change could be due to the delamination procedure already performed on the samples, weakening the bondlines where the grooves were present as the grooves allow greater water penetration.

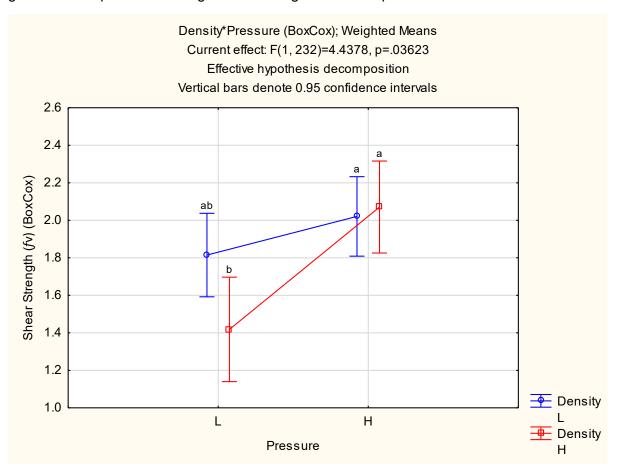


Figure 4-14: 70 x 70 mm at 45° to grain - Graph showing the effect of the interaction between density and pressure on shear strength (fv).

Figure 4-14 shows that the only significant difference was between high density material at low pressure and both high and low density material at high pressure. This indicates that the high pressure was sufficient to bond both high and low density material. This was likely due to the effect reported by Sikora et al. (2016), who recognised the effect that bonding pressure had on bond durability using a PUR adhesive. It was found that higher pressure was directly responsible for deeper penetration and consequently better bond durability.

In contrast, the low pressure seemed insufficient to bond the high density material. The low clamping pressure was unable to sufficiently flatten the stiffer, stronger material to ensure the formation of a good quality bond (Knorz et al., 2017). This was confirmed by Frihart and Hunt (2010) who stated that higher clamping pressure is required to compress the high stiffness, high density wood with large numbers of growth stresses in order to bring the wood layers and the adhesive into contact with each other. The lower pressure likely also struggled to force the adhesive into the smaller cell lumens in the high density material, preventing sufficient adhesive penetration and weakening the bond.

Total delamination (Dtot) vs Shear strength (fv)

In order to determine if a statistically significant correlation exists between the total delamination and shear strength of the samples, a linear regression model was applied to the 70 \times 70 mm samples at 45° to grain results (Figure 4-15).

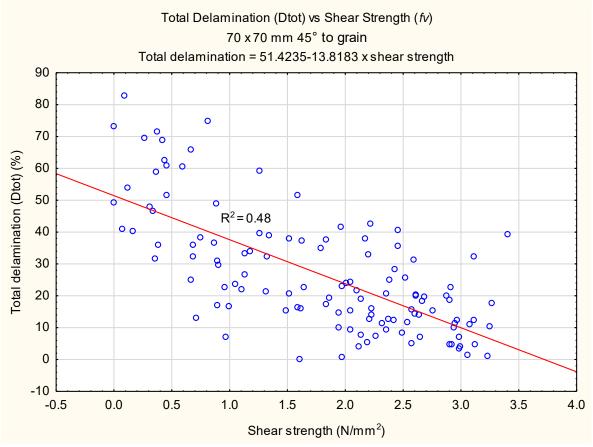


Figure 4-15: 70 x 70 mm at 45° to grain - Graph showing the linear model of total delamination (*Dtot*) vs shear strength (fv).

Figure 4-15 shows that total delamination decreased as shear strength increased.

However, an R-squared value of 0.48 indicated a fairly low goodness-of-fit for this model. This means that only 48% of the variation in results is explained by the fitted regression line, creating doubt over the accuracy of predictions made with the equation.

Wood failure percentage (WFP)

Table 4-11: ANOVA table after Box-Cox transformation of the WFP values of 70 x 70 mm samples with grain angle 45° to the load direction.

	SS	Degr. of	MS	F	р
Effect		Freedom			
Intercept	463760.4	1	463760.4	907.5572	0.000000
Density	4166.7	1	4166.7	8.1540	0.004686
Grooves	19081.7	1	19081.7	37.3419	0.000000
Pressure	26041.7	1	26041.7	50.9623	0.000000
Density*Grooves	7370.4	1	7370.4	14.4236	0.000186
Density*Pressure	400.4	1	400.4	0.7836	0.376960
Grooves*Pressure	350.4	1	350.4	0.6857	0.408465
Density*Grooves*Pressure	3526.7	1	3526.7	6.9015	0.009186
Error	118551.7	232	511.0		

The data was not normally distributed and was transformed using the Box-Cox transformation. However, the same significance for the three-way interaction between density, grooves and pressure was found for both the non-normal and transformed data. This meant that the non-normal analysis could be used as it reached the same conclusion as the analysis of the data transformed toward normality.

