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Research Article

Comparison of *Pinus Pinea* Heartwood and Sapwood Pulps Obtained by Soda-Potassium Borohydride Method

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ABSTRACT

This study was aimed to compare pulp properties of *Pinus pinea* heartwood and sapwood pulp obtained from modified soda method and to investigate effects of potassium borohydride (KBH_4) used as a protective agent in cooking processes. Eight different cooking experiments were applied to the heartwood and sapwood samples by changing KBH_4 ratio. The effects of KBH_4 on some chemical, physical and optical properties of pulps obtained from cooking processes were determined. Due to high content of soluble extraction of heartwood, yield of heartwood pulp was approximately 24% lower than that of sapwood pulp. Using KBH_4 in pulping processes increased sapwood pulp yield, while it has a decreasing impact on heartwood pulp yield. Kappa number and viscosity values of sapwood pulps were better than those of heartwood pulps. With addition of KBH_4 to liquor there was increased kappa numbers of sapwood pulp in contrast to heartwood. In generally, physical properties of sapwood pulp are better than that of heartwood pulp. But, there are no significant difference in optical properties between heartwood and sapwood pulp. With no regard to the type of pulps used, the pulp optical properties were improved by using KBH4 in pulping processes.

Keywords: *Heartwood, Pinus pinea, Potassium borohydride, Pulp, Sapwood*

Soda-Potasyum Borhidrür Yöntemiyle Fıstık Çamı Öz ve Diri Odunlarından Elde Edilen Kağıt Hamurlarının Özelliklerinin Karşılaştırılması

ÖZET

Bu çalışmada fıstık çamı öz ve diri odunlarından elde edilen kağıt hamurlarının özelliklerinin karşılaştırılması ve pişirme işlemlerinde koruyucu ajan olarak kullanılan potasyum borhidrürün (KBH_4) kağıt hamuru özellikleri üzerine etkilerinin araştırılması amaçlanmıştır. KBH_4 oranı değiştirilerek öz ve diri odunlara sekiz farklı pişirme deneyi uygulanmıştır. Pişirme deneyleri sonucundan elde edilen kağıt hamurlarının bazı kimyasal, fiziksel ve optik özellikleri belirlenmiş ve KBH_4'ün bu özellikler üzerine etkileri incelenmiştir. Öz odunun içerdiği yüksek ekstraktif maddelerden dolayı kağıt hamur verimi diri odundan yaklaşık %24 daha düşük çıkmıştır. Pişirme işlemlerinde KBH_4 kullanımı ile diri odun hamur verimleri artarken öz odun hamur verimlerinde düşüşler meydana gelmiştir. Diri odunlardan elde edilen kağıt hamurlarının kappa numaraları ve viskozite değerlerinin öz odun hamurlarından daha iyi olduğu tespit edilmiştir. Pişirme çözeltisine ilave edilen KBH_4 diri odunu hamurlarının kappa numaralarını yükseltirken öz odunu hamurlarınkini düşürmüştür. Diri odun ve öz odun hamurlarının optik özellikleri arasında belirgin bir fark bulunmazken diri odun hamurlarının fiziksel özellikleri öz oduna nazaran daha yüksek çıkmıştır. Ayrıca, KBH_4 kullanımı her iki kağıt hamuru türünün de optik özelliklerini iyileştirmiştir.

Anahtar Kelimeler: *Diri odun, Fıstık çamı, Kağıt hamuru, Öz odun, Potasyum borhidrür*

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I. INTRODUCTION

The pulp industry in Turkey has very high production costs. As a consequence the investments in this field are normally shifted to other countries, as well as brought the existing ones to a halt. Under these circumstances the input costs, especially raw material, are quite high in the pulp and paper industry. The pulp industry in Turkey has very high production costs. As a consequence the investments in this field are normally shifted to other countries, as well as brought the existing ones to a halt. Under these circumstances the input costs, especially raw material, are quite high in the pulp and paper industry. Turkey paper industry imported all bleached cellulose pulps used in and 37% of consumed paper and board [1], [2]. With the cessation of China's exports, prices of pulp have increased and there have been increases in paper prices in countries importing pulp. Therefore, attempts on pulp production in Turkey have begun to increase. Entrepreneurs consider environmental regulations and costs in pulp production. Accordingly, it is more advantageous for them to choose the most environmentally friendly pulp production methods.

