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THE EFFECT OF HOTSTACKING ON TEMPERATURE AND MOISTURE PROFILES IN MEDIUM DENSITY FIBREBOARD.

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SUMMARY: This study examined the effect of hot-stacking on the development of temperature and moisture content (MC) profiles in commercial 12 mm and 18 mm medium density fibreboard (MDF) panels. Hot-stacks were produced by removing panels from the production line prior to cooling and maintaining stacks for 48 hours. Samples were removed from the stack at specified time intervals and placed in a controlled environment to equilibrate, after which time moisture profiles were generated. The 12 mm and 18 mm individually cooled panels reached equilibrium moisture content (EMC) after 5 and 12 weeks respectively and a moisture profile was evident. Hot-stacked 18 mm panels reached EMC in 4 weeks, whereas the 12 mm hot-stacked panels attained equilibrium in 7 days. Both showed little EMC or MC variation through the thickness. Evidence suggests that the severe conditions created during panel production will alter the physical and chemical nature of the constituent wood fibres. The observed differences in EMC behaviour are likely due to changes in the hygroscopicity of the MDF caused by the temperature and MC conditions in hot-pressing and post-press treatment.

KEYWORDS: Medium Density Fibreboard, Moisture Content, Moisture Profile, Hygroscopicity, pH.

INTRODUCTION

In the production of medium density fibreboard (MDF), mechanically refined wood fibre is resinated and this furnish is then formed into a mattress before hot-pressing. In the hot-pressing process, heated platens introduce heat and compress the mattress. As the heat is transferred from the platens by conduction [6] to the fibre on the surface layers of the mattress, the moisture present vaporises to become steam. The resulting increase in the vapour pressure at the surface of the forming panel leads to the development of a pressure gradient between the surface and the core [17]. This pressure gradient results in steam flow to the core of the mattress, heating the fibre by convection, and increasing the amount of moisture in the core. Within the mattress, the dynamic temperature and moisture content conditions during processing differ from the core to the surface but also from the panel centre to the edge [3]. This variation in the hygrothermal history with position in the mattress will lead to local differences in the hygroscopicities of the particles/fibres in the panel [12]. Evidence of this dynamic situation is shown at press exit, where

the core layers have a higher moisture content (MC) and have a lower temperature than the surface layers [5].

The temperature and moisture content gradients occurring within the mattress, combine with the applied compressive force to create the vertical density profile within a composite panel [1]. As the hot platens of the press heat the surface layers of the mattress, the fibres physically soften and plasticise [18]. Plasticisation of the surface layers leads to densification as the compressive load increases, and furthermore facilitates the development of the common U-shaped density profile.

The equilibrium moisture contents (EMCs) of panel products have been compared to those of wood and were in general, found to be less hygroscopic [13]. Suematsu et al. [15] and Orman [11] found that exposure of wood to high temperatures lowered the EMC. The EMC of panel products is dependent not only on the changes in the hygroscopicity of the wood but also the hygroscopicity and quantity of the binders and additives [12]. In experiments using particleboard, Sekino and Irle [12] found that the EMC of heated particles ranged from 73% to 93% of the control particles. Furthermore, they also found that the EMC of the surface particles was lower than the core particles, and attributed this to the differences in temperature between the surface and the core.

Changes in the chemical nature of the cellulose, hemicellulose and lignin components of wood can occur with heating, and these modifications can alter its hygroscopicity [4]. Alterations in the hemicelluloses with heating can cause a release of acetic acid via the de-acetylation of acetylglucomannan [4]. Changes in particle acidity were observed by Sekino and Irle [12] and claimed to be due to the degradation of hemicellulose, forming acidic by-products during hot pressing. They also found that there was a relationship between the particle acidity and EMC, with lower pH values correlated to lower EMCs. Other studies have found evidence that hemicelluloses in paper and hardboard can be converted to less hygroscopic furfural polymers at very high temperature [14]. Also, Back [2] has hypothesised that the formation of acetyl linkages between hemicelluloses and cellulose can occur, making the material less hygroscopic. Tabarsa and Chui [16] found that hot-pressing modified the hygroscopicity of wood, and also attributed this to the degradation of hemicelluloses, which reduced the number of hydrogen bond sites available for water molecules to attach.

