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THE INFLUENCE OF MASS LOSS ON THE MECHANICAL PROPERTIES OF HEAT-TREATED BLACK PINE WOOD

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ABSTRACT

Heat has been a method used to dry and modify the properties of wood since ancient times. Nowadays, heat is used as industrial processes for the same reason. Treatment at temperatures above 150°C can change the color of wood, improve its resistance to biodegradation, and enhance its dimensional stability. However, loss in the mechanical strength of wood may also occur, and this means heat-treated wood cannot be used in a broad range of products. This article investigates the mass loss, specific gravity, compression, and bending-strength of heat-treated Camiyani Black Pine wood at temperatures of 160°C, 180°C and 200°C for 2 and 6 hours. The results show that increasing temperatures decreased specific gravity, compression, and bending strength of the specimens, whereas mass loss increased.

KEY WORDS: Camiyani black pine, heat treatment, physical and mechanical properties

INTRODUCTION

Heating wood to preserve it is an alternative to using chemical treatments. The heat-treatment process improves wood's resistance to decay and changes some of its physical properties. Nowadays, many methods are used for this process. One of these methods is to heat the wood at temperatures above 180°C and normal pressure (Viitaniemi 1997a).

The change in properties is mainly caused by the thermal degradation of hemicelluloses. Theoretically, the available hydroxyl groups in hemicelluloses have the most significant effect on the physical properties of wood. Heat treatment slows water uptake, and the wood's cell walls absorb less water because of the decrease in the number of hydroxyl groups in the group. A decrease in swelling and shrinking is a consequence of the reduced number of hydroxyl groups. In addition to better durability, the advantages of heat-treated wood are reduced hygroscopicity and improved dimensional stability. It equilibrates around 4–5% moisture content instead of 10–12% (Inoue et al. 1993). In addition, heat treatment caused various amounts of weight loss, depending on the treatment temperature and time. For spruce (*Picea abies* L.), a 24-hour heat-treatment resulted in 0.8% and 15.5% weight loss at 120°C and 200°C, respectively. Weight loss of beech (*Fagus sylvatica* L.), treated at increasing temperatures, was 8.1% and 9.8% at 150°C and 200°C, respectively (Fengel and Wegener 1989). Due to heat-treatment and thermal degradation, wood loses weight. Weight loss depends on the heat-treatment temperature and time, size of the specimen, and prevailing circumstances during the heat-treatment. The main reasons for industrial heat-treatment are increased biological durability, enhanced weather resistance, and decreased shrinking and swelling of wood (Hakkou et al. 2005, Jämsä et al. 2000, Kamdem et al. 2002, Repellin and Guyonnet 2005, Santos 2000).

A study showed that heat treatment decreased the water absorption of Uludag fir, as predicted. The reduction of the equilibrium moisture content of Uludag fir strongly depends on the heat-treatment temperature: the higher the heat-treating temperature, the lower the level of absorbed moisture and EMC. Heat treatment of wood results in a loss of dry mass, which causes the wood to shrink (Aydemir 2007, Gunduz et al. 2008).

According to Chang and Keith (Chang and Keith 1978) there is a strong correlation between loss of dry mass and volumetric shrinkage. They also showed that the anisotropy of the shrinkage is similar to the shrinkage due to heat treatment and thermal degradation. A lower equilibrium moisture content, darkened hue, and increased thermal insulation are welcome changes in the properties of wood following heat-treatment. On the other hand, as a result of heat-treatment, the wood becomes more brittle, mechanical strength and hardness decrease in relation to the level of heat-treatment (Bekhta and Niemz 2003, Sailer et al. 2000, Viitaniemi 1997b).

