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A study on the effects of potassium borohydride in pulp and paper production from black locust (*Robinia pseudoacacia* L.) wood

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Abstract: In this study, the effects of potassium borohydride (KBH_4) added to the cooking liquor (white liquor) in pulp and paper production from black locust (*Robinia pseudoacacia* L.) woods on the chemical, physical and optical properties of the pulp were investigated. Soda (NaOH) method, which is one of the environmentally friendly cooking methods, was used in the pulp production from *R. pseudoacacia* woods. By adding 0, 0.3, 0.5 and 0.7% KBH_4 to the white liquor, four different cooking experiments were carried out. The yields, kappa numbers and viscosity values of the pulps were measured with the relevant standards. Test papers were produced from each pulp and their physical and optical properties were determined. With 0.7% KBH_4 added to the cooking solution, the total yield and viscosity increased by 9.54% and 10.4%, respectively compared to KBH_4-free pulp, while the kappa number decreased by 9.48%. As a result of statistical analysis, it has been observed that there is no significant difference between the physical and optical properties of the papers produced from KBH_4-free and KBH_4-added pulps. Consequently, it was concluded that adding KBH_4 to the white liquor in the pulp production from *R. pseudoacacia* wood with soda method, positively affected the chemical properties of the pulps and did not have a significant effect on the physical and optical properties.
Keywords: Black locust, Potassium borohydride, Pulp, Soda method

Yalancı akasya (*Robinia pseudoacacia* L.) odunundan kağıt hamuru ve kağıt üretiminde potasyum borhidrürün etkileri üzerine bir çalışma

Özet: Bu çalışmada, yalancı akasya (*Robinia pseudoacacia* L.) odunlarından kağıt hamuru ve kağıt üretiminde pişirme çözeltisine (beyaz çözelti) eklenen potasyum borhidrürün (KBH_4) kağıt hamurunun kimyasal, fiziksel ve optik özellikleri üzerindeki etkileri araştırılmıştır. *R. pseudoacacia* odunlarından kağıt hamuru üretiminde çevre dostu pişirme yöntemlerinden biri olan Soda (NaOH) yöntemi kullanılmıştır. Beyaz çözeltiye %0, 0.3, 0.5 ve 0.7 KBH_4 ilave edilerek dört farklı pişirme deneyi gerçekleştirilmiştir. Kağıt hamurlarının verimleri, kappa sayıları ve viskozite değerleri ilgili standartlara bağlı kalınarak ölçülmüştür. Her bir deneyden elde edilen kağıt hamurlarından test kağıtları üretilerek fiziksel ve optik özellikleri belirlenmiştir. Beyaz çözeltiye eklenen %0.7 KBH_4 ile toplam verim ve viskozite, KBH_4 içermeyen kağıt hamurlarına göre sırasıyla %9.54 ve %10.4 artarken, kappa sayısı %9,48 azalmıştır. İstatistiksel analizler sonucunda KBH_4 içermeyen ve KBH_4 katkılı kağıt hamurlarından üretilen kağıtların fiziksel ve optik özellikleri arasında önemli bir fark olmadığı görülmüştür. Sonuç olarak, yalancı akasya odunlarından soda yöntemi ile kağıt hamuru üretiminde pişirme çözeltisine KBH_4 ilave edilmesinin kimyasal özellikleri olumlu yönde etkilediği, fiziksel ve optik özellikler üzerinde belirgin bir etki göstermediği sonucuna varılmıştır.
Anahtar kelimeler: Yalancı akasya, Potasyum borhidrür, Kağıt hamuru, Soda yöntemi

1. Introduction

Mechanical, semi-chemical, chemical and biological methods are used to produce pulp and paper. Chemical pulping is one of the most preferred methods in the world. It can be classified as sulphate (Kraft), soda and sulphite methods (Smook, 1992). Kraft method is one of the most used methods in pulp production and kraft pulp is stronger and darker. In the soda pulping known as an environmentally friendly method, the strengths of the pulp are lower than that of kraft pulps, while the pulp color is lighter (Kirci, 2006). Chemical recovery in soda method is simpler than kraft method. As one of the main chemicals used in the kraft pulping, Na_2S, mercaptans and H_2S gases are released into the atmosphere during cooking and recovery, unpleasant odors occur. In addition, it has an abrasive feature during cooking. Therefore, many factories established as environmentalists prefer to use the soda method (Tutus and Cicekler, 2016; Istek and Ozkan, 2008).