As well as chemical changes on heating, there is also the possibility of physical changes to the wood structure that can influence the EMC behaviour. Hillis [4] states that heating wood with a moisture content below 15% at a temperature of around 100°C results in a physical change that decreases the accessibility of hydroxyl groups in hemicelluloses to water, with a resultant decrease in swelling and mobility, known as hornification. Ohlmeyer and Kruse [10] have carried out a series of hot-stacked particleboard trials where unstacked panels showed differences of approximately 1.5% moisture content between the surface (5%) and core (6.5%). They attributed these differences to the reduction of sorption sites caused by the higher temperatures on the surface of the panels compared to the core during hot pressing. The panels that were hot-stacked had only marginal gradients in moisture contents, with the surface being 5.25% and the core being 5.75%.

The focus of this present study was to examine the evolution of vertical moisture gradients found in MDF panels. Gradients were measured from press exit until equilibrium was attained in a steady state environment. The influence of hot-stacking was also investigated to determine the effect on moisture content gradient and final EMC. Additionally fibre chemistry was examined to explain any observed differences.

METHODOLOGY

The panels used for this study were produced on a commercial production line and removed from the line during a standard product run. The fibre was *Pinus radiata* and the resin used was a E1 urea formaldehyde (UF) resin. Two panel thickness were evaluated, both having resin and wax loadings of 10% and 0.5% respectively. The 18 mm panel was pressed to achieve a target density of 725 kgm^{-3} and the 12 mm panel to 780 kgm^{-3}. The panels were hot pressed in a Washington Iron Works™ multi-daylight press, with a ten panel capacity. The two thicknesses of MDF panels used for the data collection in this study were produced on different production runs.

Three 18 mm and three 12 mm panels were removed from the production line prior to the first cooling wheel. Removal at this stage was done to minimise changes in panel moisture content and temperature. The panels exiting the production line measured 7.5 x 1.2 m and were immediately cut into six 1.2 x 1.2 m panels for ease of handling. One 1.2 x 1.2 m control panel from each of the two thicknesses was allowed to cool with both surfaces exposed to ambient conditions. The remaining 1.2 x 1.2 m panels were stacked "hot", to produce two hot-stacks containing 17 panels each.

Temperature measurements

The temperatures of the 18 mm control and hot-stack panels were measured, from press exit for 48 hours, using T-type thermocouples and a Fluke™ datalogger for data capture. The fibre mattress was stopped for 45 seconds before entering the multi-daylight press, which allowed five thermocouples to be inserted at half thickness into the centre of the mattress. The five panels containing thermocouples were placed uniformly through the hot-stack. Thermocouples were also placed within the hot-stack on the surface of these five panels. In the case of the 12 mm panels, no thermocouples were inserted into the mattresses prior to pressing. The temperature in the centre of the stack was monitored using a single thermocouple placed on the surface of the centre panel. For both trials the hot-stack temperature was monitored for 48 hours.

Moisture content determination

Control panel

Samples 40 x 40 mm were cut from one 1.2 x 1.2 m panel and placed in a controlled environment room, at 50% ± 5% RH and 23°C ± 2°C. Additionally one sample was cut as soon as it was removed from the production line, approximately 20 minutes after pressing. Panel sections were removed from the controlled environment at 4, 8 and 24 hours, then at 24 hourly intervals for the first two weeks and finally at weekly intervals until equilibrium had been reached. Samples were removed and cut into 40 x 40 mm blocks. The 40 x 40 mm blocks were sliced through the thickness, into layers approximately 2 mm thick. The individual layers were weighed and then placed in a humidity cabinet set at 50% ± 5% RH and 23°C ± 2°C. The slices were weighed periodically until they reached equilibrium, at which time the EMC for each different layer was calculated and compared to the EMC of the whole panel.

"Hot-stacked" panels.

The method in which samples were collected for the two thickness panels was slightly different. For the hot-stack of 18 mm panels, a panel adjacent to the central panel containing the thermocouples was removed and samples prepared as for the control panel. To maintain the same

level of load in the hot-stack, this panel was returned to the top of the stack once the moisture samples had been removed. In the case of the 12 mm hot-stack of panels, the central panel was removed and a jigsaw was used to remove a section of panel large enough for moisture samples. This panel was then returned to its central position in the stack.