Scots pine (*Pinus sylvestris*) and Norway spruce (*Picea abies* L.) are the main species used for industrial-scale heat treatment in Finland, but birch, aspen, alder, and other species are heat-treated. Heat-treated wood has a growing market in outdoor applications like exterior cladding, window and door joinery, garden furniture, and decking. There are also many indoor applications for heat-treated wood such as flooring, paneling, kitchen furnishings, and interiors of bathrooms and saunas (Viitaniemi 2000). Because it loses strength, heat-treated wood is not recommended load-bearing construction (Viitaniemi 1997b).

The purpose of this study was to determine how heat-treatment affects specific gravity, mass loss, bending, and compression strength of Camiyani Black pine wood. Camiyani Black pine is a species of naturally grown and intensively used in the forest product industry in Turkey. Improving the characteristics of Camiyani Black pine through heat treatment would offer the timber product industry many interesting opportunities.

MATERIAL AND METHODS

Camiyani Black pine (*Pinus nigra* Arn. subsp. *pallasiana* var. *pallasiana*) grows naturally in the Yenice region of Turkey. Studies have determined that the heartwood percentage for Camiyani Black pine is more than 50% in comparison to the sapwood grown in the Yenice region. Camiyani Black pine grows naturally in the Elekdag, Dursunbey, Tosya, Daday, Tavsanli, and Yenice regions of Turkey, which encompass a total area of 30,000 ha. The average habitat altitude is 866 m, and the tree prefers calcareous and sandy-clay soils. A primary forest tree species with an average height of 30 m, it rarely reaches 50 m (Gunduz 1999).

The study material were obtained from a local sawmill in Yenice, Karabuk, Turkey. Sampling methods and general requirements for the physical and mechanical tests were carried out based on ISO 3129. Small clear samples were obtained for specific gravity and mass loss (20 x 20 x 30 mm), compression strength (20 x 20 x 30 mm), bending strength (20 x 20 x 360 mm).

The samples were subjected to heat treatment at 160°C, 180°C, and 200°C for 2 and 6 hours in a small heating unit controlled to within ±1°C under atmospheric pressure. After heat-treatment, treated and control samples were conditioned at 20 ± 2°C and 65% (±5%) relative humidity (RH) in a conditioning room to reach an equilibrium moisture content (EMC) of 12%.

Tests for specific gravity, compression-strength and bending-strength were carried out based on ASTM D2395-07, TS 2595 and TS 2474, respectively. After mechanical strength tests, the moisture content of the samples was measured according to ISO 3130 and strength values were corrected based on 12% EMC. The mass loss after heat treatment was determined on a dry mass basis, after oven- drying at 103°C for 24 hour

The variance analysis method was used to analyze the results in this study. All statistical calculations were based on a 95% confidence level. ANOVA and Tukey Multiple Range Tests show that all differences were significant.

RESULTS

Tab. 1 shows the changes in mass loss, specific gravity, compression and bending strength at various treatment temperature and durations. ANOVA and Tukey's Multiple Range Tests showed that there were significant differences between groups.

According to the average values, all the parameters decreased with increasing temperature and time. It is evident from the results that these values were all lower in heat-treated samples than in control samples. It is clear from this study that the value of all measured specific gravity and compression and bending strength decreased with increasing temperature and heat-treatment time under the conditions used. It was observed that the changes in properties of heat-treated wood were significant, according to a variance test and Tukey's multiple-range tests at $p < 0.05$.

Tab. 1: Statistical values and result of Mass Loss, specific gravity, Bending and Compression Strength of Camiyani black pine wood subjected to heat treatment at varying treatment temperature and durations

