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 (KBH₄) 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% KBH₄ 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% KBH₄ added to the cooking solution, the total yield and viscosity increased by 9.54% and 10.4%, respectively compared to KBH₄-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 KBH₄-free and KBH₄-added pulps. Consequently, it was concluded that adding KBH₄ 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 üretime potasyum borhidrürün etkileri üzerine bir çalışma

Özet: Bu çalışmada, yalancı akasya (Robinia pseudoacacia L.) odunlarından kağıt hamuru ve kağıt üretime pişirme çözeltisine (beyaz çözelti) eklenen potasyum borhidrür (KBH₄) kağıt hamurunun kimyasal, fiziksel ve optik özellikleri üzerindeki etkileri araştırılmıştır. R. pseudoacacia odunlarından kağıt hamuru üretime çeviren dostu pişirme yöntemlerinden biri olan Soda (NaOH) yöntemi kullanılmıştır. Beyaz çözeltiye %0, 0.3, 0.5 ve 0.7 KBH₄ ilave edilmişdir. Dört farklı pişirme deneyi gerçekleştirilmiştir. Kağıt hamurlarının verimleri, kappa sayları ve viskozite değerleri ortalama standartlarına bağlı hale gelmiştir. Her bir deneyden elde edilen kağıt hamurlarının test kağıtları üretilerek fiziksel ve optik özellikleri belirlenmiştir. Beyaz çözeltiye eklenen %0.7 KBH₄ ile toplam verim ve viskozite, KBH₄ içermeyen kağıt hamurlarına göre sırasıyla %9.54 ve %10.4 artarken, kappa sayısı %9.48 azalmıştır. İstatistiksel analiz sonucunda KBH₄ içeren ve KBH₄ katkılı kağıt hamurlarındadaki üretim kağıtlarının fiziksel ve optik özellikleri arasında önemli bir fark olmadığı görülmüştür. Sonuç olarak, yalancı akasya odunlarından Soda yöntemi ile kağıt hamuru üretiminde pişirme çözeltisine KBH₄ ilave edilmişsinin 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, Na₂S, mercaptans and H₂S 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; İstek 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 alkaline 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 Ergolu, 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 (NaBH₄) is one of the most studied boron compounds in improving pulp properties, and the use of potassium borohydride (KBH₄) is very recent. During the cooking process, KBH₄ 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 KBH₄ 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 KBH₄ 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/m²) grammages were produced with Rapid-Kothen paper machine according to ISO 5269-2 (2014).

### Table 1. Some studies investigating the effects of NaBH₄ and KBH₄ on pulp properties

| Raw material | Pulping method | Literature |
|--------------|----------------|------------|
| Betula pendula | Kraft-NaBH₄ | Pettersson and Rydholm, 1961 |
| Pinus radiata | Kraft-NaBH₄ | Meller, 1963 |
| Wheat straw | Kraft-NaBH₄ | Tutus and Alma, 2005 |
| Abies nordmanniana | Kraft-NaBH₄ | Akgul and Temiz, 2006 |
| Populus tremula | Kraft-NaBH₄ | Istek and Ozkan, 2008 |
| Pinus brutia | Kraft-NaBH₄ | Copur and Tözüoğlu, 2008 |
| Picea orientalis | Kraft-NaBH₄ | Tutus et al., 2010 |
| Pinus nigra | Kraft-NaBH₄ | Gulsoy and Ergolu, 2011 |
| Wheat straw | Soda-NaBH₄ | Tutus and Cicekler, 2016 |
| Pinus brutia | Kraft-NaBH₄ | Tutus et al., 2012 |
| Diospyros kaki | Kraft-NaBH₄ | Tutus et al., 2014 |
| Pinnus armeniaca | Kraft-NaBH₄ | Tutus et al., 2016 |
| Pinus pinaster | Kraft-KBH₄ | Gulsoy and Ergolu, 2011 |
| Pinus brutia | Kraft-KBH₄ | Cicekler and Tutus, 2019 |
| Pinus pinea | Soda-KBH₄ | Erkan et al., 2020 |

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

| Pulping Condition | Unit | Value |
|-------------------|------|-------|
| Active alkali charge | % | 20 |
| KBH₄ charge | % | 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 KBH₄ 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 ≤ 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% KBH₄ to the white liquor, the screened and total pulp yields increased compared to the KBH₄-free pulp yields. Since boron compounds such as NaBH₄ and KBH₄ 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 KBH₄ charge added to the white liquor increased by 8.69%.