The samples were sliced as described above, at the same time intervals. The hot-stack was disassembled after 48 hours and samples from the central panel were placed in the controlled environment. The weight of the samples was observed until equilibrium had been attained.

Contact Angle

Sample strips 50 x 6 mm were prepared from the 18 mm control and 18 mm hot-stacked panels. These were sliced to give ten samples through the thickness, approximately 2 mm thick, with five replicates for each layer measured. A droplet of de-ionised water was placed on the surface of the panel layer sample and a photograph was taken using a JVC-3CCD colour video camera. The camera took 10 photographs at one-second intervals. The internal contact angle of the water droplet was determined from the digital images using an automated system with Optimas 6.0 software.

Chemical analysis

For the chemical analysis only the 18 mm control and 18 mm 24 hour hot-stack panels were used.

pH

Three replicates from the core and surface layers of the 18 mm control and 24 hour hot-stacked panels were prepared using a sled microtome. A sample containing 0.5 g of shavings was added to 50 ml of de-ionised water and stirred for 2 hours. After standing for five minutes the pH of the solution was determined using a Radiometer™ pH meter 28.

Solid state NMR

Samples were analysed using a Bruker™ 7 mm DB MAS multinuclear probe in a 200 MHz/52 mm bore Bruker™ magnet. Samples of panel were taken from the surface and core layers of the 18 mm control and 24 hour hot-stacked panels. The sample shavings were packed into 7 mm zirconia rotors for analysis. Over 10,000 scans were acquired in the FID and transformed using a line broadening on 10 Hz.

Solution state NMR

Core and surface layers from the 18 mm control and 24 hour hot-stacked panels were placed in a freezer for 12 hours at -15°C. The layers were then ground in a Wiley mill with a 5 mm mesh screen. The samples were ground as frozen to reduce the possibility of heating, which may alter the nature of the sample. A ground sample (10 g) was extracted with de-ionised water (100 ml) at 20°C for 2 hours, filtered and freeze dried. The sample of the extract was dissolved in the minimum amount of d6-DMSO and spectra acquired on a Bruker™ AC 400 MHz. Two nuclei spectra for each sample were acquired, ^{1}H and a ^{15}N run at 400.13 MHz and 40.56 MHz respectively. The ^{1}H used a standard 30 degree pulse program with 0.3 Hz line broadening and the ^{15}N ran using a DEPT135 (distortionless enhancement by polarisation transfer, with a spectral editing of 135 degrees) pulse program with CPD coupling and no line broadening.

RESULTS AND DISCUSSION

Temperature

From monitoring the cooling of control and hot-stacked panels, a series of cooling curves were generated, these can be seen in Figure 1. The temperature of the 18 mm control panel falls very rapidly and reached ambient temperature in approximately 2 hours, whereas the temperature in the centre of the 18 mm panel hot-stacked for 48 hours was approximately 40°C. For the first 6 hours in the hot-stack, the temperature of both the surface and the core of the centre panel were very similar. The slight increase in temperature evident could possibly be a result of heat transfer from the other panels within the stack or from an exothermic reaction within the panels. Also, over time a small temperature difference was evident between the surface and the core of the 18 mm hot-stacked panel. The cooling curve of the central panel of the 12 mm hot-stack (Figure 1) shows similar cooling characteristics to the 18 mm hot-stacked panels, with temperatures of 85°C but cools more rapidly. The differences in temperature after 48 hours were due to the 17 panel hot-stacks being of different sizes due to panel thickness, allowing the 12 mm panels to cool more rapidly than the 18 mm.