	Treatment Durations (h)	Statistical Values	Mass Loss (%)	Specific Gravity	Compression Strength ($N.mm^{-2}$)	Bending Strength ($N.mm^{-2}$)
Control	-	*Avg.*		0.54A	60A	112A
		±s	-	0.01	3.29	17.65
		n		30	30	20
160°C	2	*Avg.*	0.6A	0.54A	56B	109A
		±s	0.04	0.02	3.98	23.48
		n	30	30	30	20
	6	*Avg.*	2.6AB	0.54A	51C	86CD
		±s	0.07	0.01	5.26	15.29
		n	30	30	30	20
180°C	2	*Avg.*	0.6AB	0.54AB	55B	101AB
		±s	0.01	0.02	2.46	13.51
		n	30	30	30	20
	6	*Avg.*	3.5ABC	0.52BC	49C	79D
		±s	0.02	0.02	6.31	12.11
		n	30	30	30	20
200°C	2	*Avg.*	2.9BC	0.53BC	52B	96BC
		±s	0.07	0.02	1.73	18.46
		n	30	30	30	20
	6	*Avg.*	4.5C	0.51BCD	45D	76D
		±s	0.12	0.02	3.14	20.30
		n	30	30	30	20

Avg., Average; s, Standard Deviation; n, number of samples used in each test is 10, each column indicate groups that are statistically different according to Tukey's multiple range tests at $P < 0,05$. Comparisons were between each control and its test.

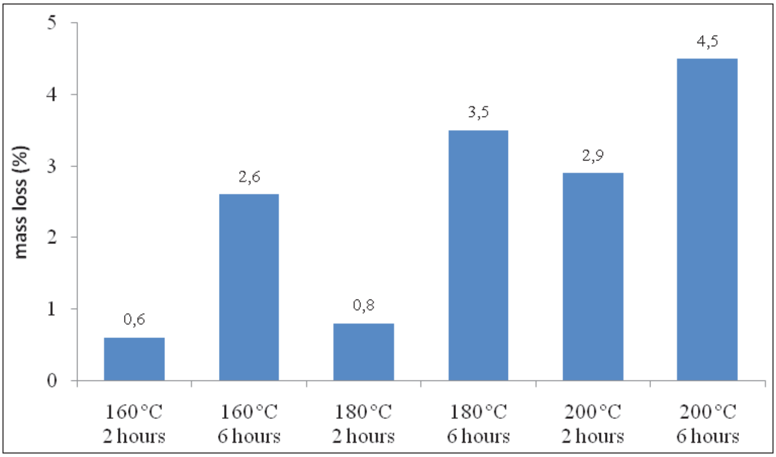

Fig. 1: Changes of mass loss with treatment temperatures and durations

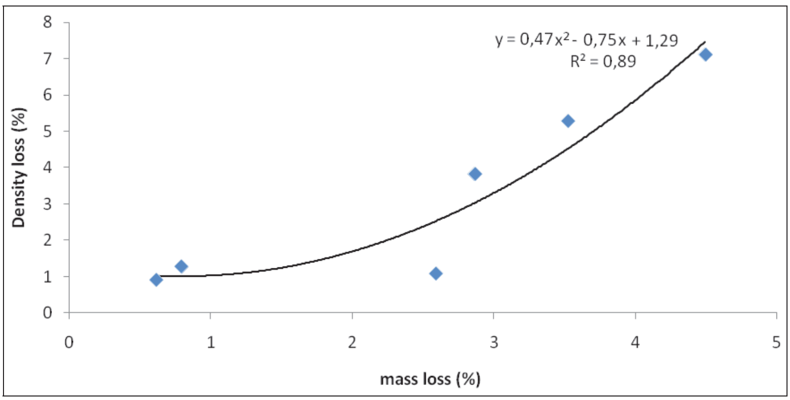

Fig. 2: The effect of mass loss on specific gravity of Camiyani black pine wood after heat treatment

According to Fig. 1, the mass loss of Camiyani Black pine wood increased with increasing treatment temperatures and times. Heating of wood results in a reduction in mass and a decrease in volume, the extent of which is dependent upon the treatment method, temperature, and time of exposure.

