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Fiber Morphology and Chemical Composition of Heartwood and Sapwood of Red Gum, Black Willow, and Oriental Beech

Sezgin Koray GÜLSOY*[1], Hasan AKSOY[2], Hülya GÜL TÜRKMEN[1], Gülcan ÇANAKCI[1]

[1] Bartın University, Faculty of Forestry, Department of Forest Industrial Engineering,74100, BARTIN
[2] Sinop University, Ayancık Vocational School, Forestry and Forest Products, 57000, SİNOP

Abstract

In this study, the differences in terms of the fiber morphology and the chemical composition between the heartwood and sapwood of red gum (*Eucalyptus camaldulensis* Dehnh.), black willow (*Salix nigra* Marsh.), and oriental beech (*Fagus orientalis* Lipsky) were investigated. The results showed that the heartwood samples had shorter fibers and lower slenderness ratios than those of the sapwood samples. The differences in the vessel element length of the heartwood and sapwood of sampled tree species were statistically insignificant. The heartwood samples had less holocellulose and more klason lignin content. In addition, the ethanol, hot water, and cold water solubility values in heartwood samples were higher. The other morphological and chemical properties of the heartwood and sapwood depended on the tree species.

Keywords: Heartwood, sapwood, fiber length, holocellulose, klason lignin.

Okaliptüs, Kara Söğüt ve Doğu Kayınının Öz Odun ve Diri Odunlarının Lif Morfolojileri ve Kimyasal Bileşimleri

Öz

Bu çalışmada, okaliptüs (*Eucalyptus camaldulensis* Dehnh.), kara söğüt (*Salix nigra* Marsh.) ve doğu kayınının (*Fagus orientalis* Lipsky) öz odun ve diri odunları arasındaki lif morfolojisi ve kimyasal bileşim bakımından farklılıklar incelenmiştir. Çalışma sonuçlarına göre, öz odun örneklerinin diri odun örneklerinden daha kısa liflere ve daha düşük keçeleşme oranına sahip olduğu görülmüştür. Öz odun ve diri odun örneklerinin trahe uzunlukları arasındaki farkın istatistiki olarak anlamsız olduğu tespit edilmiştir. Öz odun örnekleri daha az holoselüloz ve daha fazla klason lignin içeriğine sahip olduğu belirlenmiştir. Buna ilave olarak, öz odun örneklerinin etanol, sıcak su ve soğuk su çözünürlükleri daha yüksek olduğu tespit edilmiştir. Öz odun ve diri odunun diğer lif morfolojisi değerleri ve kimyasal bileşenleri ağaç türüne bağlı olarak değişim gösterdiği görülmüştür.

Anahtar Kelimeler: Öz odun, diri odun, lif uzunluğu, holoselüloz, klason lignin.

***Sorumlu Yazar (Corresponding Author):**
Sezgin Koray GÜLSOY (Dr.); Bartın University, Faculty of Forestry, Department of Forest Industrial Engineering, 74100, Bartın-TURKEY. Tel: +90 (378) 223 5080, Fax: +90 (378) 223 5066, E-mail: sgulsoy@bartin.edu.tr
ORCID: 0000-0002-3079-9015

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1. Introduction

The transverse section of a tree has histologically similar but physiologically different two zones called heartwood and sapwood. The sapwood in the outer region has physiologically active living cells produced by the cambium. The water and minerals transport from roots to the leaves due to sapwood (Pinto et al. 2004). The heartwood in the inner region contains dead cells and provides the structural support to tree. The accumulation of phenolics in heartwood is its distinctive characteristic. Therefore, heartwood has a darker color compared to sapwood (Lourenço et al. 2010).

