Chemical Composition, Fiber Morphology, and Kraft Pulping of Bracken Stalks (*Pteridium aquilinum* (L.) Kuhn)

Kemijski sastav, morfologija vlakana i sulfatni postupak proizvodnje celuloze od stabljika paprati (*Pteridium aquilinum* (L.) Kuhn)

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ABSTRACT • *In this study, kraft, kraft-$NaBH_4$, and kraft-KBH_4 pulp and paper properties of the bracken stalks (Pteridium aquilinum* (L.) Kuhn) *were determined. Also, the chemical composition and fiber properties of bracken stalks were evaluated. $NaBH_4$ and KBH_4 were separately added to cooking liquor by 0.5 %, 1 %, 1.5 %, and 2 % (oven dried wood). The boron compound-free kraft pulp were also made as control pulp. Fiber length and fiber width of bracken stalks were determined as 1.25 mm and 24 µm, respectively. Bracken stalks are composed of 73.34 % holocellulose, 32.55 % α-cellulose, and 30.79 % lignin. In addition, the pulp yield was increased with additions of both boron compounds, while kappa number was decreased. Also, highest strength increases determined in 0.5 % $NaBH_4$ added pulp. These results showed that bracken stalks can be used as a raw material for kraft pulp production.*

Keywords: *bracken, KBH_4, kraft, $NaBH_4$, Pteridium aquilinum* (L.) Kuhn., *paper properties*

SAŽETAK • *U radu su opisana istraživanja svojstava kraft, kraft-$NaBH_4$ i kraft-KBH_4 celuloze i papira proizvedenih od stabljika paprati (Pteridium aquilinum* (L.) Kuhn). *Ocijenjeni su kemijski sastav i svojstva vlakana stabljika paprati. Otopini za kuhanje odvojeno su dodavani $NaBH_4$ i KBH_4 u postotcima 0,5; 1; 1,5 i 2 % (u odnosu prema apsolutno suhom drvu). Kraft celuloza bez spojeva bora proizvedena je kao kontrolni uzorak. Duljina vlakana stabljika paprati iznosila je 1,25 mm, a širina 24 µm. Stabljike paprati sadržavaju 73,34 % holoceluloze, 32,55 % α-celuloze i 30,79 % lignina. Rezultati su pokazali da je uz dodatak obaju spojeva bora prinos celuloze povećan, dok je Kapa broj smanjen. Usto, rezultati su pokazali najveće povećanje čvrstoće za celulozu dobivenu dodatkom 0,5 % $NaBH_4$. Na temelju dobivenih rezultata može se zaključiti da se stabljike paprati mogu upotrebljavati kao sirovina za proizvodnju kraft celuloze.*

Ključne riječi: *paprat, KBH_4, sulfatni postupak, $NaBH_4$, Pteridium aquilinum* (L.) Kuhn., *svojstva papira*

[1] Authors are associated professor and M.Sc. at Faculty of Forestry, Forest Products Engineering, Bartin University, Bartin, Turkey.
[1] Autori su izvanredni profesor i magistar znanosti Sveučilišta u Bartinu, Šumarski fakultet, Odjel za industrije na bazi šuma, Bartin, Turska.

1 INTRODUCTION
1. UVOD

Bracken (*Pteridium aquilinum* (L.) Kuhn), a weed with a height of 30-200 cm, is the fifth most distributed common weed species of the world. Bracken is widely distributed from the Equator to the northern parts of Europe, in Central Asia, China and Japan, from central South America to subarctic Canada. It can easily grow and spread on many types of soil (Vetter, 2009).

Lignocellulosic biomass are included in wood and non-wood biomass. Wood-based lignocellulosic biomass, main raw material of pulp production, consists of hardwood and softwood species. Non-wood lignocellulosic biomass, such as wheat straw, rice straw, cotton stalks, canola stalks, sugarcane bagasse, switchgrass and reed, has been an important fiber resource for pulp production in countries with a shortage of wood raw material. The main differences between woody and non-wood lignocellulosic biomasses are their chemical compositions and physical properties (Zhu and Pan, 2010). The usability of a raw material in pulp production initially depends on these properties.

Global paper and paperboard production in 2000 and 2015 was 324.6 million ton and 406.3 million ton, respectively (FAOSTAT, 2016). Increasing paper production causes the decrease in forest resources. Non-wood lignocellulosic biomass is abundantly available, low cost, and easy to process. Also, it has a short growth and harvest period. For this reason, it is introduced as a potential raw material for pulp and paper production (Tye *et al.*, 2016). However, in 2015, pulp production from non-wood lignocellulosic biomass was only 12.3 million ton (FAOSTAT, 2016). Many studies have examined the utilization of alternative raw materials in the pulp and paper industry. The utilization as an alternative raw material in paper industry of fruit trees, such as orange tree pruning (Gonzáléz *et al.*, 2011), olive tree pruning (Requejo *et al.*, 2012), pomegranate tree pruning (Gülsoy *et al.*, 2015), white mulberry (Gençer *et al.*, 2013), common hazelnut (Gençer and Özgül, 2016), wild cherry (Gençer and Gül Türkmen, 2016), was previously evaluated. On the other hand, pulp and paper properties of many non-wood plant species were determined by several authors (Deniz *et al.*, 2004; Shatalov and Pereira, 2006; Çöpür *et al.*, 2007; Akgül and Tozluoğlu, 2009; Shakhes *et al.*, 2011; Gençer, 2015; Gençer and Şahin, 2015). However, there is no published report on using bracken stalks in pulp and paper production. The aim of this study was to evaluate the suitability of bracken stalks for papermaking. For this purpose, the kraft, kraft-$NaBH_4$, and kraft-KBH_4 pulp properties of bracken stalks were evaluated. The chemical composition and fiber morphology of bracken stalks (*Pteridium aquilinum* (L.) Kuhn) were also determined.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Plant material and sample preparation
2.1. Biljni materijal i priprema uzoraka

The bracken (*Pteridium aquilinum* (L.) Kuhn) were collected from the Bartin province of Turkey. The roots and leaves of bracken were removed and only the stalks were used. The stalks were chopped to 3-5 cm. Bracken stalks were air-dried and stored in dry conditions.

