WOOD RESEARCH
51 (4): 2006
37-46

DETERMINATION OF MOISTURE CONTENT OF RESIN MODIFIED WOOD

Tomáš Joščák
Faculty of Wood Sciences and Technology, Technical University in Zvolen, Slovakia

Andreas Krause, Andreas Treu, Holger Militz
Institute of Wood Biology and Wood Technology, University of Göttingen, Germany

ABSTRACT

Resins and oils can easily evaporate during drying processes at high temperatures. The aim of this research was to investigate different drying methods such as oven-drying, vacuum-drying and freeze-drying of resin modified pine- sapwood samples to determine wood moisture content (MC) and weight percent gain (WPG). The results showed that freeze-drying is the slowest process. Vacuum drying of water impregnated samples takes approx. 7 times longer compared to oven-drying. The initial moisture content of wood before impregnation used in this research has only little influence on the WPG.

KEY WORDS: oven drying, vacuum drying, freeze drying, drying, dimethylol-dihydroxy-ethylene, moisture content, resin, modification

INTRODUCTION

The moisture content (MC) has a strong influence on the mechanical and physical properties of wood. Therefore, the determination of MC is one of the most important wood characterisation. Several methods were developed for MC measurement on untreated wood. The most exactly methods use the weight analyses of wood mass and/or water.

The commonly used method to determine the moisture content of wood in laboratory trials is oven drying at 103°C. However during drying of modified wood at this temperature besides water, an unknown amount of volatile organic compounds (Shmulsky 2000, McDonald et al. 1999, Risholm-Sundman et al. 1998) and chemical substances (Kollmann and Höckele 1962) evaporate. Furthermore the drying process of modified wood with conventional oven drying showed a migration of impregnated chemicals to the outer layer of wood, causing an uneven distribution of the chemical.

Other methods, such as Karl-Fischer-Titration (Lohse and Dietrich 1972, Resch and

Ecklund 1963, Kollmann and Höckele 1962) or nuclear magnet resonance (Hartley et al. 1994) could be used to determine the wood MC, however these methods are labourous, time consuming and costly.

Determining factors for the evaporation of liquids from wood are the temperature and the pressure used (Mortimer 1987). A decreased temperature can be compensated by a low pressure (Lee and Jung 2000, Ressel 1999, Ward and Simpson 1987, Gillwald and Eichler 1964). Vacuum drying yield to a favourable moisture transport (Neumann et al. 1993). Another drying method, which uses low temperature, is freeze-drying. Because of the high costs and long drying times compared to other methods, freeze drying is up to now not applied in the wood industry (Matejak et al. 1989, Matejak and Jarczyk 1990, Wittköpper 1998), except for archaeological wood. During the process of freeze-drying water sublimates from a solid phase directly into gas, with a limited moisture transport.

The aim of this study was to determine the change of MC in modified pine wood during drying. Additionally the influence of the initial MC of wood before treatment and the leaching of extractives during the impregnation was investigated.

MATERIAL AND METHODS

Samples of pine sapwood (*Pinus sylvestris,* L.) with the dimensions 25 x 25 x 20 mm (r x t x l) were used. Each treatment consists of 5 samples (n = 5).

Impregnation

The samples were impregnated for 30 minutes at a vacuum of app 10 mbar and stored in the solution for 24 hours at atmospheric pressure. As a model substance for a cross-linking resin, dimethylol-dihydroxy-ethyleneurea was used in different concentrations. Distilled water was used as a reference. Before impregnation, the dry-weight of the samples was determined by oven drying at 103°. For the investigation of the drying behaviour, the initial moisture content (MC) of wood before impregnation was 0%. For the investigations of the effect of MC of wood before impregnation, wood samples were conditioned to an equilibrium moisture content of approx. 27%, 12%, 14% and 0% respectively. The MC of these samples was calculated from randomly selected samples.

Measurements

To determine the change of MC weight was measured during drying. During vacuum-drying and oven-drying, the temperature of the samples was constantly measured in the centre of one sample with an external sensor.

