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Effects of sodium borohydride on peroxide bleaching of *Pinus brutia* Ten. and wheat straw pulps

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Abstract: The addition of sodium borohydride ($NaBH_4$) to peroxide bleaching has been investigated for its effects on the bleaching yield, mechanical and optical properties of *Pinus brutia* and wheat straw pulps. The pulps were subjected to four different bleaching conditions by adding charges of $NaBH_4$ at 0%, 0.5%, 1.0%, and 1.5%, while keeping a constant hydrogen peroxide charge of 7%. The use of $NaBH_4$ in the peroxide bleaching processes of the two pulps increased the bleaching yields. The results show that the addition of $NaBH_4$ significantly improved the mechanical properties of the pulps, including tensile and burst indices. The optical properties of the pulps were also improved with the addition of $NaBH_4$. The increase in mechanical and optical properties can be attributed to the reduction of residual hydrogen peroxide and the removal of metal ions, while the decrease in yellowness is due to the removal of metal ions that can cause oxidative degradation of the pulp. The findings of this study suggest that the addition of $NaBH_4$ as an additive in peroxide bleaching is a promising approach to improve the mechanical and optical properties of *P. brutia* and wheat straw pulps, which can be further explored in future research.
Keywords: Peroxide bleaching, Sodium borohydride, *Pinus brutia*, Wheat straw

Sodyum borhidrürün *Pinus brutia* Ten ve buğday sapı hamurlarının peroksit ağartması üzerindeki etkileri

Özet: Peroksit ağartmasında sodyum borohidrür ($NaBH_4$) ilavesinin, kızılçam (*Pinus brutia*) ve buğday sapı hamurlarının ağartma verimi, mekanik ve optik özellikleri üzerindeki etkileri araştırılmıştır. Hamurlar, %7'lik sabit bir hidrojen peroksit oranında, %0, %0.5, %1.0 ve %1.5 oranlarında $NaBH_4$ eklenerek dört farklı ağartma koşuluna tabi tutulmuştur. Her iki hamur çeşidinin peroksit ağartma işleminde $NaBH_4$ kullanılması ağartma verimini artırmıştır. Ağartma işleminde $NaBH_4$ kullanımı kağıtların kopma ve patlama indisleri dahil olmak üzere hamurların mekanik özelliklerini iyileştirdiğini göstermiştir. $NaBH_4$ ilavesi ile hamurların optik özellikleri de iyileştirilmiştir. Mekanik ve optik özelliklerdeki artış, artık hidrojen peroksitin azalmasına ve metal iyonlarının uzaklaştırılmasına bağlanabilirken, sarılıktaki azalma, hamurun oksidatif bozunmasına neden olabilecek metal iyonlarının uzaklaştırılmasından kaynaklandığı düşünülmektedir. Bu çalışmanın bulguları, peroksit ağartmada bir katkı maddesi olarak $NaBH_4$ eklenmesinin, *P. brutia* ve buğday sapı hamurlarının mekanik ve optik özelliklerini iyileştirmek için umut verici olduğu sonucuna varılmıştır.
Anahtar Kelimeler: Peroksit ağartma, Sodyum borhidrür, *Pinus brutia*, Buğday sapı

1. Introduction

The bleaching process plays a crucial role in the production of paper and has a major effect on the financial wellbeing of the pulp and paper industry. The process is used to improve the brightness, softness, and purity of the pulp compared to unbleached pulp. The process is required to produce types of paper, including print, tissue, sanitary paper, and adsorption materials. Chemical pulp bleaching involves a multi-stage sequential process that uses two or more chemicals in order to achieve high brightness in the final product. The process is crucial in ensuring that the final product meets the desired standards for brightness, softness, and purity, which are important factors for many end-users (Sharma et al., 2020). The bleaching process of wood involves the interaction of chlorine and chlorine-based chemicals with the phenols, resins, and lignin contained in the wood, resulting in the production of dangerous pollutants. This method of chemical pulp bleaching has been widely used for many years due to its affordability and effectiveness in producing high-quality pulp. Chlorine and hypochlorite are the main chemicals used in this process, providing cost-effective and efficient bleaching results. However, the downside to this approach is the production of harmful pollutants that can have negative impacts on the environment and human health (Ashrafi et al., 2015; Gavrilescu et al., 2008).