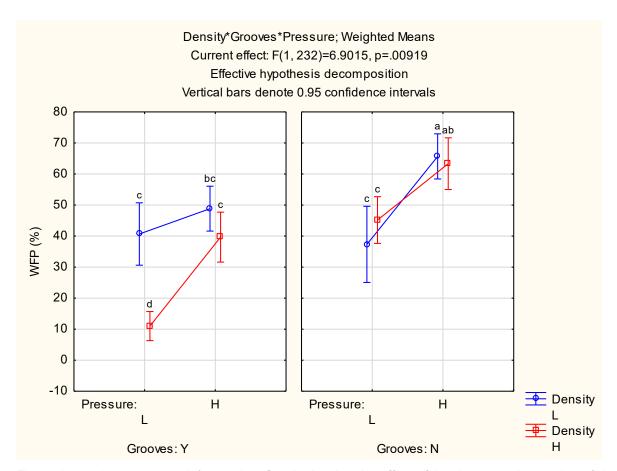


Figure 4-16: $70 \times 70 \text{ mm}$ at 45° to grain - Graph showing the effect of the three-way interaction of density, grooves and pressure on WFP (%).

Figure 4-16 shows that the difference between high and low density was insignificant in the absence of grooves and highly significant only in low pressure samples where grooves were present.

As in Figure 4-12, an increase in bonding pressure generally leads to a better bond and in this case, increased WFP. However, the effect of rolling shear where grooves were present could have led to the lack of significance between low and high pressure for the low density material.

The significantly lower WFP results for high density samples, pressed at low pressure and containing grooves was possibly explained by a few aforementioned reasons:

As was found with the shear strength results, the low clamping pressure was unable to sufficiently flatten the stiffer, stronger, high density material to ensure the formation of a good quality bond (Knorz et al., 2017). The lower pressure likely also struggled to force the adhesive into the smaller cell lumens in the high density material, preventing sufficient adhesive penetration and weakening the bond.

The high density wood likely underwent more swelling and shrinkage during the delamination test cycle, causing the delamination of the bond and leading to lower WFP. The high density wood likely failed in the bondline rather than in the wood due to its greater strength properties.

The grooves allowed for more water penetration likely leading to bond weakening and reduced WFP.

4.4 Shear strength vs WFP - A comparison and correlation analysis

The effect of pressure on shear strength and WFP was compared and the relationship between shear strength and WFP was analysed to determine the statistical correlation between the two factors.

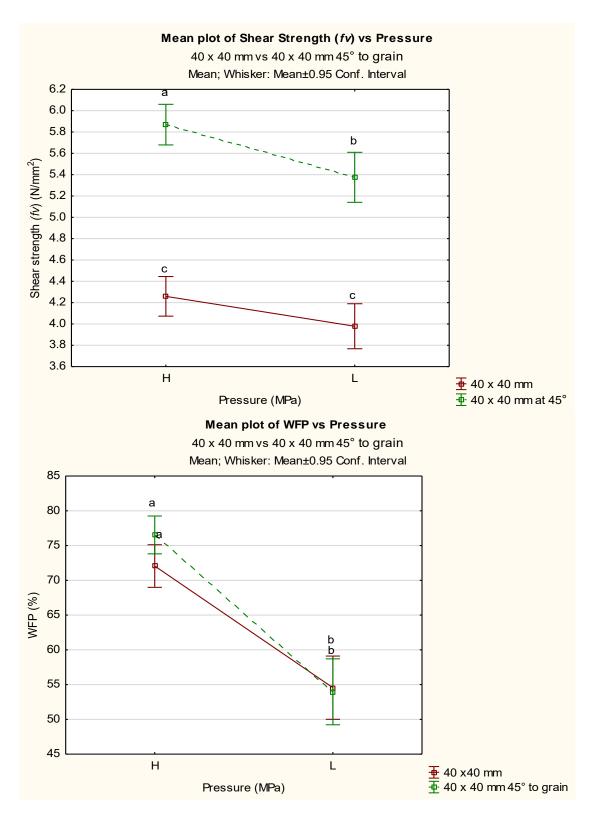


Figure 4-17: 40 x 40 mm vs 40 x 40 mm 45° to grain – Graphs of the comparison of the effect of pressure on shear strength (fv) (top) and WFP (bottom).