Today, the most used methods in chemical pulp production are kraft and soda pulping methods. The pulps obtained by kraft method are better quality but darker than those obtained by other methods. Soda method is suitable in terms of both cost and environmental reasons, and soda pulp is brighter than kraft pulp. The soda method is used in a limited way in pulp production from annual plant and some hardwood species, which can be easily pulped. Through these methods, less bleaching chemicals can also be used to gain high brightness. Chemical recovery in soda method is simpler than kraft method. In this method, as in kraft method, some additives such as anthraquinone and boron compounds are used to minimize degradation of carbohydrates. The end groups of carbohydrates are protected from peeling reactions by these chemicals as a protective agent in cooking processes [2], [3]. Almost all of the studies in pulp production have been used $NaBH_4$ as cooking additive, but use of KBH_4 is very new [3]–[6].

The cross section of wood consists of three parts: bark (xylem, phloem), wood (heartwood and sapwood) and pith. Although the pith is very small, it is dark colored part that is visible to eye, located in the center of the wood. Sapwood is physiologically active living xylem cells in the wood. Heartwood is the woody part that is formed around the pith, which is formed as a result of the protoplasm parts of living xylem cells losing their life activity after a certain period of time [7]. Although heartwood varies depending on the place of growing, tree age, soil, and climatic conditions, it usually begins to occur between the ages of 20-40. Heartwood differs from sapwood in terms of color and texture, and the absence of dark color does not mean that heartwood is absent [8]. However, although heartwood is physiologically dead, it is technically found in trees. Sapwood width is either measured in cm or expressed as annual number of rings. Age of the tree, place of growing and place where the tree is located in stand play an important role on width of sapwood. As the tree age increases, the rate of participation of sapwood width to the trunk volume decreases. At the end of this structural change, annual number of rings in sapwood remains constant. The transformation of sapwood into heartwood takes place with formation of some organic substances. These substances are called heartwood substances and have protective effects against fungi and insects [9].

Anatomical, morphological and chemical differences between heartwood and sapwood have a direct impact on pulp production [10]. Since heartwood is dark colored and contains high levels of soluble extractions, pulp produced has lower optical properties and has difficulties in pulp bleaching processes [11]. Heartwood has many disadvantages due to its extractives. The cooking solution penetration to heartwood is difficult due to its low permeability and therefore screen reject rate is increasing [12]. In addition, during paper production from heartwood pulp, problems such as paper breaks, clogging of screens and cleaners can be occurred [13]. In previous studies it was reported that heartwood pulps of *P. nigra* and *A. bornmuelleriana* [14], *E. globulus* [15], *P. pinaster* [11], *P. deltoids* [16], *A. melanoxylon* [17], and *L. leucocephala* [18] have lower pulp viscosity and yield than sapwood pulps. However, *Pinus pinea* heartwood and sapwood pulp properties have not been investigated yet.

In the present work, *P. pinea* heartwood and sapwood chips were cooked in four different cooking conditions with soda method. The KBH_4 compound, which is rarely used in pulping instead of $NaBH_4$, was added in cooking processes as protective agent. The aim is to test if there are the differences in pulp properties between the heartwood and sapwood of *P. pinea*.

II. MATERIALS AND METHODS

A. MATERIAL

Pinus pinea woods obtained from Kahramanmaraş province in Turkey were used as raw material in the study. According to relevant standards, 5 cm thick samples were taken from 15 cm above root, right in the middle of stem, and 15 cm below the crown. The holocellulose contents of the *P. pinea* heartwood and sapwood are 75.8% and 72.3%, respectively. Their solubility rates in ether (extractives) are 24.7% and 6.3%, respectively [19]. Chemicals were supplied by Merck KGaA Inc. and Sigma-Aldrich Inc.