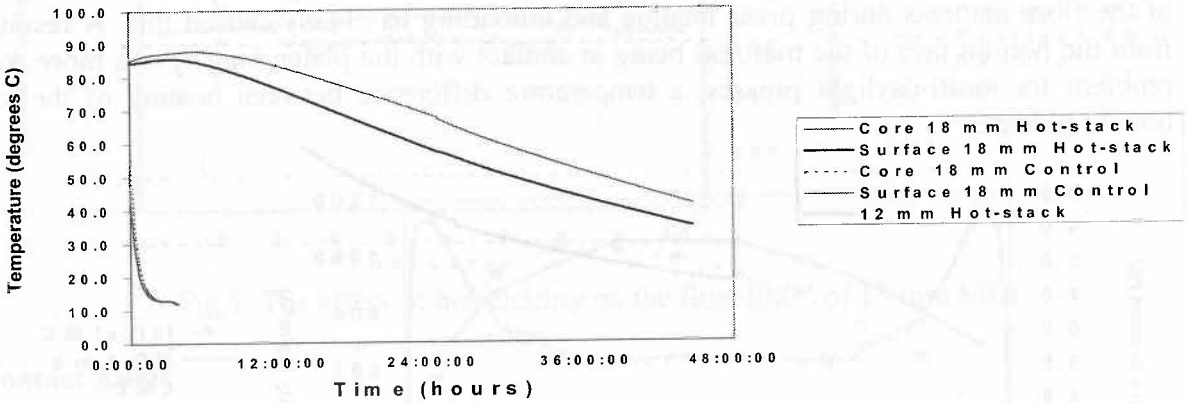

Figure1 Temperature histories of 12 mm and 18 mm commercial panels.

Moisture Content

In Figure 2 the effect of hot stacking on the EMC of 12 mm panels can be seen. The density profile of the 12 mm panel has been overlaid on this plot to relate density with the layer position. The initial moisture content is that of the panel exiting the hot-press and was the same for both the control panel and the hot-stacked panels. The moisture profile illustrates differences in moisture content of over 3% between the surface and the core of the panel upon press exit. These types of differences are typical in panel products and have been well documented [5,7]. The 12 mm control panel required five weeks in the controlled environment to come to a steady state equilibrium whereas the panel that had been hot-stacked for 48 hours required only 7 days to attain this equilibrium. There also exists differences in EMC between the control and the hot-stacked panels. The effect of hot stacking on the EMC can be clearly seen as a relative lowering of the core EMC and an increase in the surface EMC, whereas a greater EMC difference is observed between surface and core of the control panel.

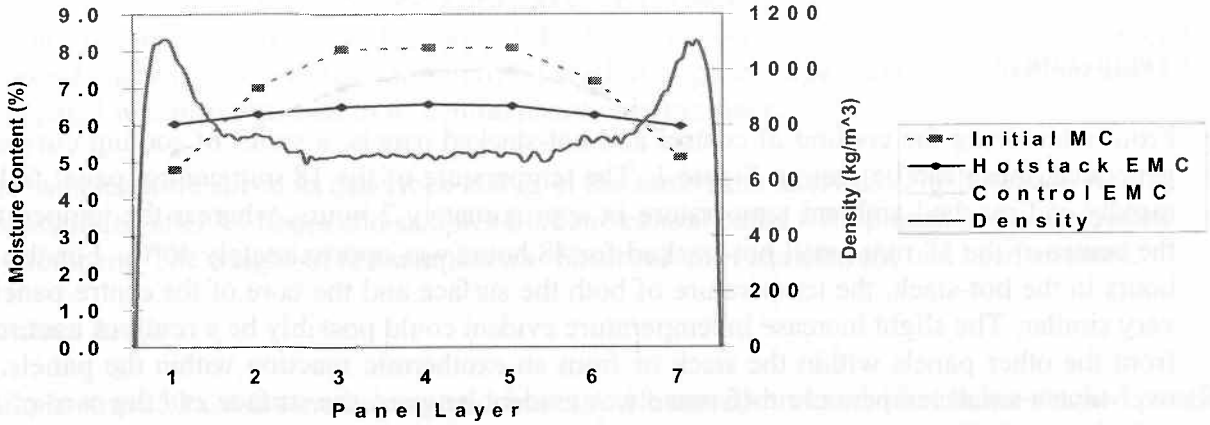

Fig.2. Effect of hotstacking on the final EMC of 12 mm MDF

Figures 3 and 4 show the change in moisture content of the 18 mm control panel and the hot-stacked panels with respect to time. As found for the 12 mm panels there was a difference of approximately 3% between MC of the surface and core layers. The initial (ex-press) moisture content profile is skewed, with the topside of the panel having a higher MC. An uneven heating of the fibre mattress during press loading and unloading has likely caused this. A result either from the bottom face of the mattress being in contact with the platen longer, or a more common problem for multi-daylight presses, a temperature difference between heating of the top and bottom platens.