Less bleaching chemicals can be used to obtain high brightness value from the pulp obtained by soda method (Misra, 1973; Eroglu, 1981). However, compared to the kraft method, the paper quality and yield are lower, and the cooking time is longer to reach the same delignification rate (Eroglu, 1981). One of the main reasons for using soda method instead of sulfate and sulfite method in pulp production from annual plants and hardwood species is that the cooking time of softwood species is very long (6-7 hours) in soda method. Another reason is that the strength properties of the pulp produced from softwood using the soda method are quite low compared to the other two methods. The fiber lengths of hardwoods are short and

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therefore their strength properties are lower than that of softwood fibers. However, papers with high opacity, better air permeability, soft and smooth surface are produced from the pulps obtained from hardwood (Smook, 1992).

With the recently developing technologies, progress has been made in terms of efficiency in soda cooking. The main reasons are minimizing the secondary peeling reaction by using low temperature and alkali ratio in cooking processes. However, it should be kept in mind that regardless of the methods used to increase the yield, it may adversely affect the strength properties of the pulp (Gulsoy et al., 2016; Cicekler and Tutus, 2019). When the temperature reaches 100 °C in alkali pulping, peeling reactions begin. The peeling reaction starts at the reducing end parts of the polysaccharide chain, separating the monomers one by one from the main chain. In this phase (primary peeling), decreases in polymerization degree and yield occur (Smook, 1992; Kirci, 2006; Brännvall, 2018). For this reason, various chemicals are used in cooking processes to increase yield and improve pulp properties. These chemicals are generally used to stop or slow down the peeling reactions that occur in cooking under alkaline conditions such as soda and kraft. Because, with the peeling reaction, shortening appears in the cellulose chains and negative effects arise on the pulp yield and physical properties (Tavast and Brännvall, 2017; Cicekler and Tutus, 2019; Birinci et al., 2020).

During cooking, aldehyde end groups are suitable for reduction, but are not very economical. For this purpose, additives such as boron compounds and anthraquinone (AQ) can be used. However, yield improvement processes also increase the chemical cost. Many attempts have been made to stop the peeling reaction and it is still the subject of many studies (Akgul and Temiz, 2006; Istek and Ozkan, 2008; Gulsoy and Eroglu, 2011; Tutus et al., 2011; Tutus et al., 2015; Tutus and Cicekler, 2016; Akgul et al., 2018). In these studies, it has been reported that AQ and boron compounds have a positive effect on the pulp properties.

As seen in Table 1, sodium borohydride ($NaBH_4$) is one of the most studied boron compounds in improving pulp properties, and the use of potassium borohydride (KBH_4) is very recent. During the cooking process, KBH_4 prevents the peeling reaction that may occur by reducing the carbonyl group at the reducing ends of the cellulose chain to the hydroxyl group. This reaction is seen not only in cellulose but also in hemicelluloses. Therefore, the yield loss caused by the peeling reaction is prevented and the yield of the obtained pulp increases (Akgul and Temiz, 2006; Istek and Ozkan, 2008).

In this study, the effects of KBH_4 added to the white liquor on the properties of the pulps produced with using soda method from *R. pseudoacacia* wood were investigated.

2. Material and method

2.1. Material

Black locust (*R. pseudoacacia*) wood samples used in pulp production were procured as logs from Osmaniye Forestry Management Directorate. 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. Chemicals used in cooking processes were purchased from Merck KGaA Inc. and Sigma-Aldrich Inc.

2.2. Pulping and papermaking

R. pseudoacacia wood samples were chipped into 25-35 mm length x 3-7 mm thickness and cleaned from dust and contaminants. In order to be used in cooking trials, 500 gr oven dried chips were weighted and stored in polyethylene bags. Soda cooking method was used to obtain pulps from *R. pseudoacacia* chips and KBH_4 were added to white liquor to improve pulp properties. The pulping conditions applied to the *R. pseudoacacia* chips were presented in Table 2.

The chips were filled by manually into an electrically heated digester and the pulp slurry was poured onto a screen (200 mesh) after cooking was completed. The obtained pulps were washed until black liquor was taken away. The black solution-free pulps were sieved through a screen with 0.15 mm slotted to remove non-fibrous and uncooked portions. The suitable (screened) and unsuitable (screen reject) pulps for papermaking were weighted and yields were calculated.

The pulps obtained from *R. pseudoacacia* chips were mixed homogeneously at a certain density in a 10-liter capacity mixer before the test paper formation and the freeness level was determined using the Schopper Riegler tool according to the ISO 5267-1 (1999) method. After the pulps were beaten at 35±5 SR freeness level in a Hollander device, test papers with 70 (g/m^2) grammages were produced with Rapid-Kothen paper machine according to ISO 5269-2 (2014).