B. PULPING PROCESS

The heartwood and sapwood of *P. pinea* were chipped into 3-5 cm length x 3-5 mm thickness, and those chips with no defects were used in cooking experiments. Modified soda (alkaline) method was used in pulping process. The effect of potassium borohydride (KBH_4) was evaluated as a protective agent in order to prevent carbohydrate degradations. Cooking conditions applied to the chips were detailed in Table 1. Eight cooking experiments for heartwood and sapwood were conducted by changing KBH_4 ratio.

Table 1. *Cooking conditions of P. pinea heartwood and sapwood*

Pulping Condition	Unit	Heartwood	Sapwood
Active Alkali Charge	%	23	22
KBH_4 Charge	%	0, 0.3, 0.5, 0.7	0, 0.3, 0.5, 0.7
Cooking Temperature	°C	160	160
Time to Maximum Temperature	min	40	40
Time at Maximum Temperature	min	110	110
Liquor-to-raw material Ratio	L/kg	5/1	5/1

Pulping experiments were carried out with an electrically heated-25 bar pressure resistant digester. In the pulping processes, 500 grams of oven-dried chips were filled by hand into the digester and discharged manually after the cooking process was completed. The pulps were washed with tap water on a screen (200-mesh) until black liquor was removed. Then, the washed pulps were passed through a shaker screen with 0.15 mm slotted to separate uncooked portions (screen rejects). The weights of the suitable (screened pulp) and unsuitable pulps (screen rejects) for paper production were determined and used in yield calculation. Kappa number and viscosity values of the screened pulps were determined according to TAPPI T236 and ISO 5351 standards, respectively.

C. PAPER PRODUCTION AND TESTS

The screened pulps were beaten to 50 ± 3 Schopper Riegler (°SR) freeness level according to TAPPI T200 by a laboratory type Hollander Beater. Test papers with 70 grammages ($gr.m^{-2}$) were produced from beaten pulps with Rapid-Kothen paper machine according to ISO 5269/2. Ten test papers were produced from pulp obtained from each cooking experiment and were subjected to physical and optical tests. Before the testing, the papers were conditioned in a conditioned room according to TAPPI T402 standard at 23 °C ± 1 temperature and 50 ± 1% relative humidity for 24 hour. Breaking length (TAPPI T 494), burst index (TAPPI T403), brightness (ISO 2469), whiteness (ISO 11476) and yellowness (ASTM E313) values of conditioned papers were determined according to mentioned standards.

III. RESULTS AND DISCUSSIONS

A. CHEMICAL PROPERTIES OF THE PULPS

Table 2 summarizes the results of kappa number, viscosity, degree of polymerization (DP), and pulps yields of *P. pinea* woods cooked different condition.

Table 2. *Some chemical properties of P. pinea heartwood and sapwood pulps*

Cooking Number	KBH_4 ratio *(%)*	Kappa Number	Viscosity *($cm^3.g^{-1}$)*	DP	Screened Yield *(%)*	Screen Reject *(%)*	Total Pulp Yield *(%)*
H-1	0.0	92.1^d	468^d	649^a	31.2^a	5.38^c	36.6^a
H-2	0.3	85.7^c	549^c	775^c	32.4^b	2.05^b	34.4^b
H-3	0.5	83.7^b	596^b	849^b	32.0^b	1.55^a	33.6^c
H-4	0.7	79.5^a	633^a	907^a	28.6^c	1.96^b	30.6^d
S-1	0.0	79.0^a	593^d	843^d	44.5^c	0.89^b	45.4^c
S-2	0.3	81.1^b	654^c	940^c	44.8^c	1.24^c	46.0^b
S-3	0.5	83.0^c	689^b	996^b	45.6^b	0.69^a	46.4^b
S-4	0.7	89.0^d	719^a	1043^a	47.9^a	0.67^a	48.6^a

*H and S refer to heartwood and sapwood, respectively. Mean values with different superscripts are significantly ($P<0.05$) different determined by a Duncan' multiple comparison test.