2.2 Chemical analysis
2.2. Kemijska analiza

The standard methods were used in the main chemical analyses of bracken. The sample preparation (TAPPI T 257), holocellulose (Wise and Karl, 1962), α-cellulose (Han and Rowell, 1997), klason lignin (TAPPI T 222), ash (TAPPI T 211), ethanol solubility (TAPPI T 204), cold and hot water solubility (TAPPI T 207), and 1 % NaOH solubility (TAPPI T 212) were carried out according to relevant standard methods. Three repetitions were made for each experiment.

2.3 Fiber analysis
2.3. Analiza vlakana

Bracken stalk samples were macerated according to chlorite method (Spearin and Isenberg, 1947). After maceration, the samples were agitated to obtain individual fibers (Berlyn and Miksche, 1976). 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.

2.4 Pulping and handsheet properties
2.4. Proizvodnja celuloze i svojstva papira

The kraft pulps made from bracken stalks were prepared under the following conditions: active alkali as Na_2O 25 %, sulfidity 30 %, liquor/wood ratio 5:1, pulping temperature 170 °C, heating time to 170 °C 90 min., and time at temperature 75 min. The same pulping conditions were applied to 0.5 %, 1 %, 1.5 %, and 2 % $NaBH_4$ and KBH_4 added samples. In pulping, laboratory-type 15-L electrically-heated rotary digester was used. In order to remove the black liquor, the pulps were washed and disintegrated. The rejects were retained by a Somerville-type pulp screen with a 0.15-mm slotted plate (TAPPI T 275). All pulps were beaten according to TAPPI T 200 to 25 °SR in a Valley Beater for comparison in the same conditions. The kappa number, screened yield and freeness levels of all pulps were determined according to TAPPI T 236, TAPPI T 210, and ISO 5267-1, respectively. Ten handsheets (75 g/m^2) were formed with a Rapid-Kothen Sheet Former (ISO 5269-2). The handsheets were conditioned according to TAPPI T 402. The tensile index, tensile energy absorption (TEA), and stretch (ISO 1924), burst index (TAPPI T 403), tear index (TAPPI T 414), and brightness (TAPPI T 525) of the handsheets were measured using the relevant standard methods.

2.5 Statistical analysis
2.5. Statistička analiza

All data was performed using SPSS software. The data belonging to the kraft and kraft-$NaBH_4$, and kraft-KBH_4 pulp properties of the bracken stalks were analyzed with analysis of variance (ANOVA). The effects of boron compound addition on paper properties were evaluated statistically. All pair wise multiple comparison procedures were performed using Duncan's test ($p<0.05$). The same letter in figures denotes that there were no statistically significant differences between the groups.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Chemical composition
3.1. Kemijski sastav

The chemical composition of a lignocellulosic raw material has been an important factor in evaluating its suitability for pulp production. The high holocellulose content and low lignin content have been desired for pulping to obtain higher pulp yield and lower kappa number. Also, pulp yield substantially depends on α-cellulose content of raw material used in pulp production. Table 1 shows the chemical composition of bracken stalks and its comparison with other lignocellulosic materials. As can be seen in Table 1, holocellulose content of bracken stalks was found to be 73.34 %, which is comparable with other annual plants, lower than eucalyptus (80.47 %) and aspen (82.68 %), and higher than maritime pine (70.21 %).

The α-cellulose content of raw material is an indicator of pulp yield. Fibers having higher α-cellulose content have been preferred for chemical pulp production. The α-cellulose content of bracken stalks (32.55 %) was lower than that of other annual plants. Also, it was lower than that of eucalyptus (52.79 %), aspen (49.03 %), and maritime pine (47.11 %), higher than that of cotton stalks (29.74 %).

The klason lignin content of bracken stalks (30.79 %) was higher than that of most annual plants. Also, it was higher than that of eucalyptus (19.96 %), aspen (16.69 %), and maritime pine (28.23 %). The high lignin content in raw material causes longer pulping time and increases the requirement of chemicals for delignification. Therefore, low lignin content in lignocellulosic raw material has been seen as an advantage for chemical pulping.

The solubility in water provides no useful information concerning the pulping value of the raw material, but it indicates the nature of certain constituents. In the cold water procedure, inorganic compounds, tannins, gums, sugars, and coloring matters are removed from raw material. The hot water procedure additionally removes starch. Treatment of raw material with 1

Table 1 Comparison of chemical composition of bracken stalks with other lignocellulosic species
Tablica 1. Usporedba kemijskog sastava stabljika paprati s drugim lignoceluloznim sirovinama