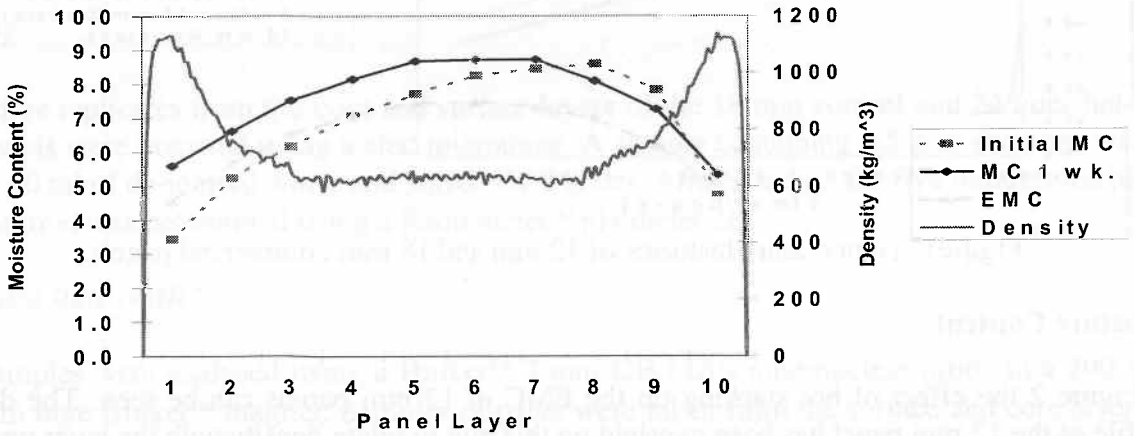

Fig.3. Moisture content changes in the 18 mm control panel

In Figure 4 it can be seen that the MC of the hot-stacked panels changed very rapidly after entering the hot-stack. The surfaces of the 4 hour hot-stacked panel still have a lower MC than the core, but within 24 hours the surfaces have adsorbed moisture and there is only a small difference from the core of the panel. The slight increase in the overall MC of the layers is because the panel is coming to EMC in the set environment.

The effect of hot stacking on the EMC of the different layers in the 18 mm panels can be seen in Figure 5, and this effect is similar to the 12 mm panels. The control 18 mm panel took 12 weeks to attain complete equilibrium, whereas the panels that had been in the hot-stack for 48 hours attained equilibrium in only 4 weeks. The longer time for the 18 mm hot-stacked panels to equilibrate was probably due to the differences in panel thickness.