The paper industry is under constant pressure from the market to improve the quality of its pulp. The demand for high-brightness bleached pulp is growing, and this has prompted the industry to find ways to improve the bleaching process. Hydrogen peroxide (H_2O_2) is a key chemical in the process of pulp bleaching and has the advantage of being environmentally friendly. However, the presence of transition metal ions, such as manganese, can have a significant impact on the effectiveness of the hydrogen

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peroxide bleach. To maximize the bleaching effect, it is necessary to remove these transition metal ions through a chelation step. This step is crucial in ensuring that the hydrogen peroxide bleach works to its full potential, resulting in a higher-quality, brighter pulp (Wang et al., 2004). Even when proper chelation procedures are followed to remove transition metal ions prior to the peroxide stage of the bleaching process, some residual transition metal ions can still remain. These leftover ions can have a negative impact on the hydrogen peroxide bleach by breaking down the extra hydrogen peroxide, reducing its effectiveness. This reduction in effectiveness can result in a lower-quality bleached pulp and can also lead to additional environmental impacts from the breakdown of hydrogen peroxide. To minimize these negative effects, it is important to continue to monitor and reduce the presence of residual transition metal ions in the peroxide stage of the bleaching process (de Oliveira Ribeiro et al., 2022; Houtman and Hart, 2022; Ni and Qiu, 2003; Schmitz et al., 2021; Wang et al., 2021). The addition of sodium borohydride ($NaBH_4$), a potent reducing agent, and stabilizers to the pulp slurry before the hydrogen peroxide (H_2O_2) stage can help reduce the negative impact of high valency metals, such as manganese, on the effectiveness of the hydrogen peroxide bleach. Sodium borohydride can effectively reduce manganese to its low valency form, thereby reducing manganese-induced hydrogen peroxide degradation (Wang et al., 2004). This reduction of hydrogen peroxide breakdown can result in improved bleaching results and a higher-quality end product. By using sodium borohydride and stabilizers in conjunction with hydrogen peroxide, the negative impact of residual transition metal ions can be minimized, leading to a more effective and efficient bleaching process.

The paper industry has been exploring more environmentally friendly methods in recent times, and one of the areas of interest is the use of boron compounds in the pulping and bleaching processes. The industry recognizes the need to reduce its impact on the environment and is seeking alternative methods that are both effective and eco-friendly. The use of boron compounds in the pulping and bleaching process has gained traction as a promising solution due to its potential to improve the process while reducing the environmental impact (Istek and Gonteki, 2009; Wang et al., 2004). The paper industry is continuously looking for ways to improve the quality of its products while minimizing its environmental footprint, and the use of boron compounds in pulping and bleaching processes may play an important role in achieving this goal. Among the various boron compounds being explored for use in the paper industry, sodium borohydride ($NaBH_4$) is the most preferred due to its affordability and ease of use. $NaBH_4$ has been widely adopted as a digester additive in the pulping process and has proven to be effective in improving the brightness and other physical properties of the pulp. This compound has gained popularity in the paper industry because of its ability to provide cost-effective and efficient results, making it an attractive alternative to traditional pulping and bleaching methods. By using $NaBH_4$ as a digester additive, the paper industry can achieve its goal of producing high-quality pulp while minimizing its impact on the environment (Gulsoy and Eroglu, 2011; Gümüşkaya et al., 2011; Istek and Gonteki, 2009; İstek and Özkan, 2008; Tutus and Cicekler, 2016). It is becoming apparent that $NaBH_4$ is increasingly being used as a reducing agent in multi-stage bleaching. $NaBH_4$ reduces carbonyl groups to alcohol groups and improves reflectivity over a wider range than sodium or zinc hydrosulfite (Çiçekler et al., 2022; Ferrandin-Schoffel et al., 2023; Martinsson et al., 2020).

