

The effects of heartwood and sapwood on kraft pulp properties of *Pinus nigra* J.F.Arnold and *Abies bornmuelleriana* Mattf.

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Abstract: The effects of heartwood and sapwood on kraft pulp properties of *Pinus nigra* J.F.Arnold. and *Abies bornmuelleriana* Mattf. were investigated. The differences in terms of chemical composition and fiber properties between the heartwood and sapwood of these species were also examined. Heartwood had more holocellulose and extractive compared to sapwood. Moreover, heartwood fiber length was shorter than that of sapwood. Kraft cookings of heartwood and sapwood each species were separately done under fixed cooking conditions. The results indicated that heartwood pulp had lower pulp viscosity and total pulp yield than sapwood did. The tear index of sapwood unbeaten and beaten kraft pulp was higher than that of heartwood. However, the burst and tensile indices of heartwood unbeaten and beaten kraft pulp were higher than those of sapwood.

Key words: Heartwood, kraft method, sapwood, softwood

1. Introduction

When a tree stem is cut transversely, a surface appears, composed of various annual growth rings present in concentric bands. The tree consists of 6 layers from outside to inside: outer bark, inner bark, vascular cambium, sapwood, heartwood, and pith. The outer bark layer protects the softer inner bark from mechanical impact and helps to restrict water loss via evaporation. In the inner bark (phloem) layer, sugars produced by photosynthesis are translocated from the leaves to the roots or growing portions of the tree. The vascular cambium layer is between the bark and the wood and produces both these tissues (Wiedenhoef and Miller 2005). The sapwood and heartwood are histologically similar to each other but are physiologically different zones of xylem (Pinto et al. 2004). Some xylem cells in the tree are living and hence physiologically active; they are called sapwood. After an indefinite length of time, the protoplasm of the living cells in the xylem physiologically dies. This part of the xylem is called heartwood (Panshin and De Zeeuw 1980). The heartwood is mostly darker in color compared to sapwood (Henriksson et al. 2009). The pith is the inner layer of tree stem. This layer comprises remnants of the early growth of tree stem before wood formation (Wiedenhoef and Miller 2005).

After heartwood formation, the bordered pits in softwoods become closed due to pit aspiration and the

vessels in some hardwoods become blocked by tyloses (Daniel 2009). Thus, heartwood does not take part in water conduction and becomes less permeable (Fujita and Harada 2001; Henriksson et al. 2009). On the other hand, heartwood has much lower moisture content than sapwood does. Therefore, heartwood is more vulnerable to fungal attacks (Jansson and Nilvebrant 2009). Heartwood extractives in some species have toxic effects. Due to these extractives, heartwood is more durable to the attack of microorganisms and insects compared to sapwood (Pereira et al. 2003; Henriksson et al. 2009). Heartwood has more lignin and less cellulose and pentosan content than sapwood (Timell 1986). On the other hand, the extractive content of heartwood is higher than that of sapwood (Fengel and Wegener 1989).

The anatomical and chemical differences between sapwood and heartwood often have a significant influence on pulping (Sjöström 1981). Heartwood containing more extractive compared to sapwood increases the consumption of pulping chemicals and reduces the pulp yield. The dark colored heartwood also decreases pulp brightness. Therefore, heartwood negatively affects bleaching (Esteves et al. 2005). The extractive causes pitch problem in paper mills such as sheet breaks, thin spots and holes in sheets, plugging of screens and cleaners, and deposits on several parts of the paper machine (Allen 2000). Heartwood containing more extractive than

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sapwood can often increase these problems. On the other hand, heartwood has lower permeability compared to sapwood. For this reason, cooking liquor cannot penetrate heartwood chips, and this increases the number of rejects (Pereira et al. 2003).

The aim of the present study was to examine the influence of heartwood and sapwood of *Pinus nigra* J.F. Arnold and *Abies bornmuelleriana* Mattf. on kraft pulp and paper properties, as well as to determine the differences in terms of chemical composition and fiber properties between the heartwood and sapwood of these species.

2. Materials and methods

The European black pine (*P. nigra*) covers approximately 3.5 million ha from western North Africa through southern Europe to Asia Minor (Isajev et al. 2004). On the other hand, the Uludağ fir (*A. bornmuelleriana*), initially described by the famous German botanist Joseph Friedrich Nicolaus Bornmüller, is an endemic species in Turkey. It is widely distributed in the Marmara and western Black Sea regions of Turkey (Saatçioğlu 1971).