In KBH_4-free cooking of heartwood (H-1) and sapwood (S-1), significant differences were found in terms of chemical properties. Total pulp yield of sapwood is 24% higher than that of heartwood. This is simply due to the differences in the extractive contents of the woods. *P. pinea* heartwood has higher extractives than sapwood [20]. Moreover, Esteves et al. (2005) determined that pulp yield negatively correlated with extractive content [11]. Decreased pulp yield in the cases where heartwood used were also reported by Atac and Eroglu (2013), Lourenço et al. (2010) and Esteves et al. (2005) [11], [14], [15]. On the other hand, the high permeability raw material used in cooking results in less screen reject. Heartwood have also low permeability compared to sapwood [14], [21], [22]. As can be seen in Table 2, screen reject ratio of sapwood (S-1) had 83.5% lower than that of heartwood (H-1). For instance Mariana et al. (2005) reported that the screen reject ratio as a result of cooking was higher in the heartwood than that of sapwood of *Eucalyptus nitens* wood [23].

Having examined kappa numbers of the pulps obtained after cooking experiments, the sapwood pulps (S-1, 79.0) have the kappa numbers lower than those of heartwood pulps (H-1, 92.1). Heartwood has ray parenchyma cells, mostly filled with phenolic extractives having negative impact on chemical consumption during pulping process [24], [25]. Normally, the delignification rate of heartwood cooking is lower than that of sapwood, causing to increased residual lignin content in the pulp. In our study, the analyzed values of viscosity and DP values of heartwood and sapwood are in agreement with literature [14], [16], [26], [27].

The addition of KBH_4 to cooking liquor had a positive effect on yield in sapwood pulping, but it has a negative effect in heartwood pulping. While 0.7% KBH_4 addition in the sapwood cooking liquor increased pulp yield about 7.04%, it decreased pulp yield about 16.4% in heartwood cooking. Carbohydrate degradation that cause yield losses in pulping processes can be prevented with KBH_4 addition to cooking liquor. During pulping process, KBH_4 prevents the peeling reaction that may occur by reducing the carbonyl group to the hydroxyl group at the reducing ends of the cellulose chain. This reaction occurs not only in cellulose, but also in hemicellulose. Therefore, yield loss caused by peeling reaction is prevented and the yield of the pulp obtained increases [2], [5], [28]–[30] It is therefore thought that yield losses occurring in the heartwood cooking process are caused by the content of extractive substances.

Kappa number of the heartwood pulps have decreased with adding KBH_4 to cooking liquor. Kappa number of pulps produced 0.7% KBH_4-added to cooking liquor decreased about 13.7% compared to KBH_4-free pulps. However, the kappa numbers of sapwood pulp have increased in contrast to heartwood with adding KBH_4 to liquor. As seen in Table 2, the kappa number increased by approximately 12.7% with 0.7% KBH_4 added to the cooking liquor in sapwood pulping process. Gulsoy et al. (2016) reported that the addition of KBH_4 to cooking liquor increased the kappa numbers of maritime pine (*Pinus pinaster*) pulp [31]. Reductions in kappa numbers have previously been reported with the addition of another protective agent $NaBH_4$ in cooking processes [32]–[34]. The use of KBH_4 in cooking processes has a positive effect on viscosity and DP values of both heartwood and sapwood pulps. This could be due to fact that KBH_4 containing a boron compound protects carbohydrates (cellulose and hemicellulose) by preventing the peeling reaction occurring in alkali pulping process, and thus the viscosity and DP of the pulps are high [2], [5], [35], [36].

B. PHYSICAL AND OPTICAL PROPERTIES OF THE PULPS

The differences in physical and optical properties of the test papers produced from heartwood and sapwood pulps were compared in Table 3.

