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Evaluation of extractives content and its influence in mechanical properties of Albanian black pine (Pinus Nigra *Arn.*)

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ABSTRACT

This study is focused on extractives content and its influence in mechanical properties, respectively bending and compression strength of Black Pine (Pinus nigra Arn.). We analyzed first, the extractives content of 18 sawdust samples, taken from cuts in six different heights of three trees. It has resulted that the content of extractives in the samples decreased from the base to the top of the tree and their content in wood was about 12 %. Secondly, we analyzed the influence of wood extractives content on the static bending and compression strength of Black Pine specimens with the same provenience as the sawdust samples. According to the UNI-ISO 31-33, we tested 160 specimens, all air dried to 12% moisture content, 2x2x32 cm for bending strength and 2x2x4 cm for compression strength. Half of the specimens are extracted in organic extractives first and then in hot water(60 ºC). The results showed that in extracted specimens, the average bending strength value for the extracted samples is reduced by 13 % for heartwood and 6% for sapwood and the average compression strength value by 10% and 5%.

Keywords: Black Pine, extractives content, bending strength, compression strength, tests.

INTRODUCTION

Extractives are compounds present in trees and are not considered to be structural components of wood. Generally are found in higher concentrations in the bark of the most of wood species and are considered to be biosynthesized in order to slow down or prevent pathogen invasions. Their production is under strict genetic control and some individual compounds are limited to individual species.

Extractives can be divided into two main classes: 1. extractives deposited in the coarse capillary structure and 2. extractives deposited in the cell wall structure [1]. They are usually divided into three subgroups: (i) aliphatic compounds; (ii) terpenes and terpenoids, and (iii) phenolic compounds.

The content of extractives and their composition vary greatly among different wood species and also within different parts of the same tree [2-4]. In general, the overall percent of the extractives in wood varies from 2 to 15 percent, with the exception of some tropical wood species, which have a higher extractives content varying from 20 to 25% [5]. Wood's extractives can be extracted by organic solvents. Some undesirable effects of extractives content in wood are related to an increased consumption of chemicals for pulping and bleaching purposes [6],metal corroding effects of tannins, interferences with setting of paints, glues and varnishes, migration during drying process, causing so the discoloration of wood. On the other hand some of them, but not all, are toxic to decay organisms or insects, playing a protective role.

Black pine is the most important coniferous species in Albanian forests covering 138247 ha, with a standing volume of 8,8 million m^3 [7] and its timber has been extensively used during the last decades in the Albanian wood processing industry. For an optimal utilization of black pine timber by the wood processing industry we have to know well the chemical, anatomical and mechanical properties of it. Among these properties, generally only the mechanical properties specifications are frequently considered by the industry. But it is known, that the chemical composition of wood has an influence on the physical and mechanical properties. In the cell wall, for example, low molecular weight substances occupy the same space in which hygroscopic water could enter. Due to this, extractives lower the equilibrium moisture content (EMC) of wood and reduce its swelling and shrinking [8,9]. A linear correlation between the extractive content and EMC at fiber saturation point (FSP) was confirmed by Popper et al. [10]. FSP is affected, among other factors, by the presence of extractives and increased after the removal of wood extractives [11]. Extractives had little effect on the monolayer adsorption of water but an appreciable effect on the polylayer adsorption. Choong *et al.* [12] indicated that removing extractives with hot water and organic solvents causes excessive shrinkage in tropical woods.

Extractives give a dark color to wood and provide a better dimensional stability [13]. The importance of extractives on the physical and mechanical properties of wood species have been emphasized in various studies [14,15].Although, there are some studies on physical and mechanical properties of some European and tropical woods, very limited data were obtained for effect of extractives on mechanical properties .