Logs of *P. nigra* and *A. bornmuelleriana* were collected in Bartın Province in Turkey. Some properties of the trees used in this study are shown in Table 1. Wood disks 10 cm thick were taken from breast height of each log. These disks were debarked and subdivided into 4 disks in terms of disk height (2.5 cm disk height). The width of the heartwood and sapwood was determined. The numbers of annual rings of heartwood and sapwood were also counted. The heartwood and sapwood of each disk were easily separated in each wood disk with a chisel. Heartwood and sapwood samples were manually chipped to 2.5 × 1.5 × 0.5 cm size chips for pulping using a chisel. The wood chips were air-dried to a final humidity of 10% and stored in dry conditions.

Specimens of heartwood and sapwood of both species were also sampled and prepared according to the TAPPI T 257 standard for chemical analysis. Holocellulose (Browning 1967), α -cellulose (Han and Rowell 1997), and

lignin (TAPPI T 222) contents of samples were determined according to the relevant methods. Solubility properties were also determined based on alcohol (TAPPI T 204), cold-hot water (TAPPI T 207), and 1% NaOH (TAPPI T 212) methods. On the other hand, Schulze's method was used in the maceration of heartwood and sapwood samples of each species. After maceration, the samples were agitated gently to disintegrate individual fibers (Berlyn and Miksche 1976) and dehydrated with ethyl alcohol and stored in glycerin after staining with safranin. The fiber dimensions of 50 randomly selected fibers were measured.

Heartwood and sapwood chips of each species were separately cooked. Four kraft cookings were carried out in this study. Each kraft cooking was performed under the following conditions: 18% active alkali as Na₂O, 30% sulfidity, 4:1 liquor/wood ratio, 170 °C cooking temperature, 90 min to cooking temperature, and 60 min at cooking temperature. In each experiment, 650 g of oven-dried wood chips were cooked in a 15-L electrically heated laboratory cylindrical-type rotary digester. At the end of pulping, the pressure was reduced to atmospheric pressure. After digestion, the pulps were washed to remove black liquor and were disintegrated in a laboratory-type pulp mixer with 2-L capacity. Disintegrated pulps were screened using a Somerville-type pulp screen with a 0.15-mm slotted plate (TAPPI T 275). The pulps were then beaten to 35 and 50 °SR in a Valley Beater according to TAPPI T 200. Kappa number (TAPPI T 236), screened yield (TAPPI T 210), viscosity (SCAN-CM 15-62), and freeness of pulps (ISO 5267-1) were determined according to relevant methods. Handsheets (75 g m⁻²) made by a Rapid- Kothen Sheet Former (ISO 5269-2) were conditioned (TAPPI T 402). Tensile index (TAPPI T 494), burst index (TAPPI T 403), tear index (TAPPI T 414), opacity (TAPPI T 519), brightness (TAPPI T 525), roughness (ISO 8791-2), and air permeability (ISO 5636-3) of the handsheets were determined in accordance with relevant standard methods. The data were compared using Student's t test at a 95% confidence level.

Table 1. Some properties of trees used in this study.

Properties	European black pine	Uludağ fir
Age	71	60
Breast height diameter (cm)	36	35
The number of annual rings in heartwood	14	35
The number of annual rings in sapwood	57	25
Heartwood ratio (%)	4.9	32.6
Sapwood ratio (%)	95.1	67.7

3. Results

Chemical components of the heartwood and sapwood of the European black pine and Uludağ fir are listed in Table 2. Chemical analysis results showed that the heartwood and sapwood were different from each other in terms of chemical composition. Experimental data on fiber dimensions of the heartwood and sapwood of the European black pine and Uludağ fir are presented in Table 3.

Kraft pulp properties of the heartwood and sapwood of the European black pine and Uludağ fir are given in Table 4. The kappa number of the heartwood kraft pulp of the European black pine was higher than that of sapwood. In contrast, the heartwood kraft pulp of the Uludağ fir had a kappa number lower than that of sapwood. This finding can be explained by the higher lignin content of European black pine heartwood and Uludağ fir sapwood.

The screened yield of heartwood kraft pulp of the European black pine was lower than that of sapwood. This result can be attributed to the lower α -cellulose content and higher extractive content of European black pine heartwood compared to sapwood. In contrast, heartwood kraft pulp of the Uludağ fir had a higher screened yield than sapwood did. This result can be explained by the lower lignin content and higher α -cellulose content of Uludağ fir heartwood compared to sapwood. The viscosity of heartwood kraft pulp of the European black pine and Uludağ fir was lower than that of sapwood.

The differences in the handsheet properties of the heartwood and sapwood of the European black pine and Uludağ fir are compared in Table 5. The tear index of sapwood unbeaten and beaten kraft pulp of the European black pine and Uludağ fir was higher than that of heartwood. This result can be attributed to the thicker walled fibers of sapwood. On the other hand, the burst and tensile indices of heartwood unbeaten and beaten kraft pulp of the European black pine and Uludağ fir were higher than those of sapwood.