Leaching during impregnation

Two solutions were used to determine the influence of leaching of extractives from wood during impregnation. Before and after oven drying, water impregnated samples were dried to constant weight to determine the mass loss. To determine the influence of leaching during resin impregnation, two sample groups were dried in parallel. Before impregnation, one group of samples was extracted by Soxhlet extraction with boiling water and applied for 6 h (simulation of leaching during impregnation). After oven-drying and after vacuum-drying the weight of both groups was compared.

Oven-drying

Oven-drying was performed in a drying oven at 103°C under atmospheric pressure. The weight of wood samples was measured with an analytical scale.

Vacuum-drying

Vacuum-drying was done at a pressure below 10 mbar. The temperature inside of the sample reached up to 50°C. To determine the weight loss during drying, the vacuum was interrupted.

Freeze-drying

Before freeze-drying the samples were frozen at a temperature of -55°C for 24 hours at atmospheric pressure. After freezing, the samples were displaced in a vacuum oven at 10 mbar for 24 hours. To determine the gradient of moisture content while freeze –drying, wood samples with dimensions of 45 x 45 x 40 mm (r x t x l) were used. Both cross-sections were sealed.

Moisture gradient

After 24 hours of freeze drying an 8 mm thick cross-section of the middle of the sample was additionally prepared. This sample was split into 25 single parts with dimensions approx. 9 x 9 x 8 mm (r x t x l). The MC of these samples was measured after 24 h drying at 103°C.

Calculation of solid content

As nominal value for a dried resin of impregnated samples the dry-matter value of oven dried 30% resin solution was used.

RESULTS

In comparison to oven and vacuum drying, freeze drying was the slowest drying process after 24 hours (Tab. 1). An amount of extractives after drying and after impregnation was observed. A tendency to minor mass loss (up to 1% of WPG) of volatile extractives was observed after oven drying. Leaching of wood extractives during impregnation with water or resin solution showed also a minor mass loss up to 1 % based on the wood dry-weight.

During oven drying, the reference samples reached 0% MC after app. 7 h and after 72 hours during vacuum drying. In the first drying phase, a rapid decrease of MC was measured by vacuum drying and oven drying. This phase takes longer in modified wood than in the references. However the evaporated volume of water was higher in the reference samples. Dry-matter of the resin solution showed a solid content of 18% after oven drying. This value was used for calculation of the nominal value.

Tab. 1: Comparison of MC during oven- drying, vacuum- drying and freeze- drying after 7 and 24 hours. Samples were impregnated with water or a resin solution.

		Moisture content after impregnation	WPG*	moisture content	
				after 7 h	after 24 h
		(%)	(%)	(%)	(%)
resin solution impregnated samples	oven-drying	172,7 (±2,6)	31,1	2,3*	1,3*
	vacuum-drying	170,1 (±6,6)	30,6	8,1*	1,5*
	freeze-drying	185,3 (±2,2) 184,5 (±2,7)	33,4 33,2	85,4* -	- 7,7*
water impregnated samples	oven-drying	143,8 (±5,7)	0,4 (±0,1)**	0	0
	vacuum-drying	157,6 (±6,9)		35,5 (±6,7)	1,5 (±0,1)
	freeze-drying	170,6 (±2,7) 126,0 (±2,8)		99,0 (±6,7) -	- 1,6 (±0,1)

* calculated ratio (the dry-matter value of oven dried resin solution was used)

** measured weight loss of volatile extractives (the difference between oven-dry weight before and after impregnation)

In another trial, the effect of MC of wood before impregnation was investigated. The initial moisture content shows little influence on the final WPG of treated wood (Fig. 1).

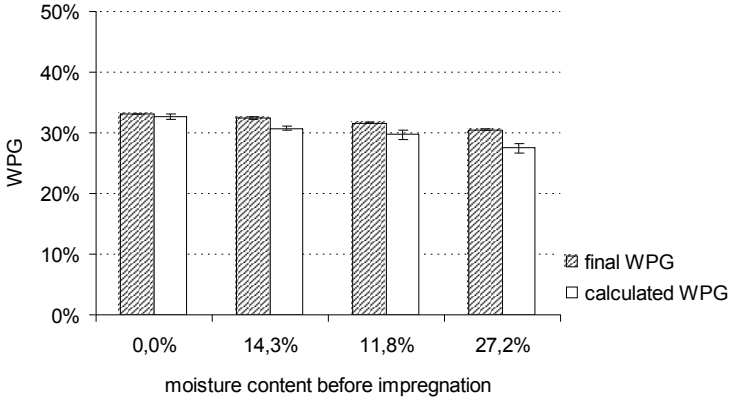

Fig. 1: Influence of an initial MC before impregnation with different concentrations of resin of pine wood samples during oven-drying. To calculate the theoretical WPG the solution uptake was multiplied by the solid content of the impregnation solution. (n = 5)