Table 1. Some studies investigating the effects of $NaBH_4$ and KBH_4 on pulp properties

Raw material	Pulping method	Literature
Betula pendula	Kraft-$NaBH_4$	Pettersson and Rydholm, 1961
Pinus radiata	Kraft-$NaBH_4$	Meller, 1963
Wheat straw	Kraft-$NaBH_4$	Tutus and Alma, 2005
Abies nordmanniana	Kraft-$NaBH_4$	Akgul and Temiz, 2006
Populus tremula	Kraft-$NaBH_4$	Istek and Ozkan, 2008
Pinus brutia	Kraft-$NaBH_4$	Copur and Tozluoglu, 2008
Picea orientalis	Kraft-$NaBH_4$	Tutus et al., 2010
Pinus nigra	Kraft-$NaBH_4$	Gulsoy and Eroglu, 2011
Wheat straw	Soda-$NaBH_4$	Tutus and Cicekler, 2016
Pinus brutia	Kraft-$NaBH_4$	Tutus et al., 2012
Diospyros kaki	Kraft-$NaBH_4$	Tutus et al., 2014
Prunus armeniaca	Kraft-$NaBH_4$	Tutus et al., 2016
Pinus pinaster	Kraft-KBH_4	Gulsoy et al., 2016
Pinus brutia	Kraft-KBH_4	Cicekler and Tutus, 2019
Pinus pinea	Soda-KBH_4	Erkan et al., 2020

Table 2. Pulping conditions applied to *R. pseudoacacia* chips

Pulping Condition	Unit	Value
Active alkali charge	%	20
KBH_4 charge	%	0, 0.3, 0.5, 0.7
Liquor-to-raw material	L/kg	5/1
Cooking temperature	°C	160
Time to maximum temperature	min	40
Time at maximum temperature	min	110

2.3. Determination of chemical, physical and optical properties

The kappa numbers and viscosity values as chemical properties of the pulps were measured according to TAPPI T236 (2013) and ISO 5351 (2010) standards, respectively. Test papers were conditioned according to TAPPI T402 (2013) standard at 23±1 °C and 50±1% relative humidity for a day. The physical, optical tests and standards applied to the papers were given in Table 3.

The tests indicated in Table 3 were applied to ten papers produced from each pulp and the effects of KBH_4 were examined using the averages of the data. Statistical analysis of the obtained data was performed with the SPSS statistical package. Data of chemical, physical and optical properties of the pulp were analyzed using a computerized statistical program to determine variance, and by applying the Duncan test at a $P \leq 0.05$ confidence level.

3. Results and discussion

3.1. Chemical properties of the pulps

The pulp yields, kappa numbers and viscosity values were given in Table 4.

By adding 0.7% KBH_4 to the white liquor, the screened and total pulp yields increased compared to the KBH_4-free pulp yields. Since boron compounds such as $NaBH_4$ and KBH_4 has the ability to stop or slow down the peeling reactions occurring in cellulose and hemicellulose chains, it increased the pulp yield (Istek and Gonteki, 2009; Cicekler and Tutus, 2019; Erkan et al., 2020). In Fig. 1, the screened yield increased as KBH_4 charge added to the white liquor increased by 8.69%.

Addition of KBH_4 into white liquor decreased the kappa number of the pulps. Since KBH_4 stops the peeling and increases the yield, the cellulose content of the pulp increases. The kappa number is generally used to estimate the lignin content in the pulp and to determine its bleachability (Correia et al., 2018). Considering the increase in the cellulose content decreased the lignin content compared to the whole pulp, the kappa numbers of the KBH_4-added pulps were found to be lower (Fig. 1). Compared to KBH_4-free pulps, the kappa number reduced by 5% with adding 0.7% KBH_4 to the cooking solution. Decreases in kappa numbers have been mentioned with the addition of boron compounds KBH_4 and $NaBH_4$ in pulping processes (Copur and Tozluoglu, 2008; Gulsoy and Eroglu, 2011; Istek and Gonteki, 2009; Cicekler and Tutus, 2019; Erkan et al., 2020). The viscosity of the pulp refers to the degree of polymerization (DP) of the cellulose. As mentioned earlier, the shortening of the cellulose chain naturally also stops, as KBH_4 stops the peeling reaction. Therefore, the DP of KBH_4-added pulps is higher than for that of KBH_4-free pulps (Fig. 1). Viscosity values of 0.7% KBH_4-added pulps were 10.4% higher than that of KBH_4-free pulps.

Some physical and optical properties of the pulps obtained from *R. pseudoacacia* chips with soda-KBH_4 method were given in Table 5 below.

Table 3. The physical and optical test and standards applied to the papers

Physical and optical tests	Standards
Breaking length (km)	TAPPI T494 (2006)
Burst index (kPa m^2/g)	TAPPI T403 (2015)
ISO whiteness (%)	ISO 11476 (2010)
ISO brightness (%)	ISO 2469 (2014)
Yellowness (E313)	ASTM E313 (2020)

Table 4. Some chemical properties of the *R. pseudoacacia* pulps.