The fiber morphology of heartwood and sapwood of several tree species such as *Picea orientalis* (Ay and Şahin, 1998) *Picea abies* (Liukkonen et al. 2007), *Tsuga heterophylla* (Rayirath and Avramidis, 2008), *Pinus nigra* (İstek et al. 2010; Ataç and Eroğlu, 2013), *Cedrus deodara* (Gao et al. 2011b), *Populus deltoides* (Saraeiran et al. 2011; Gao et al. 2011a), *Abies bornmuelleriana* (Ataç and Eroğlu, 2013), *Robinia pseudoacacia* (Özdemir et al. 2015), *Pinus pinea* (Özdemir et al. 2015), *Cerasus avium* (Gençer and Gül Türkmen, 2016), *Populus alba* (Mertoglu-Elmas, 2019), and *Sorbus torminalis* (Bahmani et al. 2020) have been extensively studied.

Many studies have been carried out on the chemical composition of heartwood and sapwood of several tree species such as *Pinus contorta* (Campbell et al. 1990), *Acacia melanoxylon* (Lourenço et al. 2008), *Eucalyptus globulus* (Lourenço et al. 2010; Morais and Pereira, 2012), *Tectona grandis* (Miranda et al. 2011), *Populus deltoides* (Gao et al. 2011a), *Cedrus deodara* (Gao et al. 2011b), *Pinus nigra* (Ataç and Eroğlu, 2013), *Abies bornmuelleriana* (Ataç and Eroğlu, 2013), *Cerasus avium* (Gençer and Gül Türkmen, 2016), *Eucalyptus citriodora* (Puntambekar et al. 2016), *Quercus faginea* (Miranda et al. 2017), *Leucaena leucocephala* (Pydimalla et al. 2019), and *Sorbus torminalis* (Bahmani et al. 2020). But, available literature related to chemical composition (Ataç, 2009; Malakani et al. 2014) and fiber morphology (Ataç, 2009) of heartwood and sapwood of oriental beech (*Fagus orientalis* Lipsky) has been limited. To the best of our knowledge, no investigation has been carried out to determine fiber morphology and chemical composition of heartwood and sapwood of red gum (*Eucalyptus camaldulensis* Dehnh.) and black willow (*Salix nigra* Marsh.). Therefore, the objective of this study was to determine the differences in terms of fiber morphology and chemical composition and between the heartwood and sapwood of red gum, black willow, and oriental beech.

2. Materials and Methods

Characteristics of the sampled tree species are presented in Table 1. The wood discs in five-cm thick were taken from breast height of each tree species. These discs were debarked. The width of the heartwood and sapwood was determined to calculate the ratios of the sapwood and heartwood. The numbers of annual rings of each wood species were also counted. The sapwood and heartwood in each wood disc were separated. The sapwood and heartwood samples were manually chipped to matchstick size using a chisel. The sapwood and heartwood samples were air-dried to a final humidity of 10% and stored in dry conditions.

Table 1. Characteristics of the sampled tree species.

	Red gum	Black willow	Oriental beech
Family	Myrtaceae	Salicaceae	Fagaceae
Sampled area	Adana	Erzurum	Yenice-Karabük
Age	25	32	58
Diameter at breast height (cm)	29.5	22	27
Heartwood ratio (%)	69	40.5	64
Sapwood ratio (%)	31	59.5	36

Heartwood and sapwood samples for the chemical analysis were prepared according to the TAPPI T 257 standard. The holocellulose (Wise and Karl, 1962), α-cellulose (TAPPI T 203), and klason lignin (TAPPI T 222) contents of samples were determined according to the relevant methods. The solubility properties were also determined based on ethanol (TAPPI T 204), cold-hot water (TAPPI T 207), and 1% NaOH (TAPPI T 212) methods. On the other hand, heartwood and sapwood samples were macerated using the chlorite method (Spearin and Isenberg, 1947). After maceration, the samples were agitated gently to disintegrate individual fibers and dehydrated with ethyl alcohol and stored in glycerin. The fiber length, fiber width, lumen width, and cell wall thickness of 50 randomly selected fibers were measured. The slenderness ratio (fiber length/fiber width), flexibility ratio [(lumen width/fiber width) × 100], and Runkel ratio [(2 × cell wall thickness)/lumen width] were calculated using the measured fiber dimensions. The length of 50 randomly selected vessel element were also measured.