With increasing the concentration of resin the drying time of freeze drying in modified pine sapwood is increased (Fig. 2). The final MC of the reference sample was 1.6 % after 24 hours. After 24 hours of freeze drying the temperature in the reference sample increased to 0°C. The MC of the larger reference samples at this point was 17%.

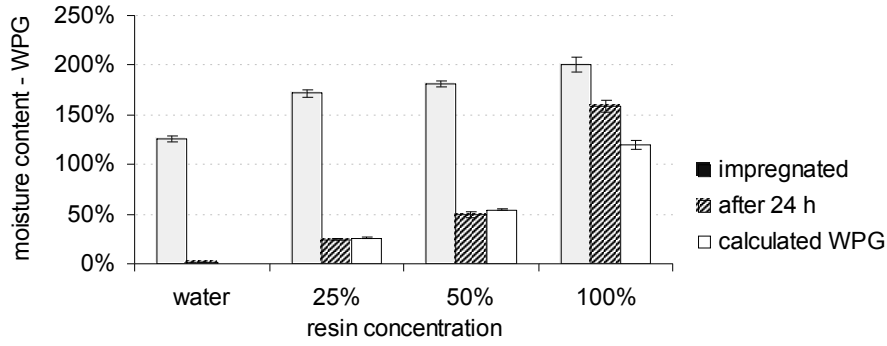

Fig. 2: Moisture content and weight gain of freeze-dried pine sapwood samples after 24 hours. (n = 5)

The moisture content distribution within a sample was determined after freeze-drying for 24 hours. The average MC of the water impregnated sample was 96.5 %. Content of water and resin within pine wood impregnated with resin solution reached 112 %. A high moisture gradient between inside and outside layers of the samples had been proven. In contrary, less moisture gradients were deformed in resin impregnated wood samples (Fig. 3).

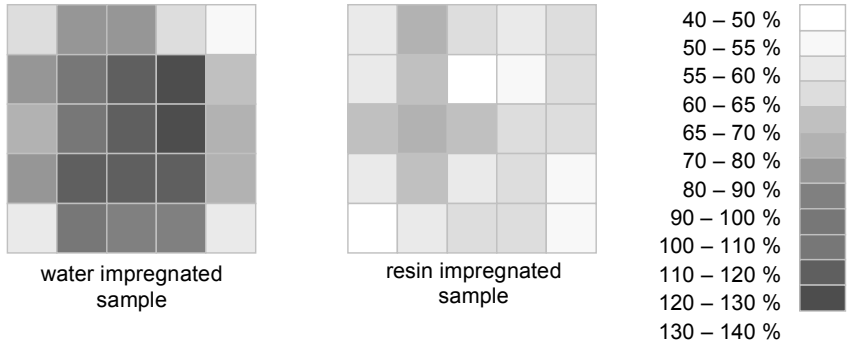

Fig. 3: MC of cross-section samples after freeze-drying of 24 hours. The MC of resin impregnated sample is based on the dry wood plus solid content of resin

DISCUSSION

At 103° C oven drying the drying speed showed to be faster than under vacuum conditions at 50°C. After 24 hours of freeze-drying a final moisture content of 1 – 3 % was reached. This is comparable with the results reported by Matejak et al. (1989), where smaller dimensions (30 x 20 x 5 mm – r x t x l) were used. On the other hand, the temperature of freeze drying was higher (-20 °C) in comparison to this study.

In general, freeze-drying is described as a very slow process. It is depending on the dimensions of wood samples and can be some weeks or longer (Matejak et al. 1989 and Matejak and Jarczyk 1990).