MATERIALS AND METHODS

Evaluation of extractive content of Black Pine : Three trees of black pine were randomly sampled from a black pine stand in Biza, Martanesh region in Central Albania. From each tree, six discs have been cut, starting at 20 cm from the base of the tree, in 25 cm intervals. The sawdust generated during the cross-cutting process was collected to form eighteen samples to be analyzed for extractives content. The sawdust samples were air dried for 2 hours, milled and screened through 40 to 60 mesh screens. The material particles used for extraction were about 0,4 mm and analysis of extractives were carried out in accordance with TAPPI method T204 om-88. Each sample was extracted with ethanol 95%. The extraction apparatus consisted of Soxhelt extraction units, connected to a 250 ml flat-bottomed flasks and condensers. Approximately 5 g per sample was weighed into tarred cellulose thimbles and extracted with 200 ml of solvent for 6 hours. After extraction, the solvents were evaporated. The remaining extracts were weighed and expressed as percentage of oven dried mass of the sample in the thimble.

Effect of extractives content on mechanical properties of Black Pine : In a second step we analyzed the influence of the wood extractives content on the static bending and compression strength of black pine (Pinus nigra *Arn.*) sample specimens with the same provenience as the sawdust samples. 160 sample specimens were obtained from quarter sawn boards including 80 samples from sapwood and 80 from heartwood. We tested 2x2x32 cm specimens for bending strength test and 2x2x4 cm specimens for compression strength tests. All tested samples were air dried to 12% moisture content. The aim was to compare the bending and compression strength values of extracted sample specimens with the unextracted ones. Half of the tested are extracted in organic extractives first and then in hot water 60 ºC. The extracted solvent is prepared as the mixer of toluene with ethanol (1:1), which has a very deep extraction effect and it is considered the best substitute of benzene, a solvent with hazard effect on human health. The samples are soaked in solvent for 15 days, changing it every two days followed by ten days water extraction at 60 °C. This procedure is considered sufficient to remove all relevant extracts in wood[5]. After the extracting procedure all sample specimens are tested according to the norm EN 408 in order to obtain characteristic values for bending strength and compression strength parallel to grain Tests are conducted in the wood mechanical properties testing laboratory located in the Department of the Wood Industry in the Faculty of Forestry Sciences in Tirana.

RESULTS AND DISCUSSION

Evaluation of extractive content of Black Pine : The percentage of extractives (E) in black pine samples is calculated by the formula below:

$$E_{\%} = (WE/WBE) \times 100 \qquad ---1$$

Where the mean weight of dried material before extraction (WBE) was approximately 4,76 g and the mean weight of extractive after the evaporation process (WE) was approximately 0,567 g. The detailed resulted for all the samples including WBE, mean WBE, WE, mean WE and standard deviation (STDV) values are given in the table1.

Tab. 1 Results for chemical extractives content in wood for Black Pine sawdust samples

Tree	No. of tests[a]	WBE (g)	Mean WBE and (STDV) (g)	WE (g)	Mean WE and (STDV) (g)	E (%)	Mean E and (STDV) (%)
1	1	4.72	4.69 (0.028)	0.387	0.394(0.021)	8.19	8.41 (0.413)
	2	4.71		0.432		9.17	
	3	4.68		0.398		8.50	
	4	4.65		0.375		8.06	
	5	4.66		0.369		7.92	
	6	4.72		0.405		8.58	
2	1	4.72	4.72 (0.057)	0.533	0.533 (0.01)	11.29	11.29 (0.16)
	2	4.72		0.548		11.61	
	3	4.67		0.521		11.16	
	4	4.63		0.519		11.21	
	5	4.77		0.542		11.36	
	6	4.8		0.534		11.13	
3	1	4.86	4.88 (0.084)	0.812	0.774(0.025)	16.71	15.86 (0.71)
	2	4.88		0.772		15.82	
	3	4.79		0.768		16.03	
	4	4.81		0.793		16.49	
	5	4.89		0.765		15.64	
	6	5.05		0.732		14.50	

[a] *The numbers of tests are lined from the base to the top of the tree*

According to the calculations has resulted that the content of extractives in the sawdust samples of Black Pine, is decreasing from the base to the top of the tree. As shown in the tab.1 and as already mentioned in the paragraphs above, the content of extractives and their composition vary greatly not only among different wood species, but also within different parts of the same tree .Their content in the Black pine samples tested was approximately 12 % in line with values between 2 to 15% of extractive content resulted from similar studies.

