Dimensional Stability of Uludag Fir Wood Treated with Liquid Nitrogen after the Steam Test

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Abstract
In this study, the effect of liquid nitrogen on dimensional stability of Uludağ fir (*Abies bornmülleriana* Mattf) wood after exposing to the steam test was determined. Test samples were treated with liquid nitrogen (LN) for 1, 4 and 10 hours. Subsequently both samples treated with liquid nitrogen and control samples were conditioned to 12 % MC. Dimensional stability of Uludağ fir wood was measured after exposing to steam for 2, 6, 12, 24, 48, and 96 hours according to the procedure of TS EN 317. Consequently, different values of dimensional stability of specimens treated with liquid nitrogen were observed.
Key Words: Uludağ fir, water steam, liquid nitrogen, dimensional stability

Introduction

Wood is a hygroscopic material, which loses and gains moisture as a result of changes in humidity. Hygroscopic materials such as wood and other lignocellulosic materials can change their dimensions with fluctuations in relative humidity. Based on the current wood moisture content and its surrounding conditions, dimensional changes in wood as a construction material will influence its effectiveness (Beekman, 1964)

The wood cell wall is mainly composed of polymers with hydroxyl and other oxygen-containing groups that attract moisture through hydrogen bonding. As the water is added to the cell wall the wood volume increases nearly proportionally to the volume of added water (Stamm 1964). Swelling increases until the cell wall is saturated with water. This point is called the *fiber saturation point*, and ranges from 20 to 50 percent weight gain (Feist and Tarkow 1967). Beyond this point, the water is free in the void structure and does not contribute to further swelling. This process is reversible, and wood shrinks as it loses moisture below the fiber saturation point (Rowell and Roger 1985).

Wood is dimensionally stable when the moisture content is greater than the fiber saturation point. Wood changes its dimensions as it gains or loses moisture below that point. It shrinks when losing moisture from the cell walls and swells when gaining moisture in the cell walls. This shrinking and swelling can result in warping, checking, splitting, and loosening of tool handles, gaps in strip flooring, or performance problems that detract from the usefulness of the wood product. Therefore, it is important that these phenomena be understood and considered when they can affect a product in which wood is used. With respect to shrinkage characteristics, wood is an anisotropic material. It shrinks most in the direction of the annual growth rings (tangentially), about half as much across the rings (radially), and only slightly along the grain (longitudinally). The combined effects of radial and tangential shrinkage can distort the shape of wood pieces because of the difference in shrinkage and the curvature of annual rings (Wood Handbook 1999).

Jones (1999) has reviewed the literature on wood treatments of and plant fibers using chemicals, pressurized thermal and steam treatments.

In an earlier research by Rowell et al. (2000) it was found that both the rate and extent of thickness swelling could be reduced and the equilibrium moisture content was reduced using the closed steaming process. It was also shown that hemicellulose sugars were lost during the steaming and furan intermediates were formed.

According to Rowell et al. (2002) high temperature steam treatment of wood fiber in a closed press during fiberboard pressing and then cooling the fiberboard while still under pressure below the glass transition temperature of lignin greatly increased the dimensional stability and decreased the hemicellulose content of the fiberboards produced. For example, after pressing aspen fiber four minutes at 200^{o}C under steam pressure, the resulting fiberboards show less than 10 percent thickness swelling after two-hour water soaking as compared to over 40 percent for non-steamed fiberboards. The addition of ferric chloride to the process greatly increases dimensional stability and reduces the hemicellulose content lower than control fiberboards.

According to Rowell et al. (2002) high temperature steam treatment of wood results in several changes in the wood chemistry. These changes include: (a) degradation of the hemicelluloses to produce simple sugars which may undergo reversion reactions to form highly branched polysaccharides, (b) degradation of both hemicelluloses and part of the cellulose to form furan-type compounds, (c) thermal softening of the cell wall matrix, mainly lignin, (d) degradation of the hemicelluloses to form volatile break down products, (e) cross-linking between carbohydrate polymers and/or between lignin and carbohydrate polymers, and/or (f) an increase in cellulose crystallinity. While no definitive mechanism has been published, there are clues in the literature to support one or more of the above theories.

The research reported in this paper was done to treat wood fiber, in a closed press, with high temperature steam to (a) degrade the hemicelluloses in order to decrease hygroscopicity and equilibrium moisture content, (b) generate furan monomers that could form polymers for increased internal bond strength, (c) thermoplastaicize the lignin matrix and reform it in a cool down process to generate a new collapsed unstressed matrix, and (d) produce a fiberboard with greatly increased dimensional stability (Rowell et al. 2002).

There is no literature study on applying LN to wood materials. It was the first study to use LN in wood.

The purpose of this study was to investigate the effect of liquid nitrogen on dimensional stability of Uludağ-fir wood after exposing to the steam test.

Material and Method

Wood Species

Uludağ fir (*Abies bornmülleriana* Mattf) was chosen randomly. A special emphasis was put on the selection of wood material. Accordingly, non-deficient, proper, knotless, normally grown (without zone line, reaction wood, decay, insect and fungi damages) wood materials were selected.

Liquid nitrogen

Nitrogen gas composes 78% of the Earth's atmosphere. It is a colourless, odourless and non-flammable gas. It is used in the electrical industry, producing chemicals safely, the food packaging industries and in the drying and preparation of refrigeration systems. At temperatures below -196 ^{o}C, nitrogen is liquid. When liquid nitrogen comes into contact with objects at room temperature it boils rapidly from the heat energy emitted by the objects.