Raw materials *Sirovina*	**H** %	**AC** %	**KL** %	**ES** %	**HWS** %	**CWS** %	**NaOH** %	**Ash** %	**Literature**
Bracken stalks / *stabljike paprati*	73.34	32.55	30.79	7.19	15.45	14.60	28.08	7.45	This study
Cotton stalks / *stabljike pamuka*	62.79	29.74	23.79	-	17.91	15.05	48.88	4.99	Ateş *et al*., (2015)
Corn stalks / *stabljike kukuruza*	64.80	35.60	17.40	9.50*	14.8	-	47.10	7.5	Usta *et al*., (1990)
Sorghum stalks / *stabljike sirka*	71.0	40.3	13.0	15.3	19.7	15.1	47.1	-	Gençer and Şahin, (2015)
Sunflower stalks / *stabljike suncokreta*	66.9	37.6	10.8	4.07*	21.1	-	50.4	-	López *et al*., (2005)
Tobacco stalks / *stabljike duhana*	67.79	39.20	18.90	7.10*	20.02	16.85	42.00	6.86	Shakhes *et al*., (2011)
Canola stalks / *stabljike uljane repice*	72.1	39.9	20.6	1.6	8.6	7.5	29.1	5.8	Tofanica *et al*., (2011)
Wheat straw / *slama od pšenice*	69.84	42.07	22.33	9.33*	14.71	11.33	53.67	11.63	Ateş *et al*., (2015)
Barley straw / *slama od ječma*	66.01	38.70	19.47	-	16.25	11.01	56.25	10.97	Ateş *et al*., (2015)
Rice straw / *slama od riže*	70.85	35.62	17.2	3.52*	16.24	10.65	49.15	16.6	Tutus *et al*., (2004)
Rye straw / *slama od raži*	74.1	44.4	15.4	9.2*	13.0	10.2	39.2	3.2	Usta and Eroglu, (1988)
Kenaf / *kenaf*	71.8	46.75	17.30	4.28*	6.42	4.56	28.50	1.56	Dutt *et al*., (2009)
Hemp *konoplja*	86.93	63.77	6.59	4.23*	9.06	7.75	29.55	-	Gümüşkaya and Usta, (2006)
Sugarcane bagasse (depithed) *ostatci od prerade* šećerne *trske*	71.03	42.34	21.7	1.85*	7.42	3.02	32.29	2.10	Agnihotri *et al*., (2010)
Bamboo / *bambus*	68.33	47.67	26.00	3.68*	5.53	4.61	19.82	1.98	Moradbak *et al*., (2016)
Paulownia / *drvo paulovnije*	75.74	43.61	20.5	3.76	10.05	8.50	24.50	0.21	Ates *et al*., (2008)
Eucalyptus globulus	80.47	52.79	19.96	1.15*	2.84	-	12.42	0.57	Jiménez *et al*., (2008)
European aspen / *drvo jasike*	82.68	49.03	16.69	3.22	3.04	3.22	15.34	0.31	Gulsoy and Tufek, (2013)
Maritime pine / *drvo primorskog bora*	70.21	47.11	28.23	4.15	3.67	1.64	9.71	0.27	Gulsoy and Tufek, (2013)

H: Holocellulose / *holoceluloza*, AC: α-cellulose / α-celuloza, KL: Klason lignin / *klason lignin*, ES: Ethanol solubility / *topljivost u etanolu*, HWS: Hot water solubility / *topljivost u vrućoj vodi*, CWS: Cold water solubility / *topljivost u hladnoj vodi*, NaOH: 1 % NaOH solubility / *topljivost u 1 %-tnom NaOH*, *: Alcohol benzene solubility / *topljivost u alkohol benzenu*, **: Acetone solubility / *topljivost u acetonu*, ***: Ethanol-toluene solubility / *topljivost u etanol-toluenu*

% NaOH causes the extraction of low-molecular-weight carbohydrates consisting mainly of hemicelluloses and degraded cellulose. Also, this treatment is an indication of decay degree in the raw material. The high content of 1 % NaOH soluble of the rapeseed stalks and depithed stalks may probably affect the pulp yields and also the chemical consumption (Tofanica *et al.,* 2011). Ethanol, hot water, cold water, and 1 % NaOH solubility of bracken stalks were found to be 7.19 %, 15.45 %, 14.60 %, and 28.08, respectively. These values were similar to those of other nonwood lignocellulosic raw materials, higher than those of hardwood and softwood species. According to chemical composition results, bracken stalks can be evaluated as an alternative raw material for pulp production, although they have low α-cellulose content and high lignin content.

Like most nonwood fibers, the ash content of bracken stalks (7.45 %) was higher than that of wood species. Also, it was similar to that of corn stalks (7.5 %), lower than that of wheat straw (11.63 %), barley straw (10.97 %), and rice straw (16.6 %). High quantity of ash content in the lignocellulosic raw material can cause problems in pulping and papermaking processes. It can have a negative effect on chemical consumption during pulping, refining, and recovery of the cooking liquor.

3.2 Fiber morphology
3.2. Morfologija vlakana

One of the important factors in evaluating the suitability of pulp production of a raw material is its fiber morphological properties. The fiber properties directly affect the runnability on paper machine, strength and optical properties of sheet, response to refining, and fiber-water interactions, such as swelling and water retention of fibers. Long fibers are predisposed to form a porous and less uniform paper structure, coarse paper surface. Also, sheets of long fibers have higher strength properties than sheets of short fibers. On the other hand, fiber flexibility depends on lumen width and cell wall thickness of fibers. Thick-walled fibers have a negative effect on the folding endurance, burst and tensile index of paper, and a positive effect on tear index. Also, the paper obtained from thick walled fibers will be bulky, with a coarse surface, and will contain a large amount of void volume. However, thin-walled fibers provide uniform and denser paper structure.

Table 2 shows fiber properties of bracken stalks and comparison of these fiber properties with some lignocellulosic materials. As seen in Table 2, average fiber length of bracken stalks was 1.25 mm. Fiber length of bracken is similar than that of tobacco stalks (1.23 mm), kenaf (1.29 mm). Fiber length of bracken stalks was shorter than that of maritime pine (2.4 mm), bamboo (1.98 mm), sorghum stalks (1.77 mm), and sugarcane bagasse (1.59 mm), and longer than that of European aspen (1.1 mm), river red gum (0.80 mm), cotton stalks (0.83 mm), sunflower stalks (0.76 mm), rice straw (0.99 mm).

The fiber width of bracken stalks (24 μm) was close to that of corn stalks (24.30 μm), tobacco stalks (24.31 μm), and European aspen (23.9 μm). It was wider than that of cotton stalks (19.60 μm), sorghum stalks (19.53 μm), and rice straw (11.99 μm), and narrower than that of maritime pine (43.7 μm), paulownia (36.3 μm), and hemp (29.5 μm).