It has been experimentally demonstrated that investigating the effect of $NaBH_4$ on the peroxide bleaching of spruce TMP (Thermomechanical pulp), it was stated that high brightness was obtained and less peroxide consumption was achieved compared to conventional methods (Zhibin et al., 2005). Another study by other authors found that bleaching of two different TMPs with $NaBH_4$-assisted hydrogen peroxide increased the optical properties of pulps and reduced the amount of peroxide required (Wang et al., 2004). This effect of $NaBH_4$ is supported by evidence (Li et al., 2015) from which the authors conclude that borohydride pretreatment is relatively effective in suppressing the effect of Fe(III) on peroxide degradation. In the same study, it was reported that the content of carbonyl groups in the pulp can be significantly reduced by treatment with $NaBH_4$ and the residual lignin becomes more stable. It was observed that chlorine dioxide and/or $NaBH_4$ increased brightness stability during cellulosic pulp processing (Rapson, 1960). A combination of borohydride and ozone treatments has been recommended to bleach and lighten cellulosic fibers (Wade, 1963). In a study of bleaching CTMP pulps, using 2% H_2O_2 in the first stage of bleaching and 1% $NaBH_4$ in the second stage increased brightness (Tutuş and Usta, 2004). $NaBH_4$-assisted bleaching processes have generally been applied to pulps made by mechanical processes. In this study, the effects of the optical and mechanical properties of $NaBH_4$ on peroxide bleaching of *Pinus brutia* wood and wheat straw pulps obtained by chemical methods were investigated.

2. Material and method

2.1. Material

The *Pinus brutia* wood samples used in this study were sourced from Ahir Mountain in Kahramanmaras, Turkey, while the wheat straws were harvested from Kahramanmaras, Turkey, at the end of the growing season. Table 1 below provides the production conditions and various properties of *P. brutia* and wheat straw pulps that are commonly used in the bleaching process. The table lists the relevant information and data that pertains to the production of these pulps, including the conditions under which they were produced and their key mechanical and optical properties. This information is critical for understanding the properties and behavior of the pulps during the bleaching process and for determining the optimal conditions for achieving the desired end results. By examining the data in Table 1, researchers, industry professionals, and other stakeholders can gain a deeper understanding of the pulps and their suitability for use in the bleaching process.

Table 1. Production conditions and some properties of pulps used in bleaching processes

Production conditions and some properties	*Pinus brutia*	Wheat straw
Active Alkali (%)	20	-
Sulfidity (%)	27	-
NaOH charge (%)	-	16
Cooking temperature (°C)	160	140
Time to maximum temp. (min)	40	40
Time at maximum temp. (min)	120	50
Liquor to raw material ratio	5/1	5/1
Brightness (ISO%)	25.6	28.7
Breaking length (km)	7.89	6.12
Burst index (kPa.m^2/g)	4.97	3.39

H_2O_2, $NaBH_4$, EDTA, $MgSO_4$, NaOH and Na_2SiO_3 chemicals were used in peroxide bleaching of *P. brutia* and wheat straw pulps. All these chemicals were purchased from Merck, a leading supplier of laboratory chemicals and reagents based in Darmstadt, Germany. Distilled water, which has been purified through a process of distillation, was used specifically in the bleaching process. This is because distilled water is free of impurities and minerals that may interfere with the bleaching chemicals and reactions. Tap water was used in the washing process to remove any remaining bleaching chemicals and impurities from the pulp, without introducing any additional impurities that may have been present in distilled water.

2.2. Peroxide bleaching of the pulps

The pulps were subjected to four different conditions as shown in Table 2. During all four conditions, the temperature and time of the bleaching process remained constant, as did the amount of H_2O_2 used. The only variable that was changed during the bleaching processes was the $NaBH_4$ charge.

Once the bleaching liquors outlined in Table 2 were prepared, the pulps were carefully placed into polyethylene bags. These bags, containing the pulps and bleaching liquor, were then immersed in a water bath. The temperature of the water bath was precisely regulated by a thermostat to ensure the bleaching process occurred at the desired temperature. Following the bleaching process, the pulps were removed from the water bath and thoroughly washed with both hot and cold water. This washing process continued until all residual chemicals were completely removed from the pulp. Once the washing was complete, the pulps were pressed to a dryness of 20-25%. To investigate the impact of $NaBH_4$ on the efficiency of the bleaching process, yield calculations were conducted following each individual bleaching treatment. Yield, in this context, refers to the amount of pulp that remained after the bleaching process. These calculations were used to evaluate the extent to which $NaBH_4$ impacted the bleaching efficiency of the pulps, and to compare the results obtained from different bleaching conditions.