At an average value of 17% MC the sample temperature increased to approx. 0°C during the freeze drying process used in this study. Comparable results shown in previous studies (Matejak et al. 1989) suggest that all water (even at very low temperature) is in a liquid phase below 15% MC during freeze drying. Under these circumstances a sublimation could not take place in own trials and the temperature increased. A vacuum drying process follows a freeze drying process at a temperature about the sublimation phase (Chen and Lamb 2001a, Matejak et al. 1989).

The resin- concentration of wood influences the freeze-drying time. A diluted solution dried two times faster than undiluted solutions because of the differences in the freezing point of the solution.

A different moisture gradient was measured in water impregnated and resin impregnated samples after freeze-drying. In the water impregnated samples a high gradient in MC between the surface (51% MC) and the centre (134% MC) was observed. Unexpectedly, the resin impregnated samples showed a lower gradient between surface (43% MC) and centre (78% MC). The reason for this phenomenon is unclear and will be looked at in further trials.

In this study the vacuum-drying was applied at 50°C at a pressure below 10 mbar. After Chen and Lamb (2002) the MC of app. 2 – 3 % should be reached theoretically as equilibrium moisture content under these vacuum conditions. In spite of this a wood moisture content of app. 0 % was measured after 70 h. To measure the weight of samples it was necessary to interrupt the vacuum and air could access into the drying oven. The long time, which was necessary to reach app. 0% MC can be explained by the higher bound energy of water at a low MC (Skaar 1988).

Leaching of resin out of the samples was observed during vacuum-drying. The difference between the pressure inside the wood and the surface could be an explanation for this leaching (Chen and Lamb 2001b, Taniguchi and Nishio 1993).

The drying of modified wood samples takes longer than drying of untreated wood. This effect could be explained by the bonding between resin-molecules and the water-molecules. Furthermore the resin partly hinders moisture transport by blocking the cell lumina.

The initial MC of wood before impregnation and its influence on the WPG after oven drying was investigated. With an increased MC before impregnation the WPG decreases slightly. The measured WPG exceed the calculated WPG. This difference increased with increasing initial MC. This indicates that a balance of the concentrations in the wood takes part. This could be explained with the diffusion of resin into the wood and a diffusion of water out of the wood.

Pine sapwood consists of about 3% – 4,5% of extractives (Sehlstedt-Persson 2001, Fengel and Wegener 1984). A part of these extractives can be leached out during the impregnation. Measured values during leaching with water and resin while impregnation were between 0,4% and 1% of dry wood mass.

At the used chemical concentration a comparable weight gain was determined by using vacuum drying and oven drying. From this reason it can be stated, that for resin modified wood vacuum drying can be used as an alternative to oven drying for a proper determination of the moisture content of wood. Generally drying of wood at a lower temperature and under vacuum conditions is more sparing.

CONCLUSION

Three different drying methods for determination of wood moisture content of resin modified wood were tested. Oven drying was the fastest method. However, oven dried samples show a tendency to lose mass (up to 1% of WPG) due to evaporation of volatile extractives during drying. Vacuum drying of reference samples takes approx. 7 times longer to reach 0% of MC compared to oven-drying, but without loosing of extractives. Freeze drying works down to app. 17% of MC, when the sample temperature increases below 0 °C and the process change to vacuum drying. The effect of MC of pine sapwood before impregnation shows only little influence on the final WPG of treated wood.

ACKNOWLEDGMENTS

A financial support by the EU "Marie Curie Fellowship" is gratefully acknowledged.