The lumen width of bracken stalks (10.30 μm) was similar to that of other nonwood fibers, such as corn stalks (10.70 μm) and wheat straw (10.54 μm). It was wider than that of sorghum stalks (6.60 μm), rice straw (5.26 μm), and river red gum (7.2 μm), and narrower than that of sunflower stalks (16.00 μm), tobacco stalks (15.38 μm), maritime pine (29.5 μm), paulownia (19.2 μm), and hemp (15.2 μm).

The cell wall thickness of bracken stalks (6.85 μm) was close to that of corn stalks (6.80 μm), sorghum stalks (6.46 μm) and European aspen (6.30 μm). It had thicker cell wall than cotton stalks (3.40 μm), canola stalks (5.26 μm), wheat straw (4.39 μm), and thinner cell wall than hemp (7.1 μm), paulownia (8.6 μm), and maritime pine (7.1 μm).

Slenderness ratio, felting ratio, and Runkel ratio, derived from fiber dimensions, have been used to determine the suitability of lignocellulosic raw materials for pulp production. High length to width ratio (slenderness ratio) of fibers results in well bonded paper. High slenderness ratio (>33) means that lignocellulosic raw material is suitable for pulp and paper production (Xu *et al*., 2006). Slenderness ratio of bracken stalks (52.8) was comparable with that of other raw materials as shown in Table 2. It was very close to slenderness ratio of river red gum (53.33), lower than that of sorghum stalks (90.37) and rice straw (86.04), and higher than that of sunflower stalks (30.06) and paulownia (22.58).

Flexibility ratio (lumen to fiber width) of fibers is classified as highly elastic fibers (>75), elastic fibers (50-70), rigid fibers (30-50), and very rigid fibers (<30). Flexibility ratio of bracken stalks (42.92) was comparable with that of other raw materials, as shown in Table 2. It was very close to flexibility ratio of corn stalks (44.03), lower than that of sunflower stalks (64.96) and maritime pine (67.5), and higher than that of sorghum stalks (33.79). According to flexibility ratio, the fibers of bracken stalks can be classified into rigid fibers group.

The flexible fibers, having low Runkel ratio (wall to lumen ratio, <1) are easily collapsible, and give large surface area for interfiber bonding. The rigid fibers, having high Runkel ratio (>1), have low bonded area. These fibers are least suitable for paper production due to their stiff fibers. Runkel ratio of bracken stalks (1.33) was comparable with that of other raw materials, as shown in Table 2. It was very close to Runkel ratio of rice straw (1.31), lower that that of switchgrass (1.5), and higher than that of maritime pine (0.5) and European aspen (1.1). Bracken stalks fibers are classified into rigid fibers category in terms of their Runkel ratio value. According to fiber morphology results, bracken stalks can be used for pulp production as

Table 2 Comparison of fiber properties of bracken stalks with other lignocellulosic biomasses
Tablica 2. Usporedba svojstava vlakana stabljike paprati s drugim vrstama lignocelulozne biomase

Raw materials *Sirovina*	**FL** (***L***) mm	**FW** (***D***) µm	**LW** (***d***) µm	**CWT** (***w***) µm	**SR** ***L/D***	**FR** ***(d/D)x100***	**RR** ***2w/d***	**Literature**
Bracken stalks / *stabljike paprati*	1.25	24.00	10.30	6.85	52.08	42.92	1.33	This study
Corn stalks / *stabljike kukuruza*	1.32	24.30	10.7	6.8	54.32	44.03	1.27	Usta *et al*., (1990)
Cotton stalks / *stabljike pamuka*	0.83	19.60	12.80	3.40	42.35	65.31	0.53	Ververis *et al*., (2004)
Canola stalks *stabljike uljane repice*	1.17	23.02	12.5	5.26	50.83	54.30	0.84	Enayati *et al*., (2009)
Sunflower stalks *stabljike suncokreta*	0.76	25.20	16.00	4.42	30.06	64.96	0.54	Omotoso and Owolabi, (2015)
Sorghum stalks / *stabljike sirka*	1.77	19.53	6.60	6.46	90.37	33.79	1.90	Albert *et al*., (2011)
Tobacco stalks / *stabljike duhana*	1.23	24.31	15.38	4.47	50.59	63.26	1.16	Shakhes *et al*., (2011)
Wheat straw / *slama od pšenice*	1.14	19.32	10.54	4.39	59	54.55	0.83	Kasmani and Samari-ha, (2011)
Rice straw / *slama od riže*	0.99	11.99	5.26	3.36	86.04	45.87	1.31	Kiaei, 2014
Sugarcane bagasse *ostatci od prerade šećerne trske*	1.59	20.96	9.72	5.64	75.86	46.37	1.16	Hemmasi *et al*., (2011)
Kenaf / *kenaf*	1.29	22.1	12.7	4.3	58.3	57.5	0.67	Ververis *et al*., (2004)
Switchgrass / *trave*	1.15	13.1	5.8	4.6	87.7	44.2	1.5	Ververis *et al*., (2004)
Miscanthus / *kineski šaš*	0.97	14.2	5.9	4.1	68.3	41.5	1.3	Ververis *et al*., (2004)
Hemp / *konoplja*	1.8	29.5	15.2	7.1	-	51.5	0.93	Dutt *et al*., (2008)
Musa paradisiaca (banana) *banana*	1.55	22	14.2	5.5	70.5	-	0.77	Goswami *et al*., (2008)
Bamboo / *bambus*	1.98	17.27	8.66	3.74	114.64	50.14	0.86	Moradbak *et al*., (2016)
Paulownia / *drvo paulovnije*	0.82	36.3	19.2	8.6	22.58	53.08	0.89	Ates *et al*., (2008)
River red gum / *drvo eukaliptusa*	0.80	15.0	7.2	4.0	53.33	48.0	1.11	Dutt and Tyagi, (2011)
European aspen / *drvo jasike*	1.1	23.9	11.4	6.3	46.0	47.7	1.1	Gulsoy and Tufek, (2013)
Maritime pine *drvo primorskog bora*	2.4	43.7	29.5	7.1	54.9	67.5	0.5	Gulsoy and Tufek, (2013)

FL: Fiber length / *duljina vlakna*, FW: Fiber width / *širina vlakna*, LW: Lumen width / *širina lumena*, CWT: Cell wall thickness / *debljina stanične stijenke*, SR: Slenderness ratio / *omjer vitkosti*, FR: Flexibility ratio / *omjer fleksibilnosti*, RR: Runkel ratio / *Runkelov omjer*

an alternative raw material, although they have relatively short and rigid fibers.