Table 2. Peroxide bleaching conditions of *P. brutia* and wheat straw pulps

Peroxide bleaching conditions	Value
H_2O_2 charge (%)	7
NaOH charge (%)	9.33
EDTA charge (%)	0.5
Na_2SiO_3 charge (%)	2
$MgSO_4$ charge (%)	0.5
$NaBH_4$ charge (%)	0, 0.5, 1.0, 1.5
Temperature (°C)	75
Time (min)	60
Consistency (%)	12

2.3. Handsheet production and analysis

Pulps were refined to a Schopper Riegler (SR) level of 35±1 SR° in order to prepare the pulps for evaluation following the peroxide bleaching process. This was achieved through the use of a Hollander beater, in accordance with the guidelines outlined in ISO 5267-1 (1999). The refinement process helped to ensure that the pulps were of a consistent and uniform quality, which was important for accurate evaluation of their properties. Handsheets with a grammage of 80 (g/m^2) were prepared using a laboratory-type Rapid Kothen paper machine to assess the quality of the pulps obtained from the various peroxide bleaching treatments. The preparation of these handsheets followed the procedures set forth in ISO 5269-2 (2004), which helped to ensure consistency in the papermaking process.

The use of quantitative optical measurements allowed for precise characterization of the properties of the handsheets, providing valuable insights into the quality of the pulps obtained from the various bleaching treatments. Using a Datacolor Elrepho spectrophotometer, the brightness, whiteness, and yellowness index of the handsheets were measured quantitatively. These measurements were conducted in accordance with ISO 2470-1 (2016), ISO 11475 (2017), and ASTM E313-20 (2020) standards, respectively. The tensile (50 mm/min) and burst indices, which are crucial measures of paper strength properties, were determined using the ISO 1924-2 (2008-tensile strength) and ISO 2759 (2014-burst strength) standards, respectively. For each treatment condition of the handsheets, ten measurements were taken, and the resulting mean values were utilized.

3. Results and discussion

Figure 1 illustrates the results of the H_2O_2 bleaching process, including the corresponding yields and the effects of $NaBH_4$ on the pulp. The figure presents a visual representation of the data, allowing us to easily identify any patterns, trends or relationships between the variables being analyzed. The H_2O_2 bleaching yields, which reflect the amount of bleached pulp produced, are depicted graphically in Figure 1, providing a clear comparison of the bleaching yields between different treatments.

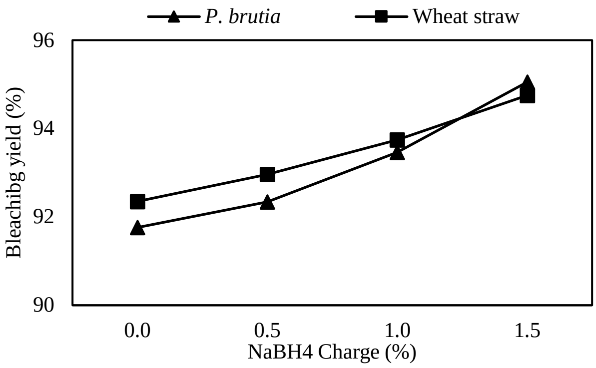


Figure 1. Effects of $NaBH_4$ addition on peroxide bleaching yield

By analyzing the information presented in Figure 1, we can draw conclusions about the effectiveness of H_2O_2 bleaching and the role of $NaBH_4$ in enhancing the bleaching process. The results of the study indicated that there was a positive correlation between the amount of $NaBH_4$ added to the bleach liquor and the corresponding yield values. One of the mechanisms through which $NaBH_4$ improves yield is by reducing peeling that can occur during the cooking/bleaching process. This is achieved by converting the carbonyl group on the reducing ends of both the cellulose and hemicellulose chains into a hydroxyl group, thus minimizing the loss of yield. As a result, the addition of $NaBH_4$ is effective in preventing yield losses and increasing the overall yield of the resulting pulp (Birinci et al., 2020; Istek and Gonteki, 2009; Tutus and Cicekler, 2016; Wigell et al., 2007a, 2007b).

The figure also indicates the comparison of the bleaching yield between wheat straw and *P. brutia*. The results of the study show that the bleaching yield of wheat straw was higher than that of *P. brutia*. This finding implies that wheat straw is better suited for H_2O_2 bleaching processes compared to *P. brutia*. This difference in bleaching yield can be attributed to several factors such as the cellulose content and structure of the materials and the presence of extractives and lignin in the raw materials (Andrade and Colodette, 2014; Laine et al., 1996; Mussatto et al., 2008). Overall, this observation underscores the importance of selecting the appropriate raw material for bleaching processes and optimizing the bleaching conditions for each material. Such considerations are critical to maximizing the efficiency and yield of the bleaching process, which can have significant economic and environmental implications.