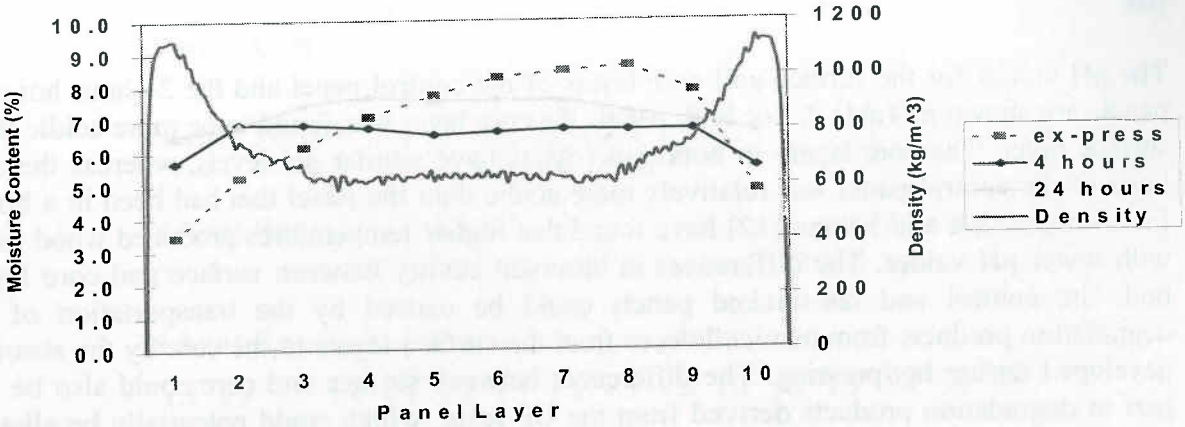

Fig.4. The effect of hotstacking time on the MC of 18 mm MDF

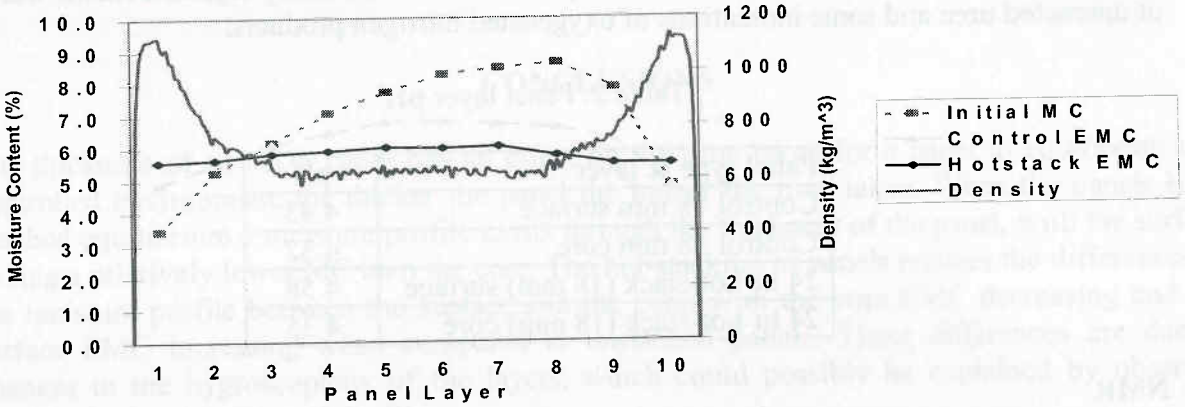

Fig.5. The effect of hotstacking on the final EMC of 18 mm MDF

Contact Angle

Contact angle measurements for the 18 mm control and hot-stacked panels are shown in Table 1. The variability in the results is due to the differences in surface porosity of the layers. The water droplet may have been applied to areas with varying amounts of void space. Generally, for the control and the hot-stacked panels the surface layers have a smaller contact angle than the core layers. This implies that the surface layers are less hydrophobic than the core layers. EMC values, however, would indicate that the surface layers are more hydrophobic (Figure 2). Hot-stacking had no effect on the contact angles of core layers, whereas the surface layers of the hot-stacked panels had comparatively lower contact angles than what was found for the control panels. Due to other factors, such as, porosity, permeability and density, which were not looked at it was difficult to draw conclusions on hygroscopicity based on contact angle measurements.

Table 1. The contact angles for the 18 mm control and 18 mm hot-stacked panels.

	Control		4 hrs hot-stack		24 hrs hot-stack		48 hrs hot-stack	
Layer	Initial (θ°)	StDev (θ°)	Initial (θ°)	StDev (θ°)	Initial (θ°)	StDev (θ°)	Initial (θ°)	StDev (θ°)
Surface	99.70	3.98	90.10	4.83	91.00	5.68	104.58	5.35
2	104.46	2.42	107.63	3.66	102.46	4.77	111.02	2.25
3	107.60	5.23	112.31	3.11	102.68	5.68	112.42	3.06
4	108.57	4.41	106.05	2.56	105.80	1.14	113.60	1.65
Core	104.28	3.78	110.11	1.95	106.61	3.84	107.27	7.38

pH

The pH values for the surface and core layers of the control panel and the 24 hour hot-stacked panels are shown in Table 2. For both panels the core layer was found to be more acidic than the surface layer. The core layers in both panel types have similar pH levels, whereas the surface layer of the control panel was relatively more acidic than the panel that had been in a hot-stack for 24 hours. Irle and Sekino [12] have found that higher temperatures produced wood particles with lower pH values. The differences in observed acidity between surface and core layers of both the control and hot-stacked panels could be caused by the transportation of acidic degradation products from hemicelluloses from the surface layers to the core by the steam front developed during hot-pressing. The differences between surface and core could also be due in part to degradation products derived from the UF resin, which could potentially be alkaline in nature. Surface layers are exposed to extreme temperatures potentially causing more degradation of the resin. Unfortunately, the solution state ^{15}N NMR has been unable to confirm the presence of degraded UF species in the water soluble panel extracts. The only signals evident were those of unreacted urea and some indications of oxygenated nitrogen products.