All data were analyzed using SPSS. The data related to fiber morphology and chemical composition of the heartwood and sapwood were analyzed statistically using the independent t-test ($p<0.05$). In Table 2 and Table 3, the same letter in the lines denotes no statistically significant differences between the groups.

3. Results and Discussion

The results of the fiber morphology of heartwood and sapwood of red gum, black willow, and oriental beech are summarized in Table 2. The fiber length of sapwood samples for all sampled tree species was statistically significantly longer than those of heartwood samples ($p<0.05$). This result can be ascribed to increasing cambial maturity (Beaulieu, 2003). Mariani et al. (2005) noted that the fiber length of *Eucalyptus nitens* sapwood was 12.6% longer than heartwood. Ataç (2009) reported that fiber length of heartwood and sapwood of *Fagus orientalis* and *Quercus robur* was 1.08 mm - 1.23 mm and 0.98 mm - 1.14 mm, respectively. Gao et al. (2011a) noted that fiber length of *Populus deltoides* was 1.15 mm in sapwood and 0.77 mm in heartwood. Özdemir et al. (2015) noted that fiber length of sapwood and heartwood of *Robinia pseudoacacia* was 1.54 mm and 1.36 mm, respectively. Gençer and Gül Türkmen (2016) in *Cerasus avium* and Mertoglu-Elmas (2019) in *Populus alba* reported that sapwood had slightly longer fibers than heartwood.

Table 2. Fiber morphology of red gum, black willow, and oriental beech.

Experiments	Red gum		Black willow		Oriental beech	
	Heartwood	Sapwood	Heartwood	Sapwood	Heartwood	Sapwood
Fiber length (mm)	0.77a	0.89b	0.71a	0.82b	0.99a	1.16b
Vessel element length (mm)	0.33a	0.35a	0.41a	0.37a	0.59a	0.60a
Fiber width (μm)	19.75a	19.5a	21.53a	23.58b	20.25a	20.20a
Lumen width (μm)	13.05a	11.5b	13.35a	17.13b	6.35a	5.70b
Cell wall thickness (μm)	3.35a	4.00b	4.09a	3.23b	6.95a	7.70b
Slenderness ratio	38.99	45.64	32.98	34.78	48.89	57.43
Flexibility ratio	66.08	58.97	62.01	72.65	31.36	28.22
Runkel ratio	0.51	0.70	0.61	0.38	2.19	2.70

The differences in the vessel element length of heartwood and sapwood of sampled tree species were statistically insignificant ($p>0.05$). Ataç (2009) reported that vessel element length of heartwood and sapwood of *Fagus orientalis* and *Quercus robur* was 0.59 mm – 0.71 mm and 0.47 mm – 0.58 mm, respectively. Gençer and Gül Türkmen (2016) reported that vessel element length of heartwood and sapwood of *Cerasus avium* was 427 μm and 424 μm, respectively.

Fiber width of black willow heartwood was narrower than that of sapwood (21.53 μm vs. 23.58 μm) as shown in Table 2. This result is in agreement with previous studies (Gao et al. 2011a, 2011b; Gençer and Gül Türkmen, 2016; Mertoglu-Elmas, 2019; Bahmani et al. 2020). In red gum and oriental beech samples, fiber width in sapwood and heartwood were similar ($p>0.05$). Ataç (2009) noted that fiber width of *Fagus orientalis* and *Quercus robur* was 23.75 μm - 22.50 μm in sapwood and 22.36 μm - 18.40 μm in heartwood, respectively.