Addition of KBH₄ into white liquor decreased the kappa number of the pulps. Since KBH₄ 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 KBH₄-added pulps were found to be lower (Fig. 1). Compared to KBH₄-free pulps, the kappa number reduced by 5% with adding 0.7% KBH₄ to the cooking solution. Decreases in kappa numbers have been mentioned with the addition of boron compounds KBH₃ and NaBH₄ in pulping processes (Copur and Tozhugu, 2008; Gulsoy and Eroğlu, 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 KBH₄ stops the peeling reaction. Therefore, the DP of KBH₄-added pulps is higher than for that of KBH₄-free pulps (Fig. 1). Viscosity values of 0.7% KBH₄-added pulps were 10.4% higher than that of KBH₄-free pulps.

Some physical and optical properties of the pulps obtained from R. pseudoacacia chips with soda-KBH₄ method were given in Table 5 below.

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

| Cooking no | KBH₄ charge (%) | Screened yield (%) | Screen reject (%) | Total yield (%) | Kappa number | Viscosity (cP/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|

Table 5. The physical and optical test and standards applied to the pulps

| Physical and optical tests | Standards |
|----------------------------|-----------|
| Breaking length (km)       | TAPPI T494 (2006) |
| Burst index (kPa m²/g)     | TAPPI T403 (2015) |
| ISO whiteness (%)          | ISO 11476 (2010) |
| ISO brightness (%)         | ISO 2469 (2014)  |
| Yellowness (E313)          | ASTM E313 (2020) |

Figure 1. Effects of KBH₄ on some chemical properties of R. pseudoacacia pulps

*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.
According to Table 5 and statistical analysis, addition of KBH\textsubscript{4} to white liquor has no significant effect on the physical and optical properties of the \textit{R. pseudoacacia} pulps. However, in many studies it was reported that NaBH\textsubscript{4} addition to cooking liquor increased the physical and optical properties of pulps. Gulsoy et al., (2016) determined that the physical properties of KBH\textsubscript{4}-added \textit{Pinus pinaster} pulps were found to be lower than those of KBH\textsubscript{4}-free pulps. In a study conducted by Erkan et al., (2020) on the effects of using KBH\textsubscript{4} in the pulp production from \textit{Pinus pinea} wood, they found that KBH\textsubscript{4} has no significant effect on physical properties. However, Cicekler and Tutus (2019) reported that using KBH\textsubscript{4} in pulp production from \textit{Pinus brutia} woods has positive effects on physical properties. According to these results, it can be understood that using KBH\textsubscript{4} in pulp production does not have a clear effect on the physical properties of the pulp. Although the use of KBH\textsubscript{4} slightly improves the optical properties of pulps, it is understood from Table 5 that there is no statistically significant effect. While NaBH\textsubscript{4} has a bleaching effect on the pulp (Saracbası et al., 2016), KBH\textsubscript{4} did not affect the optical properties in this study.

4. Conclusion

It was determined that the yields and polymerization degrees of the \textit{R. pseudoacacia} pulps produced by the KBH\textsubscript{4} added soda method increased and the kappa numbers decreased. KBH\textsubscript{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 carboxyl groups at the reducing ends of the cellulose chain to hydroxyl groups during cooking. Although KBH\textsubscript{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\textsubscript{4} to the cooking solution in pulp production from \textit{R. pseudoacacia} wood had no significant effect on the optical properties of the pulps.

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