Effect of extractives content on mechanical properties of Black Pine : In order to analyze the influence of the wood extractives content on the static bending and compression strength of black pine sample we tested 160 specimens, half of them unextracted and half extracted. After collecting the values of the forces in breaking F, the tension in bending was calculated with the formula:

$$\sigma_B = 3/2 * (F*l)/bh^2 \qquad [2]$$

where: σ_B – tension in bending in N mm^{-2} , F - force in breaking in N , l – specimen length in mm , b – specimen transversal section width in mm , h – specimen transversal section height in mm.

Meanwhile the compressive tension are calculated by the formula:

$$\sigma_C = F/S \qquad [3]$$

where: σ_C – compressive tension in N mm^{-2} , F – force in breaking in N, S – specimen transversal section in mm^2

The detailed tests results are shown in the table2 and 3 .

Table. 2. Tests results for bending strength of unextracted and extracted specimens

Unextracted sample specimens						Extracted sample specimens					
No.	**Sapwood**		**No.**	**Heartwood**		**No.**	**Sapwood**		**No.**	**Heartwood**	
	Force (N)	σ_B (N/mm^2)		Force (N)	σ_B (N/mm^2)		Force (N)	σ_B (N/mm^2)		Force (N)	σ_B (N/mm^2)
1	2001	105.1	**1**	2201	115.6	**1**	1923	101.0	**1**	1968	103.3
2	1988	104.4	**2**	2187	114.8	**2**	1926	101.1	**2**	1950	102.4
3	2081	109.3	**3**	2289	120.2	**3**	1925	101.1	**3**	1970	103.4
4	2176	114.2	**4**	2393	125.7	**4**	1923	101.0	**4**	1968	103.3
5	1990	104.5	**5**	2189	114.9	**5**	1923	101.0	**5**	1954	102.6
6	2003	105.2	**6**	2203	115.7	**6**	1923	101.0	**6**	1951	102.4
7	1995	104.7	**7**	2195	115.2	**7**	1926	101.1	**7**	1976	103.7
8	2231	117.1	**8**	2454	128.8	**8**	1925	101.1	**8**	1956	102.7
9	2130	111.8	**9**	2343	123.0	**9**	1924	101.0	**9**	1954	102.6
10	1990	104.5	**10**	2189	114.9	**10**	1924	101.0	**10**	1962	103.0
11	1994	104.7	**11**	2193	115.2	**11**	1922	100.9	**11**	1954	102.6
12	2004	105.2	**12**	2204	115.7	**12**	1916	100.6	**12**	1958	102.8
13	2100	110.3	**13**	2310	121.3	**13**	1925	101.1	**13**	1957	102.7
14	2000	105.0	**14**	2200	115.5	**14**	1920	100.8	**14**	1970	103.4
15	1989	104.4	**15**	2188	114.9	**15**	1921	100.9	**15**	1969	103.4
16	1990	104.5	**16**	2189	114.9	**16**	1923	101.0	**16**	1953	102.5
17	1994	104.7	**17**	2193	115.2	**17**	1922	100.9	**17**	1951	102.4
18	1997	104.8	**18**	2197	115.3	**18**	1922	100.9	**18**	1945	102.1
19	1999	104.9	**19**	2199	115.4	**19**	1923	101.0	**19**	1956	102.7
20	1985	104.2	**20**	2184	114.6	**20**	1925	101.1	**20**	1948	102.3
21	1890	99.2	**21**	2079	109.1	**21**	1919	100.7	**21**	1964	103.1
22	1995	104.7	**22**	2195	115.2	**22**	1926	101.1	**22**	1975	103.7
23	2001	105.1	**23**	2201	115.6	**23**	1935	101.6	**23**	1959	102.8
24	2002	105.1	**24**	2202	115.6	**24**	1931	101.4	**24**	1956	102.7
25	1995	104.7	**25**	2195	115.2	**25**	1932	101.4	**25**	1965	103.2
26	2000	105.0	**26**	2200	115.5	**26**	1933	101.5	**26**	1973	103.6
27	2000	105.0	**27**	2200	115.5	**27**	1934	101.5	**27**	1966	103.2
28	2002	105.1	**28**	2202	115.6	**28**	1929	101.3	**28**	1982	104.1
29	2002	105.1	**29**	2202	115.6	**29**	1931	101.4	**29**	1983	104.1
30	1989	104.4	**30**	2188	114.9	**30**	1933	101.5	**30**	1969	103.4
31	1995	104.7	**31**	2195	115.2	**31**	1928	101.2	**31**	1967	103.3
32	1989	104.4	**32**	2188	114.9	**32**	1929	101.3	**32**	1979	103.9
33	1999	104.9	**33**	2199	115.4	**33**	1925	101.1	**33**	1971	103.5
34	1999	104.9	**34**	2199	115.4	**34**	1926	101.1	**34**	1977	103.8
35	1995	104.7	**35**	2195	115.2	**35**	1928	101.2	**35**	1975	103.7
36	1995	104.7	**36**	2195	115.2	**36**	1925	101.1	**36**	1966	103.2
37	1999	104.9	**37**	2199	115.4	**37**	1925	101.1	**37**	1975	103.7
38	1998	104.9	**38**	2198	115.4	**38**	1922	100.9	**38**	1959	102.8
39	1999	104.9	**39**	2199	115.4	**39**	1922	100.9	**39**	1950	102.4
40	2005	105.3	**40**	2206	115.8	**40**	1922	100.9	**40**	1952	102.5
Average		**108.1**	**Average**		**118.2**	**Average**		**101.2**	**Average**		**103.1**
STDV		**2.9**	**STDV**		**3.2**	**STDV**		**0.2**	**STDV**		**0.5**