Table 2. Panel layer pH

Panel type & layer	pH
Control 18 mm surface	4.43
Control 18 mm core	4.32
24 hr hot-stack (18 mm) surface	4.58
24 hr hot-stack (18 mm) core	4.35

NMR

The spectra from the solid state ^{13}C NMR showed no evidence of any differences between the core and surface layers or the control and hot-stacked panels. However, in Figure 6, the ^{1}H NMR spectra shows that there are differences between the surface and core layers for the control and hot-stacked panels.

Table 3. The peak ratios of the ^{1}H NMR on the cold water extractives from 18 mm panels.

Sample	Relative ratio index (ppm)			
	3.5	4.5	5.5	6.5
Control Core	1.0	0.36	0.21	0.18
Control Surface	1.0	0.63	0.48	0.34
24 hour Hot-stack, Core	1.0	0.5	0.33	0.34
24 hour Hot-stack, Surface	1.0	0.55	0.41	0.3

The peaks at 3 to 5 ppm are in the typical carbohydrate backbone region whereas peaks between 5 and 7 ppm are more typical of polyphenol type materials [8,9]. In the surface layers proportionally more water-soluble polyphenolic compounds were in evidence (Table 3). An increase in the ratio of polyphenolics to carbohydrates could explain why there are differences in the EMC between the surface and the core of the control panel. Carbohydrates are more hydrophilic and polyphenols tend to be more hydrophobic in nature. The ratio values for the hot-stacked panels in Table 3 could also explain why the MC of the surface and core layers change when the panel is hot-stacked. The ratios in the surface layers decrease, leading to an increase in hygroscopicity and the ratio of the core layers increases leading to a decrease in hygroscopicity.

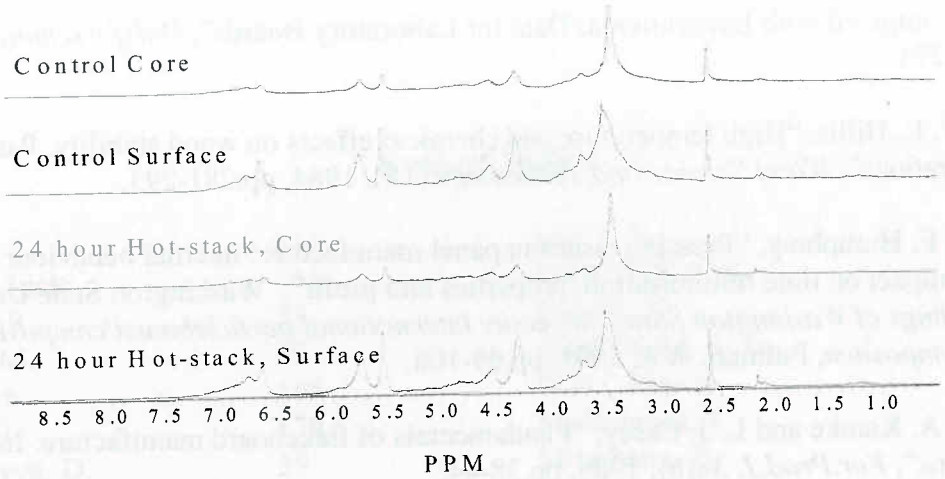

Figure 6 The ^{1}H solution state NMR spectra.

CONCLUSIONS

The thickness of an MDF panel has an effect on the time taken for a panel to equilibrate in a controlled environment, the thicker the panel the longer the time taken. When the panels have reached equilibrium a moisture profile exists through the thickness of the panel, with the surface having a relatively lower MC than the core. The hot-stacking of panels reduces the differences in the moisture profile between the surface and the core, with the core EMC decreasing and the surface EMC increasing when compared to unstacked panels. These differences are due to changes in the hygroscopicity of the layers, which could possibly be explained by observed changes in the chemistry of the fibre.