3.3 Pulp and paper properties
3.3. Svojstva celuloze i papira

Some properties of kraft, kraft-$NaBH_4$, and kraft-KBH_4 pulps are given in Table 3. The screened and total yields of bracken stalks control kraft pulp were relatively lower compared to conventional kraft pulp yield. This result can be explained by low α-cellulose content of bracken stalks (Table 2). On the other hand, pulp yields increased with the addition of $NaBH_4$ and KBH_4. The highest total pulp yield was found to be 36.9 % in 2 % KBH_4 added pulp. Similar total yield increases with the addition of $NaBH_4$ (Akgül *et al*., 2007; Istek and Gonteki, 2009; Tutus *et al*., 2010a,b; Gulsoy and Eroglu, 2011; Gümüşkaya *et al*., 2011; Erişir *et al*., 2015; Saraçbaşı *et al*., 2016) and KBH_4 (Gülsoy *et al*., 2016) have been reported by several authors. This result could be attributed to carbohydrate retention increases with the addition of boron compounds.

Residual lignin content of pulp was calculated by multiplying the kappa number by 0.13. Bleachable-grade chemical pulp (kraft or sulfite) usually contains about 1.5-4.5 % residual lignin (Gellerstedt, 2010). The residual lignin content of bracken stalks control pulp (45.1 x 0.13= 5.86 %) was slightly higher than that of bleachable-grade. This finding could be explained by high lignin content of bracken stalks (Table 2). The kappa numbers of pulps decreased with increasing boron compound addition ratios. This findng can be attributed to the acceleration of delignification rate with $NaBH_4$ and KBH_4 additions. The lowest kappa number was found to be 30.9 in 2 % KBH_4 added pulp. Several authors reported a positive effect of $NaBH_4$ on kappa number (Tutus *et al*., 2010b; Gulsoy and Eroglu, 2011; Gümüşkaya *et al*., 2011; Erişir *et al*., 2015; Saraçbaşı *et al*., 2016). On the other hand, Gülsoy *et al*., (2016) noted that KBH_4 had a negative effect on kappa number of maritime pine kraft pulp. $NaBH_4$, and KBH_4 additions to cooking liquor resulted in pulp viscosity increases. This result can be attributed to the prevention of degradation reactions by $NaBH_4$ and KBH_4 during cooking. The effect of KBH_4 on pulp properties was more prominent than the effect of $NaBH_4$. The highest pulp viscosity was found to be 841 cm^3/g in 2 % KBH_4 added pulp. Several authors reported a positive effect of $NaBH_4$ on pulp viscosity (Akgül and Temiz, 2006; Akgül *et al*., 2007; Istek and Özkan, 2008; Tutus *et al*., 2010b). On the contrary, pulp viscosity decreased with $NaBH_4$ addition (Çöpür and Tozluoğlu, 2008; Gulsoy and Eroglu, 2011; Gümüşkaya *et al*., 2011; Saraçbaşı *et al*., 2016).

Table 3 Some pulp properties of kraft, kraft-$NaBH_4$, kraft-KBH_4 pulps
Tablica 3. Neka svojstva kraft, kraft-NaBH4 i kraft-KBH4 celuloze

Cooking *Kuhanje*	Screened Yield *Prinos prosijavanja* %	Reject *Škart prosijavanja* %	Total yield *Ukupni prinos* %	Kappa number *Kapa broj*	Viscosity *Viskoznost* cm^3/g
Control	34.43	0.15	34.58	45.10	741
0.5 % $NaBH_4$	34.67	0.17	34.84	43.70	788
1 % $NaBH_4$	36.54	0.17	36.71	42.90	779
1.5 % $NaBH_4$	36.45	0.19	36.64	40.90	795
2 % $NaBH_4$	36.20	0.38	36.58	39.80	812
Control	34.43	0.15	34.58	45.10	741
0.5 % KBH_4	34.84	0.24	35.08	38.00	808
1 % KBH_4	34.94	0.06	35.00	35.20	772
1.5 % KBH_4	36.47	0.30	36.77	36.50	829
2 % KBH_4	36.75	0.15	36.90	30.60	841

The comparison of kraft pulp properties of bracken stalks with other lignocellulosic raw materials is presented in Table 4. Bracken stalks had lower screened yield and brightness, and higher kappa number than other nonwood species. This result can be attributed to low α-cellulose and high lignin content of bracken stalks.

At the similar pulp freeness levels, handsheet strength properties of bracken stalks kraft pulp were determined to be comparable with hardwood and nonwood papermaking raw materials. Tensile index (68.69 N·m/g) of handsheets of bracken stalks kraft pulp at 25 ºSR freeness level was higher than that of European aspen kraft pulp at 30 ºSR freeness level (61.13 N·m/g), kenaf kraft pulp at 30 ºSR freeness (50.23 N·m/g), sweet bamboo kraft pulp at 25 ºSR freeness (63.01 N·m/g), and lower than switchgrass kraft pulp at 30 ºSR freeness level (74.48 N·m/g).

