CHAPTER 5

Effect of Densification on Chemical Composition of Wood

5.1 INTRODUCTION

It was shown in many studies that the combination of high temperature and steam pressure, used during preparation of densified wood, may cause changes in chemical composition of wood (Fengel and Wegener, 1989; Kosikova et al. 1993; Ebringerova et al. 1993; Gardner et al. 1993; Inoue et al. 1993, 1996, Hsu et al. 1988). Generally, wood components are stable up to 100°C and up to 48 hours of treatment (Fengel and Wegener, 1989). At higher temperatures significant changes in wood components may occur. The amount of degradation is time –dependent. The hemicelluloses are the least stable. Cellulose content remains relatively constant up to 150°C and than decreases. The lignin content is stable up to 150 °C.

Kosikova et al. (1993) and Ebringerova et al. (1993) investigated an effect of steam treatment on structural changes of the lignin-polysaccharide complex. Beech, aspen and spruce flakes were subjected to various conditions of steaming, such as heating with steam at 200°C for 10 min, heating with steam under pressure of about 4.3 MPa at 255 °C for 55 sec, heating with steam at 115 °C for 5 min and heating with steam at 200 °C for 2 min under steam explosion conditions (4.3 MPa). The changes in structure of wood polymers were characterized using infrared spectroscopy. An apparent increase in the proportion of cellulose crystallinity was found in the steamed samples, particularly in the steam explosion wood. The increase was attributed to hydrogen bond destruction as well as to splitting of lignin-hemicellulose linkages in hardwoods and spruce after steam treatment.

Gardner et al. reported an increase in cellulose crystallinity and theorized crosslinking for heat-treated and steam-treated flakes pressed at a stress of 22kN for 12 minutes at 23°C, 220°C, and 120°C with steam.

5.2 EXPERIMENTAL

5.2.1 Materials

The material for the analysis was obtained from the log sections of yellow-poplar (*Liriodendron tulipifera),* loblolly pine (*Pinus taeda*) and aspen (*Populus tremuliodes).* Wood sections were separated into juvenile, mature and intermediate portions. Juvenile wood was defined as the wood inside the tenth growth ring and mature wood as that material outside the thirtieth growth ring. The material was then sealed and allowed to air dry at ambient laboratory conditions for about 12 months.

5.2.2 Methods

Desorption experiments

Desorption experiments were performed in the pressurized sorption apparatus (Lenth, 1999). Mature wood specimens were placed in the sorption vessel with three liters of water. The vessel was then sealed and heated to 160 °C. The pressure was increased to the level necessary for the first relative humidity step (95%), and the mass of the specimen was recorded throughout the duration of that step. The chamber was then depressirized and the specimen's weight was recorded. Subsequently, the specimen was oven-dried at 103 ± 2°C and weighed again. The procedure was repeated for all the relative humidity steps. Once the desorption experiment was complete, the apparatus was cooled, and the specimens were taken out, weighed, and placed in a convection oven at 160°C to dry.

Chemical Analysis

Chemical composition of the specimens, with respect to the relative amount of cellulose, hemicellulose and lignin before and after desorption experiments at 160°C, were quantitatively assessed by acid hydrolysis followed by high performance liquid chromatography (HPLC). No extraction was performed. The analytical method for woody materials described by Kaar et al. (1991) was utilized. Ash content was also determined by thermogravimetric analysis (TGA) at 700°C with a rate of 10 °C/min. One sample per specimen type was evaluated.

5.3 RESULTS AND DISCUSSION

The results of the chemical analysis are listed in Table 5.1. The observed changes in the amount of total sugars were not significant in light of the accuracy of the technique. The amount of total lignin increased after desorption, but also not significantly. Generally, mature wood samples tended to lose 2 to 3.5 % of their hemicelluloses during desorption, with southern pine recording the highest losses. As a result of these hemicellulose losses, there was an increase in percent of lignin and cellulose content resulting from desorption. Southern pine exhibited the largest increase in total lignin, and the smallest increase in cellulose. Since no extraction was performed, most degradation products occurring during the treatment would remain in the wood, and thus included in the results in Table 5.1.

5.4 CONCLUSIONS

The results of the chemical analysis of the wood specimens before and after desorption experiments did not reveal significant changes in chemical composition of wood when subjected to 160 °C, pure steam for up to 8 hours.

5.5 REFERENCES

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Table 5.1 Composition of wood samples (Southern Pine, Aspen, Yellow Poplar) before and after desorption experiment.

Sample ID	Sugar Composition[1] ,%						Lignin, %			Ash Content[2], %
	Cellulose	Non-Cellulose				Total Sugar	Acid Insol. (Klason)	Acid Soluble	Total Lignin	
	Glucan	Xylan	Galactan	Arabinan	Mannan					
SPMu	43.94	6.97	2.22	0.00	15.17	68.29	25.55	0.29	25.84	0.03
SPMt	44.43	5.63	1.54	0.00	13.12	64.71	29.41	0.58	29.99	0.58
ASMu	42.95	17.04	0.00	0.00	1.85	61.85	19.24	3.07	22.31	0.43
ASMt	44.40	14.80	0.00	0.00	1.66	60.86	21.59	2.97	24.56	0.13
YPMu	43.91	13.76	0.00	0.00	1.51	59.18	22.06	3.91	25.97	0.41
YPMt	47.58	12.26	0.00	0.00	1.68	61.52	25.35	3.18	28.53	0.02
Sigma Oat Spelts Xylan (control)	11.19	66.66	0.00	6.49	0.00	84.34	2.20	0.57	2.77	3.22

u = untreated wood, t = after desorption at 160 °C

1) Sugar composition and lignin content determined according to procedure described by Kaar et. al., 1991, *J. Wood Chem. Technol.*, 11(4):447-469
2) Ash content determined by thermogravimetric analysis (700°C, 10°C/min)
3) Klason lignin also includes extractives in this analysis
4) Total composition of a sample is not 100 %, due to not measuring uronic acids and acetyl content