Heartwood of red gum and oriental beech had wider fiber lumen and wider cell wall than sapwood. On the contrary, sapwood of black willow had wider fiber lumen and wider cell wall. Ataç (2009) stated that lumen width and cell wall thickness of *Fagus orientalis* were 6.04 μm and 8.86 μm in sapwood and 6.14 μm and 8.11 μm in heartwood, respectively. Lumen width was 17.14 μm in sapwood and 16.05 μm in heartwood of *Robinia pseudoacacia* (Özdemir et al. 2015). Bahmani et al. 2020 stated that lumen width of sapwood and heartwood of *Sorbus torminalis* were 6.6 μm and 4.1 μm, respectively. Gençer and Gül Türkmen (2016) reported that cell wall thickness of heartwood and sapwood of *Cerasus avium* was 4.93 μm and 4.35 μm, respectively. Mertoglu-Elmas (2019) noted that sapwood and heartwood of *Populus alba* had the similar cell wall thickness. Cell wall thickness was 10.3 μm in sapwood and 8.7 μm in heartwood of *Sorbus torminalis* (Bahmani et al. 2020).

The derived values from fiber properties of heartwood and sapwood of each sampled tree species are also presented in Table 2. Slenderness ratio of sapwood fibers of each species was higher than that of heartwood fibers. This result can be explained by longer fibers of sapwood. Slenderness ratio of sapwood and heartwood fibers of *Populus deltoides* was 51.76 and 42.56, respectively (Gao et al. 2011a). Slenderness ratio was 58.1 in sapwood fibers and 54.2 in heartwood fibers of *Robinia pseudoacacia* (Özdemir et al. 2015). Heartwood of red gum and oriental beech had more flexible fibers than sapwood. In contrast to this, heartwood of black willow had less flexible fibers. The higher flexibility ratio in heartwood was determined in *Cerasus avium* by Gençer and Gül Türkmen (2016). However, close values in flexibility ratio were observed in *Robinia pseudoacacia* by Özdemir et al. (2015). The runkel ratios of heartwood and sapwood fibers were varied depending on the tree species. Although sapwood fibers of red gum and oriental beech had higher Runkel ratio than heartwood. Heartwood had higher Runkel ratio in black willow. Runkel ratio of sapwood and heartwood fibers of *Populus deltoides* was 0.59 and 0.40, respectively (Gao et al. 2011a). Runkel ratio was 0.56 in sapwood fibers and 0.54 in heartwood fibers of *Robinia pseudoacacia* (Özdemir et al. 2015). Gençer and Gül Türkmen (2016) stated that sapwood fibers of *Cerasus avium* had higher Runkel ratio than heartwood fibers.

The results of chemical composition of heartwood and sapwood of red gum, black willow, and oriental beech are summarized in Table 3. The holocellulose and α-cellulose content of sapwood was significantly higher than those of heartwood in both red gum and oriental beech as shown in Table 3 (p<0.05). The difference between sapwood and heartwood in terms of holocellulose and α-cellulose content in black willow was insignificant (p>0.05). Ataç (2009) noted that holocellulose and α-cellulose content of *Fagus orientalis* were 78.84% and 41.05% in sapwood and 77.51% and 42.61% in heartwood, respectively. Ataç (2009) also reported that holocellulose and α-cellulose content of *Quercus robur* were 73.60% and 40.63% in sapwood and 70.11% and 39.97% in heartwood, respectively. On the other hand, Ruiz-Aquino et al. (2015) stated that holocellulose and α-cellulose content differences between sapwood and heartwood of *Quercus laurina* and *Quercus crassifolia* were insignificant.

Table 3. Chemical composition of red gum, black willow, and oriental beech.