At the similar pulp freeness levels, tear index of handsheets of bracken stalks kraft pulp (7.12 mN·m^2/g) was higher than kraft pulps of thistle stalks (6.7 mN·m^2/g), switchgrass (6.67 mN·m^2/g), European aspen (6.38 mN·m^2/g), and river red gum (6.6 mN·m^2/g). Also, it was lower than kenaf (10.30 mN·m^2/g). Burst index of handsheets of bracken stalks kraft pulp (2.98 kPa·m^2/g) were similar to kenaf kraft pulp (2.94 kPa·m^2/g). It was lower than kraft pulps of nonwood (thistle stalks and switchgrass) and hardwood (European aspen and river red gum) species (Table 4).

The effect of $NaBH_4$ and KBH_4 additions on tensile index of handsheets is given in Figure 1. Tensile index increased with $NaBH_4$ and KBH_4 additions except for 1.5 % $NaBH_4$ and 1 % KBH_4 added pulps ($p<0.05$). The highest tensile index was determined in 0.5 % $NaBH_4$ added pulp with 74.01 N·m/g. Increased tensile index can be attributed to increasing hemicellulose retention in boron compounds added pulps. Higher hemicellulose content results in an increase of pulp strength. Gulsoy and Eroglu (2011) noted that tensile index increased in European black pine kraft pulp, and Gümüşkaya *et al*., (2011) noted that it increased in stone pine AS-AQ pulp with $NaBH_4$ addition. On the contrary, some authors noted that $NaBH_4$ had a negative effect on tensile index of handsheets (Akgül *et al*., 2007; Çöpür ve Tozluoğlu, 2008; Istek and Özkan, 2008).

Table 4 Comparison of kraft pulp properties of bracken stalks with other lignocellulosic raw materials
Tablica 4. Usporedba svojstava kraft celuloze od stabljika paprati s drugim lignoceluloznim sirovinama

	Bracken stalks *Stabljike paprati* (1)	**Kenaf** *Kenaf* (2)	**Thistle stalks** *Stabljike čička* (3)	**Switchgrass** *Trave* (4)	**Sweet bamboo** *Bambus* (5)	**European aspen** *Jasika* (6)	**River red gum** *Eukaliptus* (7)
Cooking type / *Tip kuhanja*	Kraft	Kraft	Kraft	Kraft	Kraft	Kraft	Kraft
Screened yield, % *Prinos prosijavanja,* %	34.43	51.0	40.00	42.74	46.32	53.8	43.9
Kappa number / *Kapa broj*	45.1	30.2	15.0	14.01	12.54	12.8	24.4
Brightness, % / *Sjajnost*, %	16.29	-	-	29.30	19.67	27.78	25.9
Freeness level (ºSR) / *Razina slobode*	25	30	24	30	25	35	25
Tensile index, N·m/g *Vlačni indeks,* N·m/g	68.69	50.23	63.6	74.88	63.01	61.13	60.2
Tear index, mN·m^2/g *Indeks cijepanja,* mN·m^2/g	7.12	10.30	6.7	6.67	-	6.38	6.6
Burst index, kPa·m^2/g *Indeks pucanja,* kPa·m^2/g	2.98	2.94	3.4	4.16	3.19	4.71	3.2

1: This study, 2: Dutt *et al*., (2009), 3: Gominho *et al*., (2001), 4: Madakadze *et al*., (1999), 5: Kamthai and Puthson, (2005), 6: Gulsoy and Tufek, (2013), **7:** Khristova *et al*., (2006).

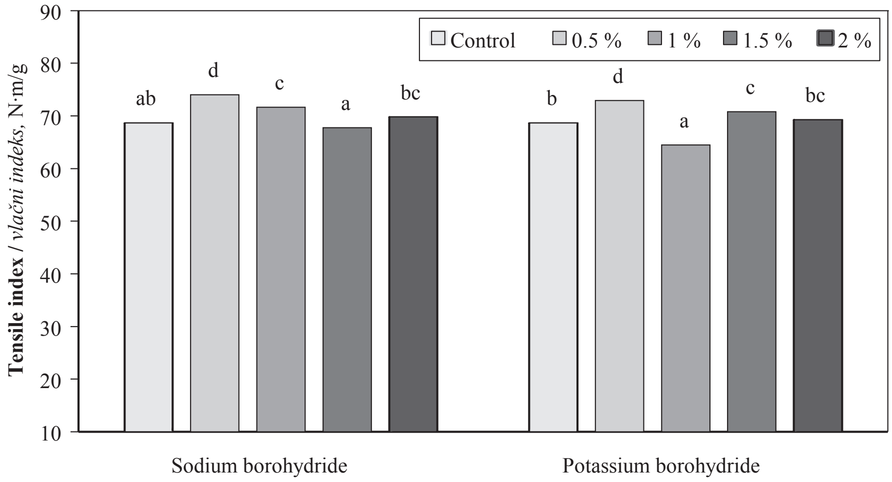

Figure 1 Effect of $NaBH_4$ and KBH_4 additions on tensile index of handsheets
Slika 1. Utjecaj dodavanja $NaBH_4$ i KBH_4 na vlačni indeks uzoraka papira

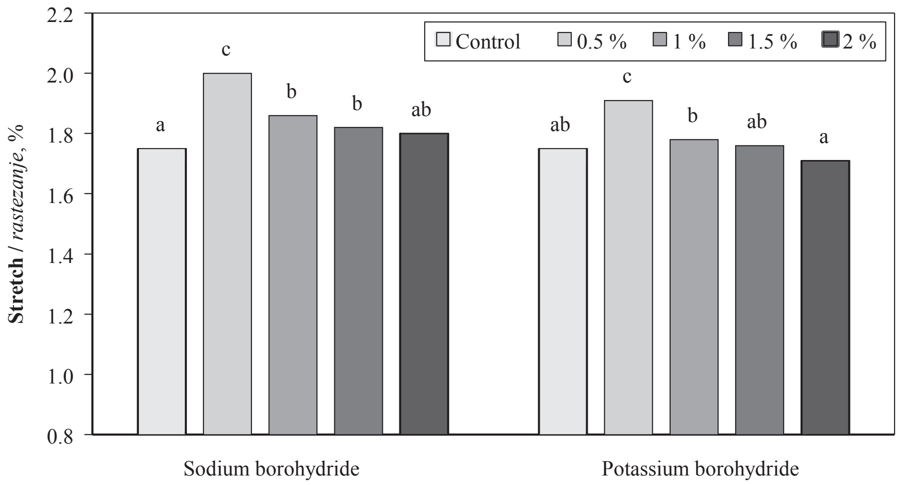

Figure 2 Effect of $NaBH_4$ and KBH_4 additions on stretch of handsheets
Slika 2. Utjecaj dodavanja $NaBH_4$ i KBH_4 na rastezanje uzoraka papira