Table 3. *Some physical and optical properties of the heartwood and sapwood pulps*

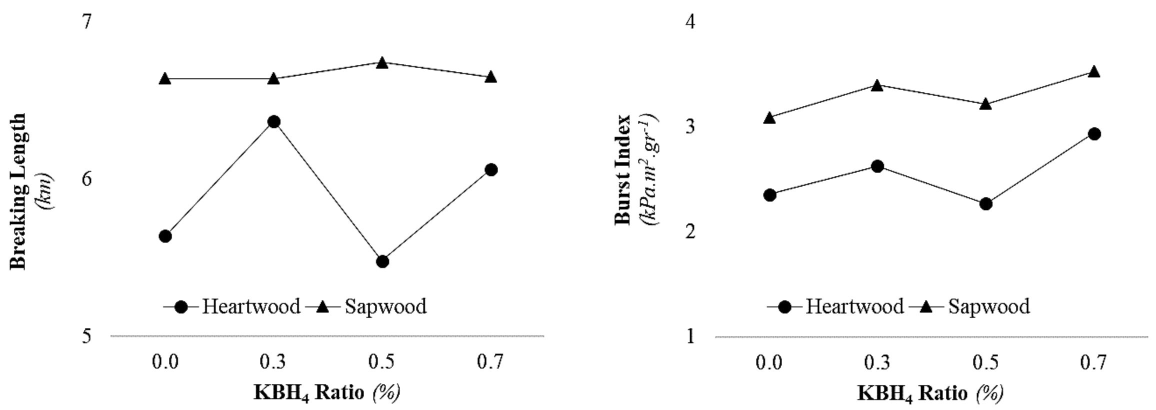
Cooking Number	**Breaking Length** *(km)*	**Burst Index** *($kPa.m^2.gr^{-1}$)*	**Brightness** *(ISO%)*	**Whiteness** *(ISO%)*	**Yellowness** *(E313)*
H-1	5.64^c	2.36^c	22.32^d	14.35^c	55.97^c
H-2	6.37^a	2.63^b	23.53^c	15.29^b	54.61^b
H-3	5.48^c	2.27^c	24.41^b	15.93^b	54.10^b
H-4	6.06^b	2.94^a	26.55^a	17.41^a	53.40^a
S-1	6.64^a	3.09^d	22.83^c	14.68^c	55.35^c
S-2	6.64^a	3.40^b	24.04^b	15.69^b	53.53^b
S-3	6.74^a	3.22^c	25.87^a	17.01^a	52.86^a
S-4	6.65^a	3.53^a	25.06^a	16.09^b	55.18^c

* Mean values with the same lower-case letters are not significantly different according to Duncan's mean separation test.

The breaking length (tensile strength) and burst strength, as strength properties are important physical properties of the papers. Breaking length of papers produced with heartwood pulps (H-1) were lower than that of sapwood pulps (S-1). Fiber length is an effective parameter on tensile strength of the paper. As fiber lengths used in paper production increase, tensile strength of the papers produced from these fibers increases. As shown in Table 1, sapwood fibers were normally longer than heartwood fibers. For this reason, the breaking length of the paper produced from sapwood pulps was about 17.7% higher than that of the heartwood. Saraeian et al. (2011) reported that tensile strength of papers produced with *Populus deltoides* sapwood were better than that of heartwood [37]. Furthermore, Gao et al. (2011a, 2011b) stated that tensile index of heartwood pulp were lower than that of sapwood [16], [26]. One of the factors affecting burst strength is fiber length and the other is internal bonding [38]. As in the tensile strength, burst strength of papers produced from sapwood pulp was also higher than that of heartwood. In our study, there were no significant differences between heartwood and sapwood pulps in terms of brightness, whiteness, and yellowness. The heartwood has a dark structure and it is expected that pulps obtained from that to be dark. However, both pulp types were found to have similar optical properties in this study. In previous studies, optical properties of sapwood pulp are better than those of heartwood pulp [14], [17], [27].

