Effects of heat treatment of small clearwood samples on equilibrium moisture content and deformation

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ABSTRACT

High Temperature drying has by numerous studies been shown to affect strength properties as well as moisture properties of the wood treated. The present study aims at determining the effects of different heat treatments of small clearwood samples on material properties. Samples were treated in dry and moist air, saturated steam and water. Temperatures varied from 20 °C to 150 °C. Treatment periods varied from 6 h to 96 h. The results of the study indicate that the effects of treatment are dependent not only on the method of treatment and time, but also on the pre treatment of the samples such as ingoing moisture content.

INTRODUCTION

Strength properties of Norway spruce planks after industrial drying in elevated temperatures have been studied in a series of projects at SP Technical Research Institute of Sweden (Betzold, 1999), (Bengtsson and Källander, 2001), (Imbaud, 2001), (Källander et al. 2001), and (Dahlberg, 2002). Laboratory studies on clearwood samples were then initiated to investigate degrade mechanisms at elevated temperatures. As expected the laboratory studies on clearwood samples showed different results as compared to the full size planks (Källander and Bengtsson, 2003) and (Landel, 2004). It is evident that the result of strength tests on small clearwood samples will rely on different material properties than corresponding tests on planks, were the stresses around local defects will determine the result. However, it can also be expected that the kiln drying climate will lead to different internal wood climate in a plank than in a small sample, and thus influence the wood material properties differently.

The combined effect of the factors above is shown in Figure 1 where the results from tensile tests at SP of full size planks after High Temperature (HT) drying are compared with tests on small clearwood samples as reported by Stamm, 1953. The "loss of tensile strength" is 100 - 1000 times faster in the planks than in the clearwood samples.

Figure 1. Heating time versus temperature to reach various degrees of wood degradation. Solid line corresponds to 10% loss of modulus of rupture on dry clearwood samples. The square marks the time and temperature range resulting in similar strength loss in planks in the SP studies (Källander and Bengtsson, 2003).
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Chemical processes causing degrade of wood material

Fengel and Wegener, 1989 describes three different chemical processes that will lead to degrade of wood at elevated temperatures: 1) hydrolysis, 2) dehydration, and 3) oxidation.

1) Hydrolysis is generally accepted as the main cause of degrade during HT treatment of wood. During hydrolysis the polysaccharide is broken as water is bound to the molecule. It is obvious that hydrolysis requires water, and that the total mass of the polymer (parts) will increase as the water molecule is added. Hydrolysis is accelerated in acidic environment (Fengel and Wegener, 1989), and hence accelerated by organic acids like acetic acid and formic acid formed during the hydrolysis. The organic acids will act as catalysts for the hydrolysis, but since acetic acid and formic acid are highly soluble in water, part of the acids can evaporate together with the moisture.

The main part of the resulting degrade compounds are shorter saccharides. These saccharides are not volatile and will not evaporate from a solid piece of wood during drying. However, if the hydrolysis is carried out on sawdust in an acidic solution, a large portion of the reaction products can be expected to be extracted with reduced total mass as a result. During a drying process of sawn planks water will always be present, either as bound or free water or as water vapour or steam. On the other hand, small samples quickly dries out in high temperatures, resulting in a dry treatment process. It is clear that the size of the sample treated and the treatment agent will influence the result.

2) Dehydration implies that the polysaccharide is broken as water molecules are released (Fengel and Wegener, 1989). Dehydration does not require presence of water since the water is produced in the process. When dehydration takes place at temperatures above 100 °C, the water molecules produced will evaporate from the wood in the form of water vapour or steam, leading to a reduction of the mass of the sample treated.

3) Oxidation reactions occur in parallel with hydrolytic degradation during for instance acidic pulping (Fengel and Wegener, 1989). Oxidation leads by definition to an increase in molecule weight. The sample size and treatment atmosphere can be expected to have great influence. If oxidation has a significant effect on wood properties after drying is not known to the authors.

MATERIAL AND METHODS

A total of 1108 clear wood samples from Norway Spruce and Douglas-fir for heat treatment were prepared from the sapwood of taper sawn 60 mm Norway spruce planks (576 samples) and a 0.55 m diameter Douglas fir log (532 samples). Sample dimension 40 mm x 40 mm x 40 mm. All samples were conditioned in standard climate 20 °C / 65 % RH to approximately 12 % MC and density of the samples determined.

The samples were divided into sets of 10 samples with corresponding average density. Two sets from each wood species were used for each thermal treatment, or 20 samples per species. The samples were exposed to elevated temperatures in moist air, in steam, and in hot water for times ranging from 6 h to 96 h. Temperatures ranged from 95 °C to 125 °C. Treatments were done with samples impregnated with water (MC at start 60 % - 150 %) as well as conditioned samples (MC at start 12 %).