Figure 2 provides an overview of the mechanical properties of the bleached pulps. The figure illustrates how the mechanical properties of the pulps are affected by the bleaching process and the addition of $NaBH_4$. The mechanical properties of the pulp are important indicators of its overall quality and suitability for various applications. Figure 2 presents information on key mechanical properties such as tensile strength and burst index which are commonly used to evaluate the strength and durability of paper products.

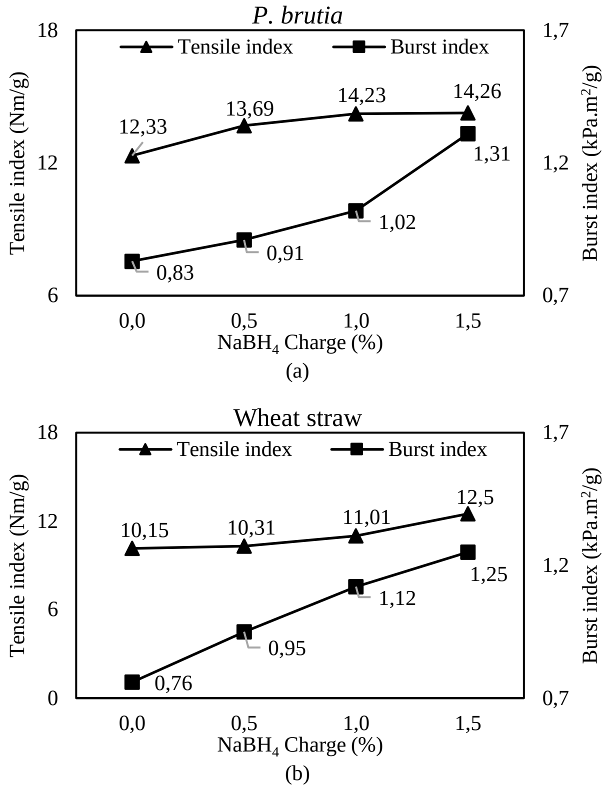


Figure 2. Effects of $NaBH_4$ addition during peroxide bleaching on mechanical properties. (a) *P. brutia* pulps, (b) Wheat straw pulps

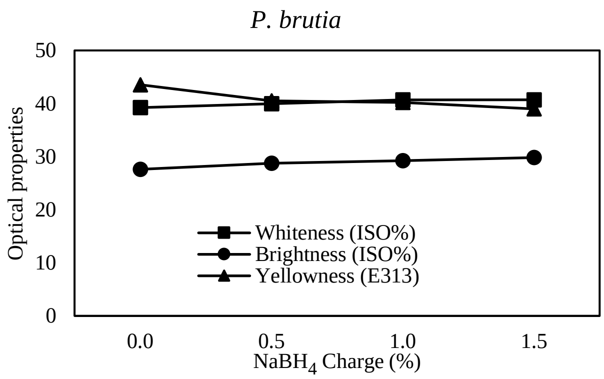
By analyzing the data presented in Figure 2, we can draw conclusions about the effect of bleaching and $NaBH_4$ on the mechanical properties of the pulp. This information can be useful in optimizing the bleaching process and developing high-quality pulp products that meet the desired mechanical specifications. Despite the primary purpose of pulp bleaching chemicals being to brighten or remove lignin, their effects are not limited to lignin alone. The chemicals used in pulp bleaching can also impact other components of the pulp, including carbohydrates such as cellulose and hemicellulose (Hannuksela et al., 2004; Schönberg et al., 2001). These compounds are important structural components of the pulp, and any alterations to their properties can have significant implications for the overall quality and properties of the resulting pulp products (Molin and Teder, 2002; Young, 1994). Therefore, a comprehensive understanding of the effects of bleaching chemicals on all pulp components is crucial in optimizing the bleaching process and developing high-quality pulp products.

The addition of $NaBH_4$ into the peroxide bleaching liquor has been demonstrated to have a positive impact on the mechanical properties of pulps. This improvement can be attributed to the reduction of residual hydrogen peroxide and the elimination of metal ions that may have been present in the bleaching process (Wang et al., 2004; Zhibin et al., 2005). When $NaBH_4$ is added to the peroxide bleaching process, it acts as a reducing agent, which aids in the decomposition of hydrogen peroxide into water and oxygen. This reaction leads to a decrease in the concentration of residual hydrogen peroxide, which can be detrimental to the mechanical properties of the pulp if left unreacted (Dang et al., 2007; Kordsachia and Patt, 1988; Rushdy et al., 2017).