The production process causes both physical and chemical differences between core and surface layers. Differences in the chemistry occur between the core and surface layers of the hot-stacked and unstacked panels. The post-press treatment process of hot-stacking also alters the differences between the pH of the core and surface layers.

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REFERENCES

1. C. K. Andrews, P. M. Winistorfer and R. M. Bennett, "The influence of furnish moisture content and press closure rate on the formation of the vertical density profile in orientated strandboard", *For.Prod.J.* 51(5), 2001, pp.32-39.

2. E. L. Back, "Thermal auto-cross-linking in cellulose material", *Pulp & Paper Mag.Can.* (68), 1967, pp.165-172.

3. A. J. Bolton, P. E. Humphrey and P. K. Kavvouras, "The Hot Pressing of Dry-formed Wood-based Composites. Part 3. Predicted Vapour Pressure and Temperature Variation with

Time, Compared with Experimental Data for Laboratory Boards", *Holzforschung* 43(4), 1989, pp.265-274.

4. W. E. Hillis, "High temperature and chemical effects on wood stability. Part 1: General considerations", *Wood Science and Technology* (18), 1984, pp.281-293.

5. P. E. Humphrey, "Pressing issues in panel manufacture: Internal behaviour during pressing and its impact on time minimisation, properties and profit", Washington State University. *Proceedings of Washington State University international particleboard/composite material series symposium*, Pulman, WA, 1991, pp.99-108.

6. F. A. Kamke and L. J. Casey, "Fundamentals of flakeboard manufacture: Internal-mat conditions", *For.Prod.J.* 38(6), 1988, pp.38-44.

7. F. Kayihan, J. A. Johnson and C. Lubon, "Preliminary Calculations of Heat and Mass Transfer During the Pressing Operation of Wood Composite Materials Manufacture", R. W. Lewis, K. Morgan and B. A. Schrefler Ed., *Numerical Methods in Thermal Problems*. (2), 1981, pp.277-288.

8. A. G. McDonald, A .B. Clare and A. R. Meder, "Chemical Characterisation of the Neutral Water Soluble Components from Radiata Pine High Temperature TMP Fibre", *Proceedings 53rd Appita Conference.* (2), 1999, pp.641-647.

9. R. T. Morrison and R. N. Boyd, "Spectroscopy and Structure", *Organic Chemistry*, (16), Boston. Allyn and Bacon, Inc., 1987, pp.585.

10. M. Olhmeyer and K. Kruse, "Hot Stacking and its effects on panel properties", *Proceedings of the Third European Panel Products Symposium*, 1999, pp.293-300..

11. H. R. Orman, "The response of New Zealand timbers to fluctuations in atmospheric moisture conditions", N.Z. Forest Service No. 8, Forest Res. Inst. Tech. Pap., 1955.

12. N. Sekino and M. A. Irle, "Particleboard Hygroscopicity: Its Prediction and the Influence of Hot-pressing", *Mokuzai Gakkaishi* 42(1), 1996, pp.43-50.

13. S. Shinda, "Equilibrium Moisture Content of Some Wood-based Materials" *Mokuzai Gakkaishi* 37, 1991, pp.981-985..

14. A. J. Stamm, *Wood and Cellulose Science*, New York, The Ronald Press Company, 1964.

15. A. Suematsu, N. Hirrai, and F. Saito, "Properties of hot pressed wood. Part 1", *Mokuzai Gakkaishi* 26, 2001, pp.581-586.

16. T. Tabarsa and Y. H. Chui, "Effects of hot-pressing on properties of white spruce", *For.Prod.J.* 47(5), 1997, pp.71-76.

17. J. H. van Houts, "Residual stresses and their implications in medium density fibreboard", PhD Dissertation. University of Auckland, 2001.

18. J. C. F. Walker, "Wood panels: particleboards and fibreboards", *Primary Wood Processing. Principles and Practice.* (12), London, Chapman and Hall, 1993, pp.419-478.