The effect of $NaBH_4$ and KBH_4 additions on stretch values of handsheets is given in Figure 2. The stretch values of handsheets increased with $NaBH_4$ and KBH_4 additions except for 2 % KBH_4 added pulp ($p<0.05$). The highest stretch value was determined in 0.5 % $NaBH_4$ added pulp with 2.00 %. Istek and Gonteki (2009) noted that $NaBH_4$ addition to maritime pine kraft cooking caused losses in stretch values of handsheets.

The effect of $NaBH_4$ and KBH_4 additions on TEA values of handsheets is given in Figure 3. The TEA val-

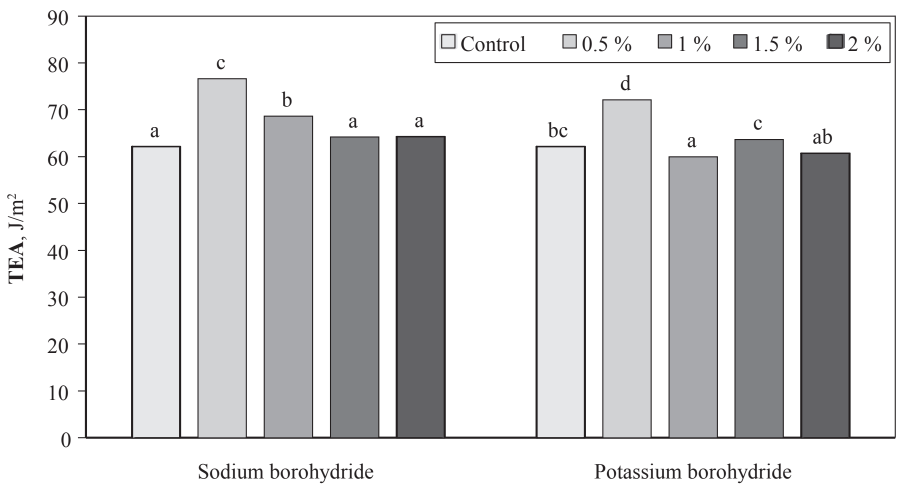

Figure 3 Effect of $NaBH_4$ and KBH_4 additions on TEA of handsheets
Slika 3. Utjecaj dodavanja $NaBH_4$ i KBH_4 na TEA uzoraka papira

ues of handsheets increased with $NaBH_4$ and KBH_4 additions except for 1 % and 2 % KBH_4 added pulps ($p<0.05$). The highest TEA value was determined in 0.5 % $NaBH_4$ added pulp with 76.66 J/m^2. Similar TEA losses were reported by Istek and Gonteki (2009).

The effect of $NaBH_4$ and KBH_4 additions on tear index of handsheets is given in Figure 4. Tear index decreased with $NaBH_4$ and KBH_4 additions ($p<0.05$). Tear index losses can be attributed to higher pulp yield of $NaBH_4$ and KBH_4 added pulps. Increasing in pulp yield causes the decrease of the fiber per unit weight of oven-dried pulp. The lowest tear index was determined in 2 % $NaBH_4$ added pulp with 5.82 mN·m^2/g. Similar tear index losses were reported in earlier studies (Akgül *et al*., 2007; Çöpür and Tozluoğlu, 2008; Istek and Gonteki, 2009; Gulsoy and Eroglu, 2011; Gümüşkaya *et al*., 2011).

The effect of $NaBH_4$ and KBH_4 additions on burst index of handsheets is given in Figure 5. Burst index increased with $NaBH_4$ and KBH_4 additions except for 1 % KBH_4 added pulp ($p<0.05$). The highest burst index was determined in 0.5 % KBH_4 added pulp with 3.27 kPa·m^2/g. Burst index of European black pine kraft pulp (Gulsoy and Eroglu, 2011) and stone pine AS-AQ pulp (Gümüşkaya *et al*., 2011) increased with 1 % $NaBH_4$ addition. On the contrary, Çöpür and Tozluoğlu (2008) noted in Brutia pine kraft pulp and Istek and Gonteki (2009) in maritime pine that $NaBH_4$ had a negative effect on burst index of handsheets.

The effect of $NaBH_4$ and KBH_4 additions on brightness of handsheets is given in Figure 6. Brightness increased with $NaBH_4$ and KBH_4 additions ($p<0.05$). This result can be attributed to lower kappa number of $NaBH_4$ and KBH_4 added pulps than that of control pulp (Table 3). The highest brightness was determined in 2 % KBH_4 added pulp with 20.16 %. Similar brightness increases were reported in earlier studies (Akgül *et al*., 2007; Çöpür and Tozluoğlu, 2008; Istek and Gonteki, 2009; Gulsoy and Eroglu, 2011).