According the tests results shown in table2 and 3 we obtained decreasing values of bending and compression strength for the extracted specimens. Bending strength is decreasing in average by about 13% for the heartwood and by about 6 % for sapwood in extracted sample specimens. In the same direction are also the values for compression strength, which are decreasing in average by about 10 % for heartwood and 5 % for sapwood sample specimens in extracted sample specimens. Any significant differences in mechanical performance between sapwood and heartwood are usually attributed to the radial changes in wood density or anatomical structure and not to whether the sample is heartwood or sapwood, *per se* .This results show as already expected, that the presence of extractives in wood affects the mechanical resistance properties of wood, in our case that of Black Pine timber. In general heartwood is more durable and last longer than sapwood and a higher content of extractive in heartwood leads to a better mechanical resistance of this part of wood in comparison with sapwood.

Table. 3 Tests results for compression strength of unextracted and extracted specimens

Unextracted sample specimens						Extracted sample specimes					
	Sapwood			Heartwood			Sapwood			Heartwood	
No.	Force (N)	σ_C (N/mm^2)	No.	Force (N)	σ_C (N/mm^2)	No.	Force (N)	σ_C (N/mm^2)	Nr	Force (N)	σ_C (N/mm^2)
1	2001	105.1	1	2201	115.6	1	1923	101.0	1	1968	103.3
2	1988	104.4	2	2187	114.8	2	1926	101.1	2	1950	102.4
3	2081	109.3	3	2289	120.2	3	1925	101.1	3	1970	103.4
4	2176	114.2	4	2393	125.7	4	1923	101.0	4	1968	103.3
5	1990	104.5	5	2189	114.9	5	1923	101.0	5	1954	102.6
6	2003	105.2	6	2203	115.7	6	1923	101.0	6	1951	102.4
7	1995	104.7	7	2195	115.2	7	1926	101.1	7	1976	103.7
8	2231	117.1	8	2454	128.8	8	1925	101.1	8	1956	102.7
9	2130	111.8	9	2343	123.0	9	1924	101.0	9	1954	102.6
10	1990	104.5	10	2189	114.9	10	1924	101.0	10	1962	103.0
11	1994	104.7	11	2193	115.2	11	1922	100.9	11	1954	102.6
12	2004	105.2	12	2204	115.7	12	1916	100.6	12	1958	102.8
13	2100	110.3	13	2310	121.3	13	1925	101.1	13	1957	102.7
14	2000	105.0	14	2200	115.5	14	1920	100.8	14	1970	103.4
15	1989	104.4	15	2188	114.9	15	1921	100.9	15	1969	103.4
16	1990	104.5	16	2189	114.9	16	1923	101.0	16	1953	102.5
17	1994	104.7	17	2193	115.2	17	1922	100.9	17	1951	102.4
18	1997	104.8	18	2197	115.3	18	1922	100.9	18	1945	102.1
19	1999	104.9	19	2199	115.4	19	1923	101.0	19	1956	102.7