REFERENCES

1. Chen, Z., Lamb, F. M., 2001a: Vacuum drying of small wood components at room temperature. Forest Products Journal 51(10): 55-57
2. Chen, Z., Lamb, F. M., 2001b: Investigation of boiling front during vacuum drying of wood. Wood and Fibre Science 33(4): 639-647
3. Chen, Z., Lamb, F. M., 2002: Theoretical equilibrium moisture content of wood under vacuum. Wood and Fibre Science 34(4): 553-559
4. Fengel, D., Wegener, G., 1984: Wood: chemistry, ultrastructure, reactions. Berlin –New York, Walter de Gruyter, 613 pp.
5. Gillwald, W., Eichler, H., 1964: HF-vacuum drying of sawn timber. Holztechnologie 5(3): 161-167
6. Hartley, I. D., Kamke, F. A., Peemoeller, H., 1994: Absolute moisture content determination of aspen wood below the fibre saturation point using pulsed NMR. Holzforschung 48(6): 474-479
7. Kollmann, F., Höckele, G., 1962: Comparison of procedure for the determination of moisture content in wood. Holz als Roh- und Werkstoff 20(12): 461-473
8. Lee, N. H., Jung, H. S., 2000: Comparison of shrinkage, checking, and absorbed energy in impact bending of Korean ash squares dried by a radio-frequency/vacuum process and a conventional oven. Forest Products Journal 50(2): 69-72
9. Lohse, G., Dietrich, H. H., 1972: Investigations on the suitability of Karl Fischer's Titration method for determining wood moisture content. Holz als Roh- und Werkstoff 30(12): 468-474
10. Matejak, M., Jarczyk, A., 1990: Untersuchungen zum Schwindverhalten von gefriergetrocknetem Holz. Holztechnologie 30(2): 79-81
11. Matejak, M., Niemz, P., Jarczyk, A., 1989: Freeze drying of wood. Przemysl drzewny 40(8-9): 7-10
12. McDonald, A. G., Gifford, J. S., Dare, P. H., Steward, D., 1999: Characterisation of the condensate generated from vacuum-drying of radiata pine wood. Holz als Roh- und Werkstoff 57(4): 251-258

13. Mortimer, Ch. E., 1987: Chemie: Das Basiswissen der Chemie. Georg Thieme Verlag Stuttgart – New York, 660 pp.
14. Neumann, R., Mielke, A., Glos, P., 1993: Moisture transport in wood during convective vacuum drying. Holz als Roh- und Werkstoff 51(5):156-162
15. Resch, H., Ecklund, A., 1963: Moisture content determination for wood with highly volatile contents. Forest Products Journal 13: 481-482
16. Ressel, J. B., 1999: State of the art for vacuum drying in the wood working industry. Cost Action E15, 13-14 October 1999, Edinburgh, Germany
17. Risholm-Sundman, M., Lundgren, M., Vestin, E., Herder, P., 1998: Emission of acetic acid and other volatile organic compound from different species of solid wood. Holz als Roh- und Werkstoff 56(2): 125-129
18. Sehlstedt-Persson, M., 2001: The effect of extractive on moisture diffusion properties for Scots pine and Norway spruce. 3rd European COST E15 Workshop on wood drying: Softwood drying to specific end-uses, 11-13 June 2001 Helsinki, Finland. Pp. 1-10
19. Shmulsky, R., 2000: Influence of lumber dimension on VOC emission from oven-drying lobloly pine lumber. Forest Products Journal 50(3): 63-66
20. Skaar, Ch., 1988: Wood-water relations. Berlin – Heidelberg – New York – London – Paris, Springer – Verlag, 238 pp.
21. Taniguchi, Y., Nishio, S., 1993: High-frequency electric-power vacuum-drying of wood V. Mokuzai Gakkaishi – Journal of Japan Wood Research Society 39(3): 357-362
22. Ward, J. C., Simpson, W. T., 1987: Comparison of four methods for drying bacterially infected and normal thick red oak. Forest Products Journal 37(11/12): 15-22
23. Wittköpper, M., 1998: Current development in the preservation of archaeological wet wood with melamine/amino resins at the Römisch – Germanisches Zentralmuseum. Archäologisches Korrespondenzblatt 28: 637-645

Tomáš Joščák
Faculty of Wood Sciences and Technology
Technical University in Zvolen
T. G. Masaryka 24
SK-96053 Zvolen
Slovakia

Andreas Krause
Institute of Wood Biology and Wood Technology
University of Göttingen
Büsgenweg 4
D-37077 Göttingen
Germany

Andreas Treu
Institute of Wood Biology and Wood Technology
University of Göttingen
Büsgenweg 4
D-37077 Göttingen
Germany

Holger Militz
Institute of Wood Biology and Wood Technology
University of Göttingen
Büsgenweg 4
D-37077 Göttingen
Germany