Cooking no	KBH_4 charge (%)	Screened yield (%)	Screen reject (%)	Total yield (%)	Kappa number	Viscosity (cm^3/g)	DP
1	0.0	40.46c	0.42b	40.88c	23.2b	559c	790c
2	0.3	40.94c	0.38b	41.02c	25.6c	540c	760c
3	0.5	42.28b	0.29a	43.28b	23.4b	602b	858b
4	0.7	43.98a	0.79c	44.78a	21.0a	617a	881a

*DP refers to degree of polymerization. Mean values with the same lower-case letters are not significantly different at 95% confidence level (p>0.05) according to Duncan's mean separation test.

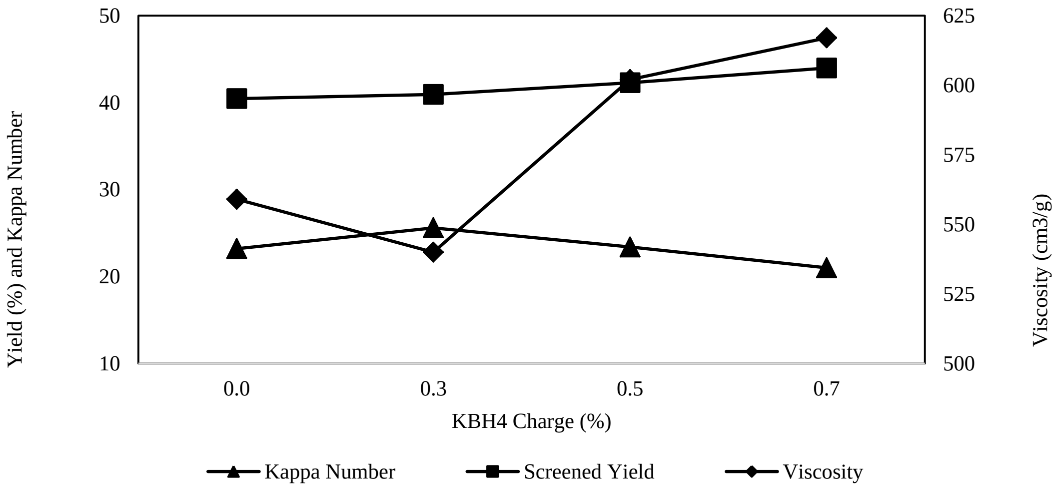

Figure 1. Effects of KBH_4 on some chemical properties of *R. pseudoacacia* pulps

Table 5. Some physical and optical properties of the *R. pseudoacacia* pulps.

Cooking no	KBH_4 Charge (%)	Breaking length (km)	Burst index (kPa.m^2/g)	Whiteness (ISO%)	Brightness (ISO%)	Yellowness (E313)
1	0.0	5.49a	2.87a	22.73a	16.50a	41.09a
2	0.3	5.35a	2.80a	21.53a	15.91a	40.27a
3	0.5	5.43a	2.84a	22.02a	16.12a	41.10a
4	0.7	5.47a	2.66a	23.11a	16.99a	40.56a

*Mean values with the same lower-case letters are not significantly different at 95% confidence level (p>0.05) according to Duncan's mean separation test.

According to Table 5 and statistical analysis, addition of KBH_4 to white liquor has no significant effect on the physical and optical properties of the *R. pseudoacacia* pulps. However, in many studies it was reported that $NaBH_4$ addition to cooking liquor increased the physical and optical properties of pulps. Gulsoy et al., (2016) determined that the physical properties of KBH_4-added *Pinus pinaster* pulps were found to be lower than those of KBH_4-free pulps. In a study conducted by Erkan et al., (2020) on the effects of using KBH_4 in the pulp production from *Pinus pinea* wood, they found that KBH_4 has no significant effect on physical properties. However, Cicekler and Tutus (2019) reported that using KBH_4 in pulp production from *Pinus brutia* woods has positive effects on physical properties. According to these results, it can be understood that using KBH_4 in pulp production does not have a clear effect on the physical properties of the pulp. Although the use of KBH_4 slightly improves the optical properties of pulps, it is understood from Table 5 that there is no statistically significant effect. While $NaBH_4$ has a bleaching effect on the pulp (Saracbasi et al., 2016), KBH_4 did not affect the optical properties in this study.

4. Conclusion

It was determined that the yields and polymerization degrees of the *R. pseudoacacia* pulps produced by the KBH_4 added soda method increased and the kappa numbers decreased. KBH_4, which was added to the solution in the pulp production from wood with using soda method, prevents the peeling reaction that may occur by reducing the carbonyl groups at the reducing ends of the cellulose chain to hydroxyl groups during cooking. Although KBH_4 prevented degradation of carbohydrates, it did not have a statistically significant effect on the physical properties of pulps. It was also observed that the addition of KBH_4 to the cooking solution in pulp production from *R. pseudoacacia* wood had no significant effect on the optical properties of the pulps.

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