Experiments	Red gum		Black willow		Oriental beech	
	Heartwood	Sapwood	Heartwood	Sapwood	Heartwood	Sapwood
Holocellulose (%)	80.99a	85.12b	72.42a	73.84a	81.45a	83.84b
α-cellulose (%)	46.08a	52.22b	42.35a	42.34a	44.70a	45.94b
Klason lignin (%)	27.52a	21.84b	27.23a	24.64b	21.65a	20.22b
Ethanol solubility (%)	15.50a	6.86b	7.17a	6.84b	2.43a	2.07a
1% NaOH solubility (%)	26.52a	15.48b	19.60a	20.02a	15.46a	14.61a
Hot water solubility (%)	12.17a	6.54b	7.06a	5.99b	3.35a	2.72b
Cold water solubility (%)	8.59a	5.87b	5.15a	4.50b	3.16a	2.48b

In the all sampled species, klason lignin content of sapwood was significantly lower than heartwood (p<0.05). Malakani et al. (2014) reported that klason lignin of *Fagus orientalis* was 24.01% in heartwood and 21.11% in sapwood. In addition, klason lignin of *Fagus orientalis* was 22.88% in heartwood and 22.43% in sapwood (Ataç, 2009). Some studies revealed that sapwood had lower klason lignin content than heartwood (Ataç, 2009; Gao et al. 2011b; Gençer and Gül Türkmen, 2016; Pydimalla et al. 2019; Bahmani et al. 2020). On the contrary, sapwood of *Eucalyptus nitens* (Mariani et al. 2005), *Acacia melanoxylon* (Lourenço et al. 2008), *Eucalyptus globulus* (Lourenço et al. 2010), *Populus deltoides* (Gao et al. 2011a), and *Eucalyptus citriodora* (Puntambekar et al. 2016), *Quercus faginea* (Miranda et al. 2017) had higher klason lignin than those of heartwood.

In the all sampled species, heartwood samples had higher solubility values such as 1% NaOH, ethanol, hot water and cold water solubility. Previous researchers found that heartwood of *Eucalyptus urograndis* (Gominho et al. 2001), *Quercus oleoides* (Hernández and Salazar, 2005), *Acacia melanoxylon* (Lourenço et al. 2008), *Eucalyptus globulus* (Miranda et al. 2007; Lourenço et al. 2010; Morais and Pereira, 2012), *Populus deltoides* (Gao et al. 2011a), *Quercus faginea* (Miranda et al. 2017), and *Sorbus torminalis* (Bahmani et al. 2020) contained more extractives than sapwood. The higher extractives in heartwood can be ascribed to accumulation of soluble compounds during heartwood formation (Lourenço et al. 2008). Also, the higher extractive content of heartwood is considered a heartwood specific characteristic (Sjöström, 1993). Seikel et al. (1971) noted that total extractibles of *Quercus rubra* were 6.2% in sapwood and 8.2% in heartwood. Malakani et al. (2014) reported that hot water, 1%NaOH, and alcohol-benzene solubility values of heartwood and sapwood samples of *Fagus oriestalis* were 5.34% - 4.70%, 22.85% - 20.46%, and 2.85% - 2.29%, respectively. On the contrary, Ataç (2009) stated that heartwood of *Fagus oriestalis* had lower hot water, cold water, ethanol, 1% NaOH solubility values than those of sapwood. On the other hand, Ruiz-Aquino et al. (2015) noted that differences between sapwood and heartwood in terms of total extractive content were insignificant (5.29% and 5.35% in *Quercus laurina*, and 8.24% and 8.88% in *Quercus crassifolia*, respectively).

4. Conclusions

The differences in terms of fiber morphology and chemical composition between the heartwood and sapwood of red gum (*Eucalyptus camaldulensis* Dehnh.), black willow (*Salix nigra* Marsh.), and oriental beech (*Fagus orientalis* Lipsky) were evaluated. The results showed that sapwood of all sampled species had longer fibers and higher slenderness ratios compared to sapwood samples. In addition, sapwood samples had more holocellulose and less klason lignin, ethanol solubility, hot water solubility, and cold water solubility than those of sapwood samples. The other morphological and chemical properties of heartwood and sapwood fibers were varied depending on the tree species.

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