Four varieties of treatments were used: 1) moist air treatment, 2) saturated steam treatment, 3) hot water treatment with free samples and samples wrapped in aluminium foil to reduce extraction of reaction products from the samples, and 4) hot water treatment followed by moist air treatment.

The moist air treatment was done to simulate the wood climate during the diffusion phase of the drying process and the wood surface will be exposed to water vapour. The treatments were done in closed chambers were steam from boiling water was continuously fed into the chamber. Saturated steam treatments were done to simulate the capillary phase. The treatments were done in a pressurized vessel with samples placed above boiling water and excess steam evacuated by a pressure valve. Hot water treatments were made to simulate the climate inside the evaporation front of the wood. The treatments were made in 20 litre stainless steel cauldrons.
Kiln climate and internal wood temperature were recorded during treatments. After treatments samples were inspected, weighed and reconditioned in 20 °C / 65 % RH before shear testing and energy of rupture testing. The wood samples were stored more than 2 years in 20 °C / 65 % RH before the final MC was determined by dry weight measurement.

**RESULTS ON EMC**

The results show a drop in average EMC of the samples during treatment from 11.8 % to 10.6 % for Douglas and from 12.5 % to 11.2 % for Spruce. The drop in EMC seems to happen early in the treatment process. Figure 2 shows EMC after treatment in humid air at 105 °C for different periods.

The results show a faster drop in EMC during treatment in humid air as compared to saturated steam, see Figure 3. The total EMC drop in humid air seems not to be influenced by the treatment temperature at 105 °C and above, the final MC is approximately 9 % for Douglas samples and 10 % for spruce. Treatment in saturated steam shows a slower effect on the EMC, and a increase with temperature. Water treatment in temperatures up to 100 °C show no significant effect on average EMC in this study.
The temperature measurements during treatment show that the internal wood temperature rapidly increases to the level of the treatment climate, indicating that all or most of the initial moisture in the samples has been removed. Figure 4 shows temperature readings from treatment of water impregnated samples in 125 °C.

![Figure 4. Temperature development during treatment in 125 °C humid air. Internal wood temperature shows that samples have dried out after approximately 1h 30 min.](image)

**Results on deformations and internal checking**

The initially cubical samples were generally more or less deformed into rhombic shape during treatment due to the anisotropy of the wood. Several of the samples that first were boiled and then immediately humid air treated showed radial checking and deformations as if there was collapse in the interior parts. These samples also showed internal radial checking when the samples later were split. Figure 5 shows radial checking in the earlywood of Douglas samples boiled for 12 h followed by drying 12 h in 125 °C.

![Figure 5. Radial checking in earlywood of Douglas samples boiled 12 h and dried 12 h in 125 °C.](image)
DISCUSSION

The study has resulted in a significant reduction of EMC after heat treatment. This confirms that the methods of heat treatment used have influenced the chemical composition of the wood.

The results seem to show a rapid reduction in EMC of the samples during the early stages of the temperature treatment, after which the rate of EMC reduction is greatly reduced or halted. At the same time the temperature readings show that the internal wood temperature rapidly increases above temperatures where free water can be present, and within 2 hours reaches the surrounding temperature. As the wood temperature is at the same level as the surrounding atmosphere, little or no evaporation can occur. The wood is dry. As the moisture of the wood is removed, the rate of EMC reduction is greatly reduced.

The lower reduction rate in EMC during treatment in saturated steam is somewhat unexpected. Earlier studies have indicated a higher effect on wood properties from treatment in steam as compared to air (MacLean, 1953 and Stamm, 1956). The results in this study can possibly be linked to the water impregnation of the samples prior to treatment, by which the air treated wood has been supplied with a reservoir of water for quick hydrolysis. A different pattern could have shown if the treatment periods with steam had been longer. Then it is possible that the EMC would have continued to be further reduced. The treatment periods and temperatures in this study have been limited to drying schedules used in European wood industry.

This study has not shown any stronger effects on the EMC of the wood material after the initial moisture of the wood has been removed. This indicates that the wood has not been seriously affected by oxidation or dehydration.

The results indicate that the difference between the effects of a specific treatment climate on planks as compared to small clearwood samples is linked to the water content of the wood. The small samples quickly dries out at elevated temperatures, after which hydrolysis is decelerated or stopped. This would in turn indicate that tests on small clearwood samples need to be performed in such a way that the internal wood climate corresponds to the internal climate of planks during drying in order to result in corresponding results.

CONCLUSIONS

- The study has lead to significant reduction in EMC of small clearwood samples after heat treatment.

- The hydrolysis during thermal treatment seems to be dependent on the water in the wood rather than the humidity of the atmosphere.

- As the small samples dry out, hydrolysis is halted.

- The procedure during test on small clearwood samples need to be adapted to the internal wood climate in order to provide results that correspond to industrial drying of planks.
REFERENCES