4. Discussion

The chemical components of the heartwood and sapwood of the European black pine and Uludağ fir were different from each other in term of composition. These differences were observed in both main components and several solubilities of wood. Heartwood had lower holocellulose content than sapwood did. This finding is in agreement with those reported by Campbell et al. (1990), Mariana et al. (2005), and Gao et al. (2011b) in *Pinus contorta*, *Eucalyptus nitens*, and *Cedrus deodara*, respectively.

The α -cellulose content of European black pine heartwood was lower than that of sapwood. Similar results were previously reported by several researchers (Campbell et al. 1990; Mariana et al. 2005; Gao et al. 2011b). However, the α -cellulose content of Uludağ fir heartwood was higher than that of sapwood. A similar result was reported by Khalid et al. (2010) in an acacia hybrid.

The lignin content of European black pine heartwood was higher than that of sapwood. Similar results were previously reported by several researchers (Ritter and Fleck 1926; Bertaud and Holmbom 2004; Mariana et al. 2005; Khalid et al. 2010; Gao et al. 2011b). However, the lignin content of Uludağ fir heartwood was lower than that of sapwood. Similar results were reported by Lourenco et al. (2008) and Gao et al. (2011a) in *Acacia melanoxylon* and poplar I-69, respectively.

Furthermore, 1% NaOH, cold water, and hot water solubilities of European black pine heartwood were higher than those of sapwood. A similar result was reported by Ritter and Fleck (1926) in the redwood. However, Uludağ fir heartwood had NaOH solubility 1% lower than that of sapwood. A similar result was reported by Ritter and Fleck (1926) in the red mulberry. There were no statistically significant differences in terms of cold and hot water solubilities between the heartwood and sapwood of the Uludağ fir. The heartwood of the European black pine and Uludağ fir had higher extractive than sapwood did. This result confirms previous studies (Fengel and Wegener

Table 2. Chemical compositions of heartwood and sapwood of the European black pine and Uludağ fir.

Experiments	European black pine		Uludağ fir	
	heartwood	sapwood	heartwood	sapwood
Holocellulose (%)	65.4a	67.5b	70.0a	71.7b
α -Cellulose (%)	41.8a	44.6b	46.4a	45.4b
Lignin (%)	26.9a	25.3b	26.6a	27.8b
1% NaOH solubility (%)	22.3a	9.4b	7.6a	8.6b
Hot water solubility (%)	4.3a	1.7b	2.3a	2.4a
Cold water solubility (%)	2.4a	1.3b	1.5a	1.4a
Alcohol solubility (%)	16.6a	4.3b	2.8a	1.0b

Table 3. Fiber dimensions of heartwood and sapwood of the European black pine and Uludağ fir.

Experiments	European black pine		Uludağ fir	
	heartwood	sapwood	heartwood	sapwood
Fiber length (mm)	2.3a	3.6b	2.7a	3.6b
Fiber width (µm)	46.6a	52.4b	41.3a	49.6b
Lumen width (µm)	33.6a	38.2b	31.7a	39.5b
Double wall thickness (µm)	13.0a	14.2b	9.6a	10.1b

1989; Campbell et al. 1990; Mariana et al. 2005; Miranda et al. 2006, 2007; Lourenco et al. 2008, 2011).

The fiber dimension measurements of the heartwood and sapwood of the European black pine and Uludağ fir showed that sapwood had longer fibers than heartwood did. Similar results were previously reported by several researchers (Ay and Şahin 1998; Mariana et al. 2005; Liukkonen et al. 2007; Rayirath and Avramidis 2008; Saraeian et al. 2011; Gao et al. 2011a, 2011b). On the other hand, sapwood had fibers with larger lumina and thicker walls. Larger fibers in sapwood were reported by Ay and Şahin (1998) and Mariana et al. (2005) in *Picea orientalis* and *Eucalyptus nitens*, respectively. Thicker fibers in sapwood were reported by Mariana et al. (2005) and Liukkonen et al. (2007). In contrast, Ay and Şahin (1998) noted that the heartwood of *Picea orientalis* had thicker walled fibers than sapwood did.

The kappa number of the sapwood kraft pulp of the European black pine was lower than that of the heartwood. In contrast, the kappa number of the sapwood kraft pulp of the Uludağ fir was higher than that of the heartwood. Mariana et al. (2005), Lourenco et al. (2008), and Gao et al. (2011a, 2011b) noted that the kappa number of *Eucalyptus nitens*, *Acacia melanoxylon*, poplar I-69, and *Cedrus deodara* heartwood kraft pulp was higher than that of sapwood, respectively. However, Saraeian et al. (2011) found that the kappa number of *Populus deltoides* heartwood kraft pulp was lower than that of sapwood.