Mass loss was determined to be 0.61% and 2.59% after heat treatment of Camiyani Black Pine at 160°C for 2 and 6 hours. This increase in mass loss was due to evaporation at 160°C. Mass loss at 180°C for 2 and 6 hours was 0.79% and 3.52%. The mass loss at maximum heat-treatment temperature was 2.86% for 2 hours and 4.49% for 6 hours. Mass loss during the heat treatment was increased at temperatures of 160°C and 180°C. However, it decreased at temperatures between 180°C and 200°C. Mass loss at temperatures between 160°C and 180°C is caused by evaporation of water (bound water and free water) and the release of different byproducts such as hemicelluloses during the degradation of the wood components, but mass loss at temperatures between 180° and 200°C caused byproducts only during the degradation of the wood components since the water (bound water and free water) is fully evaporated.

Also, the studies showed that mass loss is related to treatment temperature, time and heating rate, wood species, specimen size, and treatment methods. Specific gravity of heat-treated Camiyani Black pine wood was lower than that of untreated wood. This is mainly due to the changes of the sample mass during the treatment when wood loses its weight.

According to Fig. 2, specific gravity was decreased in conjunction with increasing heat treatment temperatures and times. The smallest decreases were 0.543 and 0.542 at treatment temperatures of 160°C for 2 and 6 hours.

The largest decreases were 0.519 and 0.509 at 180°C for 6 hours and 200°C for 6 hours. The minimum and maximum decrease rate of specific gravity is 0.928% at 160°C for 2 hours and 7.66% at 200°C for 6 hours. The decreasing specific gravity is mainly related to mass loss, so increasing mass loss of heat-treated wood simultaneously affects both specific gravity and mechanical properties of wood.

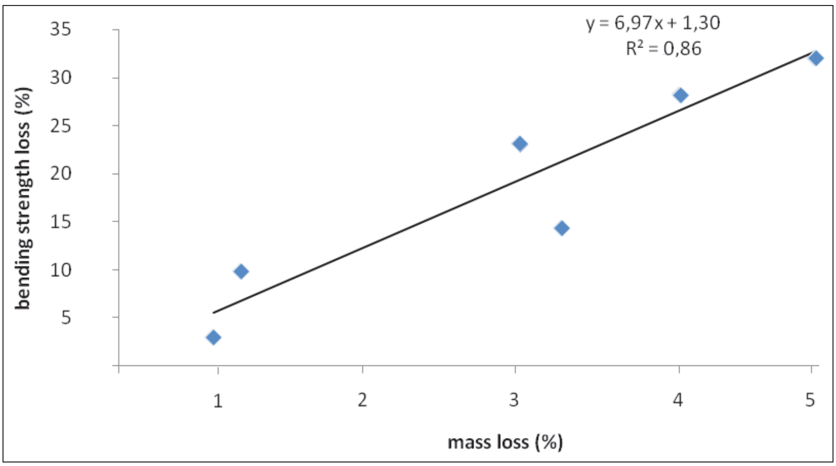

Fig. 3: The effect of mass loss on bending strength after the heat treatment

The bending strength decreased at increased heat-treatment temperatures and times (Fig. 3). This is probably due to the breakup of the hemicelluloses and cellulose polymers. The bending strength of heat-treated Camiyani Black pine decreased because of increasing mass loss and decreasing specific gravity.

The bending strength showed a slight decrease with increased temperature and treatment time. There was a linear trend in the decrease in bending strength for Camiyani Black pine wood as time and temperature increased. Bending strength was 108.69 $N.mm^{-2}$ and 86.23 $N.mm^{-2}$ after heat-treatment of Camiyani Black pine wood at 160°C for 2 and 6 hours. Bending strength in wood treated at 180°C for 2 and 6 hours is 101.02 $N.mm^{-2}$ and 79.11 $N.mm^{-2}$. The bending strength at maximum heat-treatment temperature is 95.92 $N.mm^{-2}$ in wood treated for 2 hours and 76.17 $N.mm^{-2}$ in wood treated for 6 hours. Therefore, it can be said that temperatures might have greater influence on strength properties than time.