Test performance

All test samples (20x100x100 mm) were equilibrated in a climate chamber at relative humidity 65% and temperature 20 °C before exposing to liquid nitrogen (LN). Later, test samples were exposed to the LN tank (Figure 1) for 1, 4 and 10 hours. After each exposing time, test samples were re-conditioned to 12% MC. Then, samples were put in a test equipment 60 cm diameter and 120 cm length containing 49±2 ^{o}C and 85±3 relative humidity. For testing the dimensional stability, test samples were exposed to steam for 2, 6, 12, 24, 48 and 96 hours according to the procedure of TS EN 317. The measurement points of test samples are shown in Figure 2. After each period the test samples were measured then re-exposed to the steam test. Radial (thickness) swelling was measured from 4 different points and their average value was recorded as a single value.

Fig. 1. Liquid nitrogen tank

Data Analyses

There were used three different exposing liquid nitrogen times, six different exposing steam test times, and 15 repetitions for each factor. For control samples, five repetitions for each factor were used; a total of 350 samples (3×7×15+5x7) were prepared for the steam test.

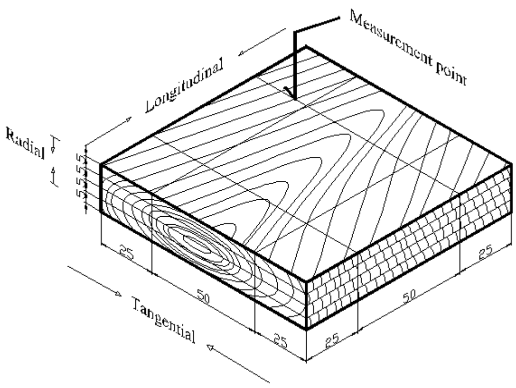

Fig. 2. Steam test sample (mm)

Results and Discussion

The average air-dry density of Uludağ fir samples is 0.40g/cm^3. The average dimensional stability values are shown in Table 1.

The width, thickness and weight increments value of control samples at 96 hours steaming time were found 6.08 %, 2.85% and 67.21% respectively. After 1 hour exposing to LN; width, thickness and weight increments value versus control samples at 96 hours steaming time were 5.07%, 3.27%, and 59.20% respectively. The width, thickness and weight increments value versus control samples at 96 hours steaming time exposed to 4 hours LN were 5.11%, 5.03%, and 51.03% respectively. The width, thickness and weight increments value versus control samples at 96 hours steaming time exposed to 10 hours LN were 4.64%, 5.34%, and 51.07%, respectively.

The multiple variance analysis applied to the data obtained from the steam test is shown in Table 2.

Table 1. The average dimensional stability values

Experimental conditions		Width		Thickness		Weight	
Liquid nitrogen time (hours)	Steam time (hour)	Mean (%)	Std. Dev.	Mean (%)	Std. Dev.	Mean (%)	Std. Dev.
Control (non-LN)	2	0.62	0.30	0.42	0.32	0.41	0.13
	6	1.20	0.34	1.99	0.38	2.45	0.11
	12	1.43	0.25	1.64	0.36	3.37	0.13
	24	4.43	0.23	2.57	0.44	10.45	0.26
	48	5.69	0.23	3.12	0.44	38.45	0.31
	96	6.08	0.39	2.85	0.32	67.21	0.24
1	2	0.66	0.29	0.70	0.31	1.68	0.13
	6	2.65	0.37	2.30	0.35	8.53	0.32
	12	2.52	0.34	2.13	0.26	6.51	0.39
	24	4.22	0.28	2.66	0.35	11.15	0.25
	48	5.07	0.29	3.13	0.24	27.41	0.41
	96	5.07	0.22	3.27	0.26	59.20	0.28
4	2	0.60	0.30	2.28	0.31	3.36	0.25
	6	0.23	0.33	0.25	0.30	9.91	0.48
	12	1.42	0.33	3.47	0.30	13.58	0.49
	24	3.52	0.20	4.60	0.29	13.58	0.29
	48	5.18	0.31	4.98	0.16	30.46	0.44
	96	5.11	0.22	5.03	0.23	51.03	0.60
10	2	0.50	0.30	2.22	0.33	1.71	0.20
	6	1.02	0.40	2.50	0.30	3.76	0.18
	12	1.29	0.29	3.33	0.26	5.52	0.19
	24	3.47	0.24	4.69	0.32	10.98	0.26
	48	4.87	0.34	5.35	0.26	32.28	0.60
	96	4.64	0.23	5.34	0.22	51.07	0.62

Table 2. The multiple variance analysis values

Source	Type III Sum of squares	df	Mean square	F	Sig.
Corrected model	57.209[a]	27	2.119	23.839	0.000
Intercept	115173.906	1	115173.906	1295836.632	0.000
LN time	3.851	3	1.284	14.441	0.000
Steam time	44.430	6	7.405	83.314	0.000
LN time * Steamtime	1.460	18	0.081	0.913	0.564
Error	28.619	322	0.089		
Total	143759.618	350			
Corrected total	85.828	349			

a. R squared = 0.667 (Adjusted R squared = 0.639)

According to the variance analysis, the effects of LN time and steam time were statistically significant. The two-way interaction between LN time and steam time was not statistically significant. The interaction between factors was statistically identical ($p \leq 0.05$).

Conclusion

The width, thickness and weight increments value of control samples at 96 hours steaming time were 6.08 %, 2.85% and 67.21% respectively. After 1 hour exposing to LN, width, thickness and weight increments value versus control samples at 96 hours steaming time were 5.07%, 3.27%, and 59.20% respectively. The width, thickness and weight increments value versus control samples at 96 hours steaming time exposed to 4 hours LN were 5.11%, 5.03%, and 51,03% respectively. The width, thickness and weight increments value versus control samples at 96 hours steaming time exposed to 10 hours LN were 4.64%, 5.34%, and 51.07% respectively.

Consequently, while LN and steam time are increased, retention amounts were enhanced.

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