20	1985	104.2	20	2184	114.6	20	1925	101.1	20	1948	102.3
21	1890	99.2	21	2079	109.1	21	1919	100.7	21	1964	103.1
22	1995	104.7	22	2195	115.2	22	1926	101.1	22	1975	103.7
23	2001	105.1	23	2201	115.6	23	1935	101.6	23	1959	102.8
24	2002	105.1	24	2202	115.6	24	1931	101.4	24	1956	102.7
25	1995	104.7	25	2195	115.2	25	1932	101.4	25	1965	103.2
26	2000	105.0	26	2200	115.5	26	1933	101.5	26	1973	103.6
27	2000	105.0	27	2200	115.5	27	1934	101.5	27	1966	103.2
28	2002	105.1	28	2202	115.6	28	1929	101.3	28	1982	104.1
29	2002	105.1	29	2202	115.6	29	1931	101.4	29	1983	104.1
30	1989	104.4	30	2188	114.9	30	1933	101.5	30	1969	103.4
31	1995	104.7	31	2195	115.2	31	1928	101.2	31	1967	103.3
32	1989	104.4	32	2188	114.9	32	1929	101.3	32	1979	103.9
33	1999	104.9	33	2199	115.4	33	1925	101.1	33	1971	103.5
34	1999	104.9	34	2199	115.4	34	1926	101.1	34	1977	103.8
35	1995	104.7	35	2195	115.2	35	1928	101.2	35	1975	103.7
36	1995	104.7	36	2195	115.2	36	1925	101.1	36	1966	103.2
37	1999	104.9	37	2199	115.4	37	1925	101.1	37	1975	103.7
38	1998	104.9	38	2198	115.4	38	1922	100.9	38	1959	102.8
39	1999	104.9	39	2199	115.4	39	1922	100.9	39	1950	102.4
40	2005	105.3	40	2206	115.8	40	1922	100.9	40	1952	102.5
Average		**108.1**	**Average**		**118.2**	**Average**		**101.2**	**Average**		**103.1**
STDV		**2.9**	**STDV**		**3.2**	**STDV**		**0.2**	**STDV**		**0.5**

APPLICATIONS

This study is focused on extractives content and its influence in mechanical properties, respectively bending and compression strength of Black Pine (Pinus nigra Arn.). The extractives contain (i) aliphatic compounds; (ii) terpenes and terpenoids, and (iii) phenolic compounds.

CONCLUSIONS

Concluding we can say that the results of the study show for Black Pine similar values related to the chemical extractives content in the wood comparing to studies for other tree species. The content of chemical extractives in the Black Pine samples tested is about 12% comparing to 2 – 15% content values we found in the field literature. We found that there is an effect of the chemical extractives content in the mechanical properties of Black pine samples tested for bending and compressive strength. Tests show decreasing values of bending and compressive strength in specimens tested after being extracted. Decreasing rate is higher in the heartwood specimens comparing to sapwood confirming similar findings in different studies in the field.

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