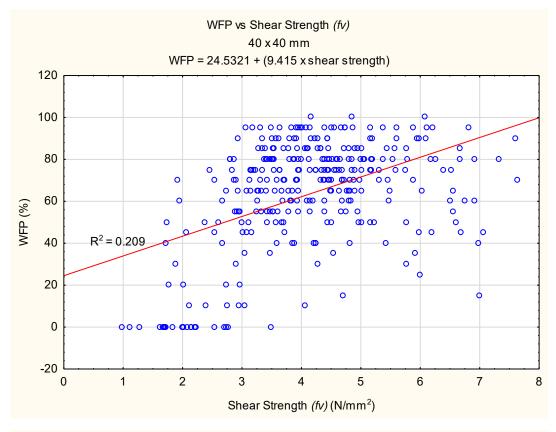
It was observed from Figure 4-17 that the shear strength was substantially higher for both high and low pressure in the 40×40 mm samples at 45° to the grain. This was expected and was likely due to the limited presence of rolling shear in the 40×40 mm samples at 45° to the grain. A similar relationship was observed for both sets of data with high pressure having higher shear strength than low pressure. This was expected as high pressure forms a better bond.

However, a steeper trend was observed in the 40 x 40 mm samples at 45° to the grain, with the high pressure samples being significantly higher than the lower pressure samples. This too could possibly be explained by the limited presence of rolling shear allowing the true effect of a stronger bond at high pressure to show itself.

Figure 4-17 also displayed the effect of pressure on WFP for both sets of data. No significant difference was observed between the 40×40 mm samples and the 40×40 mm samples at 45° to the grain. Rather, a highly similar trend was observed for both with very similar WFP results being recorded. This is very different to the trend observed for shear strength. It was expected that the WFP values for the 40×40 mm samples at 45° to the grain would be lower than the 40×40 mm samples as the rolling shear effect was probably removed.

The significant difference between high and low pressure for both sets of data for WFP was in contrast with the slight significance in the 40 x 40 mm samples at 45° to the grain and no significant difference in the 40 x 40 mm samples for shear strength. The significant difference between high and low pressure for WFP was likely caused by bonding quality. The high-quality bondline at high pressure was stronger than the wood itself and thus caused a bigger part of the failure in the wood material, whereas low pressure specimens with poor bonding failed to a greater extent on the bondline. The same explanation was reported by Pröller (2017).

In order to determine if a statistically significant correlation existed between the WFP and shear strength of the samples, a linear regression model was applied to the 40×40 mm and 40×40 mm at 45° to grain sample results (Figure 4-18).



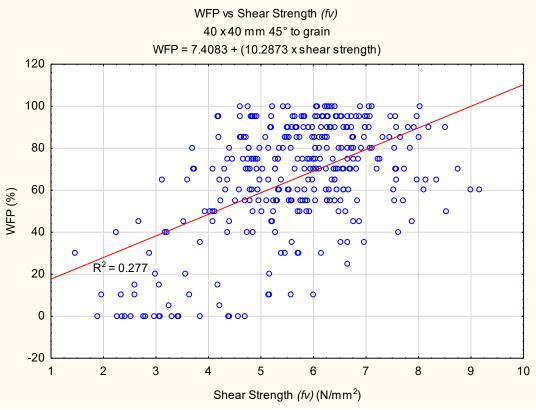


Figure 4-18: 40 x 40 mm (top) and 40 x 40 mm at 45° to grain (bottom) - Graphs showing the linear model of WFP (%) vs shear strength (fv).

Figure 4-18 showed that WFP increased as shear strength increased. This is due to both higher shear strength and WFP values indicating a stronger bond.

However, R-squared values of 0.209 and 0.277 respectively, indicated a fairly low goodness-of-fit for this model.

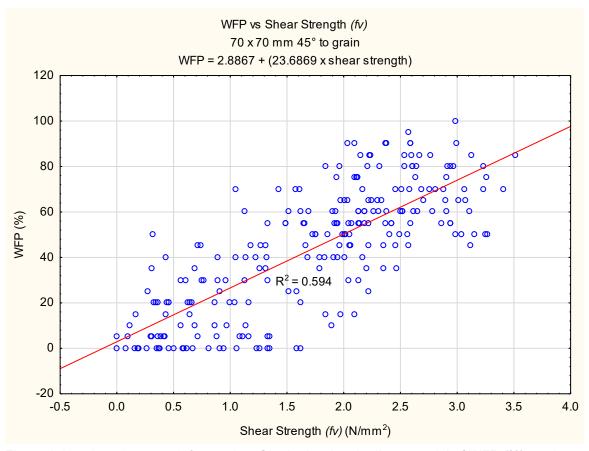


Figure 4-19: $70 \times 70 \text{ mm}$ at 45° to grain - Graph showing the linear model of WFP (%) vs shear strength (fv).

A linear regression model of WFP vs shear strength (fv) was performed on the 70 x 70 mm at 45° to grain sample results (Figure 4-19) to determine if a statistically significant correlation existed between the WFP and shear strength.