The sapwood screened yield of the European black pine was higher than that of heartwood. In contrast, the sapwood screened yield of the Uludağ fir was lower than

that of the heartwood. Previous studies about the screened yield (Mariana et al. 2005; Esteves et al. 2005; Miranda et al. 2007; Lourenco et al. 2008; Lourenco et al. 2011; Gao et al. 2011a, 2011b; Saraeian et al. 2011) indicated that the kraft pulp yield of heartwood was lower than that of sapwood. The total yield of the heartwood kraft pulp of the European black pine and Uludağ fir was lower than that of sapwood.

Heartwood had less permeability compared to sapwood (Rayirath and Avramidis 2008; Brännvall 2009). In kraft pulping, high permeability of wood results in a low reject ratio. As can be seen in Table 4, the reject ratios of heartwood kraft pulp of the European black pine and Uludağ fir were lower than those of sapwood. This result can be attributed to the heterogeneity in thickness of chips prepared by hand. Mariana et al. (2005) noted that the reject ratio of *Eucalyptus nitens* heartwood kraft pulp was higher than that of sapwood.

The viscosity of heartwood kraft pulp of the European black pine and Uludağ fir is in agreement with previous studies (Gao et al. 2011a, 2011b). However, Lourenco et al. (2008) noted that heartwood kraft pulp had higher viscosity compared to sapwood kraft pulp.

The differences in the handsheet properties of the heartwood and sapwood of the European black pine and Uludağ fir can be attributed to the higher permeability of sapwood handsheets. Gao et al. (2011a, 2011b) noted that the tensile, tear, and burst indices of heartwood kraft pulp were lower than those of sapwood. Saraeian et al. (2011) found that the strength properties of kraft pulp of *Populus deltoides* sapwood were higher than those of heartwood.

Table 4. Kraft pulp properties of heartwood and sapwood of the European black pine and Uludağ fir.

Experiments	European black pine		Uludağ fir	
	heartwood	sapwood	heartwood	sapwood
Kappa number	50.9	45.0	39.8	46.4
Screened yield (%)	38.7	43.9	46.8	45.2
Reject (%)	1.9	2.7	0.4	2.6
Total yield (%)	40.6	46.6	47.2	47.8
Viscosity (cm ³ g ⁻¹)	1021.1	1042.2	1020.6	1065.9

Table 5. Handsheet properties of heartwood and sapwood of the European black pine and Uludağ fir.

Cooking	Freeness level (°SR)	Tear index (mN m ² g ⁻¹)	Burst index (kPa m ² g ⁻¹)	Tensile index (N m g ⁻¹)	Air permeability (mL min ⁻¹)	Roughness (mL min ⁻¹)	Opacity (%)	Brightness (%)
European black pine heartwood	13	1.03	3.28	58.48	-	560.40	99.79	18.87
	35	0.79	6.53	115.24	25.4	383.9	98.84	15.91
	50	0.60	7.20	118.50	6.8	402.8	98.11	14.68
European black pine sapwood	13	1.34	2.44	46.78	-	962.6	99.53	20.80
	35	1.34	6.34	105.82	31.7	588.1	98.01	15.29
	50	0.83	6.45	109.10	10.4	851.3	97.08	14.10
Uludağ fir heartwood	15	1.39	2.90	59.06	-	597.8	99.73	21.35
	35	0.96	7.25	119.56	44.2	409.2	98.64	17.46
	50	0.95	6.90	131.64	4.6	414.7	98.37	16.92
Uludağ fir sapwood	15	1.58	2.18	43.88	-	932.2	99.47	18.81
	35	1.09	6.40	114.52	54.3	533.1	98.73	15.90
	50	0.96	6.58	116.16	10.6	467.9	98.22	14.80

Mariana et al. (2005) noted that the burst index of heartwood kraft pulp was lower than that of sapwood. The tensile and tear indices of handsheets of heartwood kraft pulp were higher than those of sapwood.

As shown in Table 5, sapwood handsheets of the European black pine and Uludağ fir were more permeable and rougher compared to heartwood handsheets. The brightness of heartwood handsheets of European black pine was lower than that of sapwood. This result can be explained by the higher extractive content and kappa number of heartwood in comparison to sapwood. The brightness of heartwood handsheets of the Uludağ fir was higher than that of sapwood. This result can be attributed to the lower kappa number of heartwood kraft pulp in comparison to sapwood kraft pulp. Lourenco et al. (2008) noted that the brightness of heartwood handsheets of *Acacia melanoxylon* was lower than that of sapwood. On the other hand, there was no significant difference in terms of opacity between heartwood and sapwood handsheets of the European black pine and Uludağ fir.

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