4 CONCLUSIONS
4. ZAKLJUČAK

Chemical composition analysis showed that bracken stalks had higher holocellulose and lignin, and lower α-cellulose content than those of other lignocel-

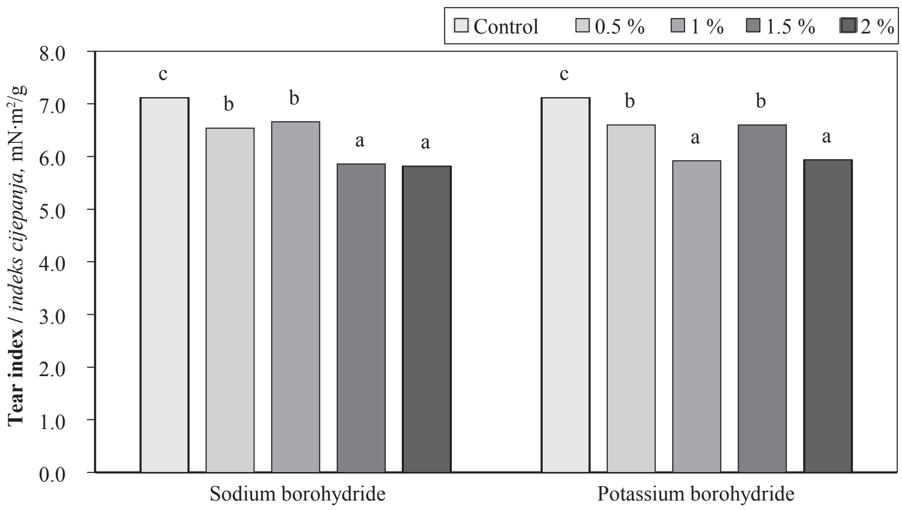

Figure 4 Effect of $NaBH_4$ and KBH_4 additions on tear index of handsheets
Slika 4. Utjecaj dodavanja $NaBH_4$ i KBH_4 na indeks cijepanja uzoraka papira

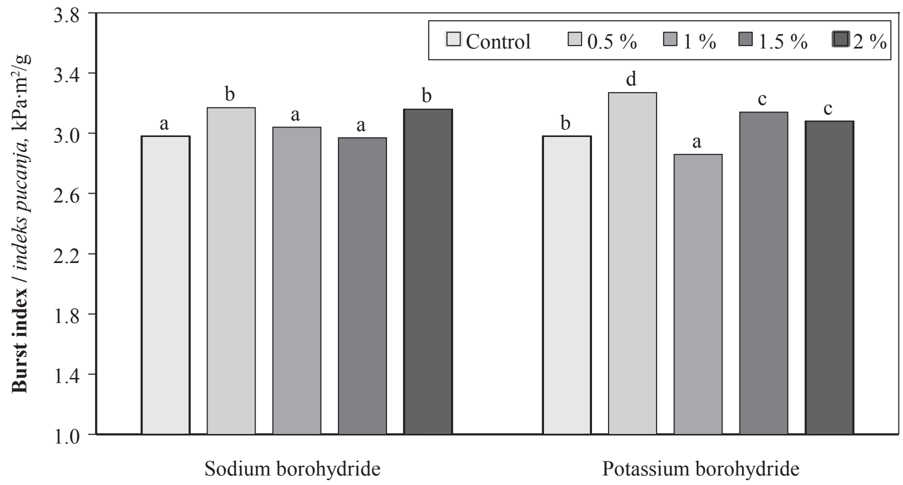

Figure 5 Effect of $NaBH_4$ and KBH_4 additions on burst index of handsheets
Slika 5. Utjecaj dodavanja $NaBH_4$ i KBH_4 na indeks pucanja uzoraka papira

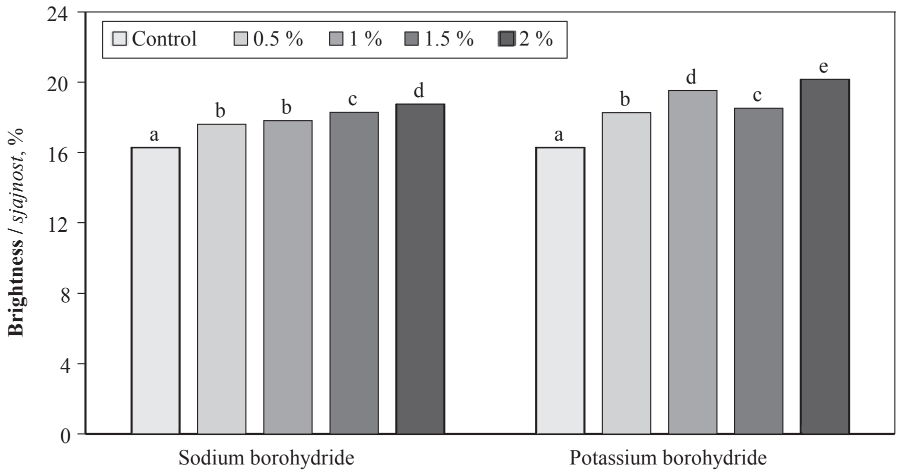

Figure 6 Effect of $NaBH_4$ and KBH_4 additions on handsheet brightness
Slika 6. Utjecaj dodavanja $NaBH_4$ i KBH_4 na sjajnost uzoraka papira

lulosic nonwood resources. The fiber properties of bracken stalks were similar to the fibers of other nonwood resources and aspen. Kraft pulp properties of bracken stalks were determined to be comparable with those of hardwoods (aspen and eucalyptus) and common nonwood papermaking raw materials. $NaBH_4$ and KBH_4 additions caused the increase of pulp yield and decrease of kappa number. Also, strength properties of bracken stalks kraft pulp increased with $NaBH_4$ and KBH_4 additions except for tear index. $NaBH_4$ and KBH_4 had a positive effect on pulp brightness. Consequently, the bracken stalks can be used as a fiber source for paper production.

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Corresponding address:

Assoc. Prof. SEZGIN KORAY GÜLSOY, Ph.D.

Faculty of Forestry, Forest Products Engineering
Bartin University
Bartin, TURKEY
e-mail: sgulsoy@bartin.edu.tr
szgngulsoy@yahoo.com