Figure 4-19 showed that WFP increased as shear strength increased. This is due to both higher shear strength and WFP values indicating a stronger bond.

The relatively good R-squared value of 0.594 indicated a fairly high goodness-of-fit for this model. The relatively high goodness of fit was likely due to the delamination test being performed prior to the shear test, indicating that bondline degradation played a large part in determining both the shear strength and WFP results. The weakening of the bond during the water soaking and drying cycle, caused failure in the bondline rather than in the wood, leading to a high correlation between shear strength and WFP.

4.5 Comparison of different test methods revisited

From the comparison of test rankings and results from the different tests as discussed in section 4.2, it was mentioned that in most cases all four tests gave relatively similar, comparative results, in terms of rankings of the groups, even though different aspects of bonding quality were tested. Just by observing the ranking of groups according to different test methods, it seemed as if Test D gave a combination of the durability type test (Test A) and the shear type tests (Test B and C) (Table 4-1). In light of ANOVA results of different groups, the test comparisons will be revisited.

From the results of the various test methods it can be seen that bonding failure is a fairly complex phenomenon. Viewing, for example, the effect of clamping pressure in isolation, the following observations can be made:

- In the delamination test (Test A), high clamping pressure gave a consistently better result than low pressure (Figure 4-1).
- For the shear test (Test B), high clamping pressure only gave comparatively good results where no grooves were present – presumably due to the effect of rolling shear (Figure 4-5).
- For the shear test at 45° to the grain direction (Test C), high pressure also only gave comparatively good results when no grooves were present possibly also due to rolling shear (Figure 4-8).
- For the combined delamination and shear test (Test D), high pressure again gave consistently better results (Figure 4-14).
- It seems that rolling shear possibly influenced both the individual shear tests (Tests B and C) to such an extent that the effect of clamping pressure on bonding quality could not be effectively evaluated.
- In the combined delamination and shear test (Test D), the results seems to be closer to that of Test A, with high clamping pressure giving relatively better results than lower clamping pressure (although there was an interaction with density) (Figure 4-14).

The question is then which tests will give the best evaluation of CLT face bonding quality? It seems clear from the results that the different factors influenced bond quality differently in the different tests. The problem with the shear tests (Tests B and C), seems to be that rolling shear has a large effect, overriding bond quality, especially where grooves were present. Test D still seems to be the most objective method for evaluating bond quality since it incorporate a bond durability aspect and uses a more objective measurable (shear strength) than delamination and WFP used in Test A. However, rolling shear will probably still play a role in this method.

A word of caution is also required: An observation was made when the delaminated and sheared samples were analysed that bonding failure may have been affected by the glue spreading system, which at times was seen to not cover the entire surface area when glue failure was observed. This might have been the reason for the poorer results experienced in some of the samples in groups 3 and 5. Care should be taken in the future to ensure complete coverage of the lamella face and that a good even squeeze-out is achieved when pressing takes place.

Chapter 5: Conclusion

From this study the following conclusions can be made:

- Eucalyptus grandis CLT made with 1C-PUR adhesive can obtain excellent face bonding quality as long as the right processing variables are used: Clamping pressure should be high (0.7 MPa) and no stress relief grooves should be present;
- 2. Results from ranking different groups (treatments) in terms of various test results show that delamination testing (Test A) and normal shear testing (Test B), as described in the EN 16351 (2015), gave the biggest ranking differences of all tests evaluated. This suggested that evaluation of bonding quality should have both a durability component, such as provided by the delamination test, and a bond strength test as provided by the shear test:
- 3. Results suggest that rolling shear does influence shear test outcomes and it seems as if a 45° angle shear test still experienced rolling shear when stress relief grooves were present;
- 4. The combined delamination and shear tests (Test D), seems to have potential as a good test for bond quality since it is a combination of a durability and shear strength test (although rolling shear possibly still played a role in this method where grooves were present);
- 5. Complex failure behaviour in the different tests and various interactions between the factors evaluated (density, grooves, pressure) makes it difficult to reach firm conclusions on the effect of each factor. In general though, no grooves and high clamping pressure were preferable. High wood density performed better in some cases in the two shear tests (Tests B and C) while low wood density performed better in some cases in the delamination and combined delamination and shear test (Tests A and D).

There are still questions about the relative advantages of specific test methods for bond quality, especially on the effect of rolling shear. Further work should focus on this aspect and the use of stress models might be a way of gaining further insights.

It is recommended that further studies that are conducted take into account the following additions and improvements:

- The density effect be re-evaluated with bigger distinction between high and low density;
- Improvements could be made to the glue application system by using a roller to ensure complete coverage rather than the method used in this study which may have led to bond quality issues.

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