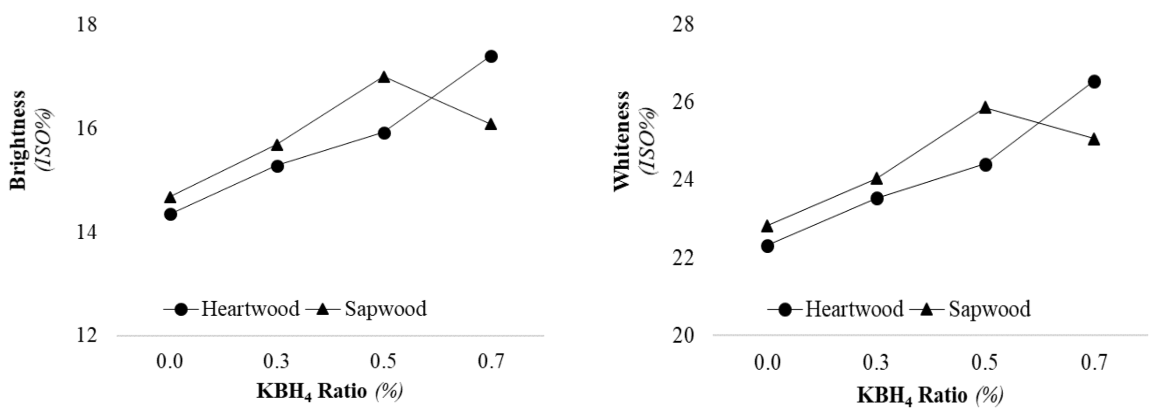
While KBH_4 did not show a significant effect on the breaking length of pulp produced from sapwood, it showed a positive effect for pulp produced from heartwood (Fig. 1a). Breaking length of pulp produced with 0.3% KBH_4-added cooking were 12.9% higher than that of KBH_4-free cooking. Increases in burst strengths of both the pulps occurred with the addition of KBH_4 to the cooking liquor (Fig. 1b).

Burst indices of the heartwood and sapwood pulps obtained from 0.7% KBH_4 added cooking have increased about 0.58 and 0.44 unit, respectively. As mentioned, KBH_4 protects carbohydrates during cooking and prevents them from degradation. Especially cellulose and its binding ability positively affect strength properties of pulp and paper. In this study, KBH_4 has a positive effect on the physical properties of pulps as seen in Table 3. Cicekler and Tutus (2019) reported supporting evidence on this positive effect of KBH_4 on physical properties of *Pinus brutia* pulps [5]. However, Gulsoy et al., 2016 reported that the physical properties of Maritime pine pulps decreased with using KBH_4 in cooking liquor [31].



(a) **(b)**

Figure 1. *Effects of KBH_4 on breaking length (a) and burst index (b) of P. pinea heartwood and sapwood pulps*



(a) **(b)**

Figure 2. *Effects of KBH_4 on brightness (a) and whiteness (b) of P. pinea heartwood and sapwood pulps*

While the brightness and whiteness values increased by addition KBH_4 in heartwood and sapwood pulping (Figures 2a and 2b), the yellowness values decreased. In a study conducted to determine the effect of KBH_4 on paper brightness values, it was reported that the brightness values increased with the addition of KBH_4 in maritime pine pulping process [31]. In another study with adding $NaBH_4$ to cooking liquor, boron compounds had a positive effect on brightness values [33]. Boron compounds have a positive effect on optical properties of pulps due to their bleaching properties, reported by many studies [5], [28].

IV. CONCLUSION

In conclusion, the pulp properties produced from *P. pinea* sapwood are better than heartwood. Heartwood pulp had lower yields, viscosities, breaking length and burst index in comparison to sapwood pulp. This is due to the fact that the extractive content of heartwood is higher than that of sapwood. The use of KBH_4 as a protective agent in pulping processes reduced the kappa number and yield of heartwood pulps while improved the polymerization degree, physical and optical properties. It has also positive effects on the chemical, physical and optical properties of the sapwood pulps except the kappa numbers. The high rate of heartwood in pulp production from *P. pinea* wood has negative effects on pulp quality, and this should be taken into account when harvesting *P. pinea* tree.

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