The largest decreases were 76.17 $N.mm^{-2}$ and 79.11 $N.mm^{-2}$ in wood treated at 200°C for 6 hours and 180°C for 6 hours. The minimum and maximum decrease rate of bending strength is 3.2% in wood treated for 2 hours at 160°C and 47.2% in wood treated at 200°C for 6 hours.

The lowest bending strengths for beech and spruce were observed when the wood samples were treated at 200°C for both 6 hours and 10 hours. The decrease was 63.9% and 63.6% for beech and 63.8% and 72.7% for spruce in samples treated at 200°C for 6 hours and 10 hours.

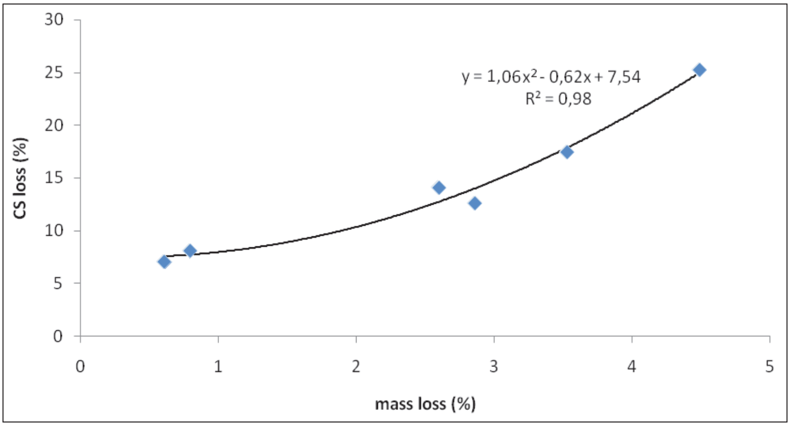

Fig. 4: The effect of mass loss on compression strength of Camiyani Black pine after heat treatment

As shown in Fig. 4, the compression strength decreased slightly as temperature and treatment time increased. With heat-treatment conditions at 200°C for 2 and 6 hours, the decrease in compression strength was 12 to 25%; at 180°C for 2 and 6 hours; it was 7 to 15%; and at 160°C for 2 and 6 hours it was 7 to 14%. The maximum decreases for all parameters were recorded at a temperature of 200°C for 6 hours. The lowest and highest compression strength values obtained after heat-treatment were 44.773 $N.mm^{-2}$ for 200°C and 6 hours and 55.659 $N.mm^{-2}$ for 160°C and 2 hours. The maximum loss ratio compared to the control was calculated to be 33.56%. Also, the minimum compression strength parallel to grain ratio decreased 7.5% in Camiyani Black pine (*Pinus nigra* Arn. *Pallasiana* subsp. *pallasiana*) wood samples at 160°C in a 2 hour treatment.

Wood samples treated at a temperature of 200°C for 6 hours showed the lowest specific gravity, compression strength, and the highest mass loss when compared with other conditions studied. The maximum specific gravity, mass loss, and compression strength decreases for all parameters were recorded at a treatment temperature of 200°C for 6 hours.

Based on the findings in this study, compression strength values decreased with increasing treatment temperatures and treatment times. Increase in temperature and duration further diminished compression strength values of the wood specimens.

DISCUSSION

The mass loss starts to evaporate at lower temperatures. However, this phenomenon becomes significant above 100°C. At high temperatures, the physical bonds between water and the hydroxyl groups of the wood are broken, which facilitates the movement of water. This phenomenon accelerates the mass loss rate with increasing temperature in this region, but the quantity of water in the wood is limited. After a maximum rate is reached, the mass loss slows down as water evaporates. Acceleration of the mass loss observed is probably caused by the release of different by-products during the degradation of wood components such as hemicelluloses (Poncsak et al. 2006).

Heating of wood results in a reduction in mass and a decrease in volume, the extent of which is dependent upon the treatment method, temperature, and time of exposure (Stamm 1956).

The diminutions in the specific gravity were related to the rate of thermal degradation and losses of substance after heat treatments. The decrease is mainly due to the de-polymerization reactions of wood polymers (Fengel and Wegener 1989).

The primary reason for the loss of specific gravity is the degradation of hemicelluloses, which are less stable to heat than cellulose and lignin. Changes in or loss of hemicelluloses play key roles in the physical properties of wood heated at high temperatures (Aydemir 2007).

Yildiz (2002) reported that the lowest bending strengths for beech and spruce were observed when the wood samples were treated at 200°C for both 6 and 10 hours. The decrease was 63.9% and 63.6% for beech and 63.8% and 72.7% for spruce, both treated for 6 and 10 hours at 200°C.

Korkut (2008) reported for Uludag fir that the lowest bending strength values were obtained in samples treated at 180°C for 10 hours (92.914 $N.mm^{-2}$). The bending strength loss compared to the control was 32.68%.

The heat-treated wood becomes more brittle, and bending and tension strength decrease by 10 to 30%. Therefore, the use of heat-treated wood in load-bearing construction is restricted (Jämsä et al. 2000).

Korkut (2008) obtained similar compression strength results for Uludag fir (*Abies bornmulleriana* Mattf.) for the same treatment time and temperature. In this case it can be said that temperature has greater influence on strength properties than time. Similarly, another study showed that for Eucalyptus (*Eucalyptus saligna*), compression strength parallel to grain decreased at 110 to 155°C after 10 to 160 hour treatments (Vital and Lucia 1983).

Gunduz et al. (2008) found that the maximum decreases for all parameters were recorded with a treatment at 180°C for 10 hours. The lowest compression strength value obtained was 41.432 $N.mm^{-2}$ total loss compared to the control was calculated to be 27.2%. Unsal and Ayrilmis (2005) also found that the maximum compression strength parallel to grain decrease in Turkish river red gum (*Eucalyptus camaldulensis* Dehn.) wood samples was 19.0% at 180°C after a 10-hour treatment.

CONCLUSION

In conclusion, it was determined that specific gravity, compression strength parallel to grain decrease, and bending strength of the Camiyani Black pine decreased for all treatment conditions (temperatures and times), but mass loss increased after heat-treatment.

With increasing treatment temperatures and durations, the mass loss of Camiyani Black pine increased. Heating of wood results in a reduction in mass and a decrease in volume, the extent of which is dependent upon the treatment method, temperature, and time of exposure. The largest decreases in specific gravity were 0.519 and 0.509 at 180°C for 6 hours and 200°C for 6 hours, respectively. The minimum and maximum decrease rate of specific gravity is 0.928% at 160°C for 2 hours and 7.66% at 200°C for 6 hours. The decrease is mainly due to mass loss. The higher mass loss increased, the more density decreased. The smallest decrease in compression strength was observed after treatment at 160°C for 2 hours; also, the largest decrease was measured after treatment at 200°C for 6 hours. The highest decrease ratios of the compression strength were 30% and 35% when the treatment temperature was 200°C for 6 hours. A significant loss in compression strength was not observed for the samples treated below 200°C. A clear reduction in compression strength can be found in wood treated at a temperature of 200°C and above.

The bending strength showed a slight decrease with temperature and treatment time. There was a linear trend in the decrease in bending strength as treatment time and temperature increased for Camiyani Black pine wood. Wood treated at high temperatures and for long durations should not be used for load-bearing purposes for the time being. Camiyani Black pine wood could be utilized if proper heat treatment techniques were used without any losses in strength values for such things as window frames, where strength is an important factor.

Camiyani Black pine wood is one of the most desirable softwood species in Turkey, and its hue darkens over time. The desired hue is generally reached after five years. In this study, the desired hue of Camiyani Black pine is obtained when it is treated at 180°C for 2 hours, and minimum mass loss and loss of bending and compression strength also °C ocurred. It is clearly indicated that heat treatment can be used to modify the hue of Camiyani Black pine wood.

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