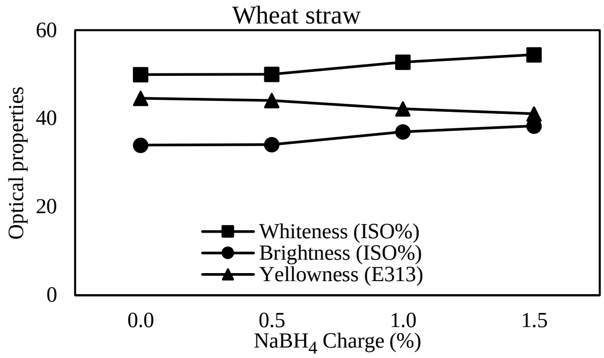
Additionally, $NaBH_4$ can effectively remove metal ions, such as copper and manganese, that may have been present in the pulp (Wang et al., 2004). These metal ions can cause oxidative degradation of the pulp, leading to a decrease in its mechanical strength (Sharma et al., 2020). By removing these metal ions, $NaBH_4$ helps to maintain the integrity of the pulp fibers, resulting in improved mechanical properties. Overall, the addition of $NaBH_4$ to peroxide bleaching liquor has been shown to be a promising approach to improve the mechanical properties of pulps.

The effect of $NaBH_4$ on the optical properties of peroxide bleached pulps has been evaluated and presented in Figure 3. The optical properties of the pulps, including brightness, whiteness, and yellowness, were measured and analyzed to assess the effectiveness of $NaBH_4$ in improving these characteristics.

The results depicted in Figure 3 show that the addition of $NaBH_4$ to the peroxide bleaching process has a positive effect on the optical properties of the pulps. The brightness of the pulp, which is a measure of the amount of light reflected from the surface (Wistara et al., 2015), is significantly increased with the addition of $NaBH_4$. This increase in brightness is attributed to the reduction of residual hydrogen peroxide and the elimination of metal ions that may have been present in the bleaching process (Tutuş and Usta, 2004; Wang et al., 2004; Zhibin et al., 2005). The addition of $NaBH_4$ to the bleaching liquor helps to reduce the concentration of residual H_2O_2 by providing electrons for its decomposition into water and oxygen (Dang et al., 2007; Li et al., 2015). This reaction leads to a more efficient removal of H_2O_2, resulting in a brighter and whiter pulp product. Furthermore, the yellowness of the pulp, which is an indicator of the degree of degradation of the pulp (Schmidt et al., 1995), is reduced with the addition of $NaBH_4$. This reduction in yellowness is attributed to the removal of metal ions that can cause oxidative degradation of the pulp, as well as the reduction of residual hydrogen peroxide. In summary, the addition of $NaBH_4$ to the peroxide bleaching process has a positive impact on the optical properties of the pulps, leading to improved brightness, whiteness, and reduced yellowness. These results suggest that $NaBH_4$ can be an effective additive in improving the overall quality of peroxide bleached pulps. The obtained data demonstrate that the use of 1.5% $NaBH_4$ provides the best results in the bleaching of *P. brutia* and wheat straw pulps.



(a)



(b)

Figure 3. Effects of $NaBH_4$ addition during peroxide bleaching on optical properties. (a) *P. brutia* pulps, (b) Wheat straw pulps

4. Conclusion

The addition of sodium borohydride ($NaBH_4$) to peroxide bleaching has been shown to have a significantly positive effects on the bleaching yield, mechanical and optical properties of *P. brutia* and wheat straw pulps. Using $NaBH_4$ as an additive in peroxide bleaching can effectively reduce the level of residual hydrogen peroxide and remove metal ions that may be present, which can cause yellowing and reduce the brightness of pulp fibers (Wang et al., 2004). The results of the study showed that the addition of $NaBH_4$ to the peroxide bleaching process led to an improvement in the mechanical properties of the pulps. This improvement can be attributed to the reduction in residual hydrogen peroxide and the elimination of metal ions that cause the breakdown of carbohydrates that may have been present in the bleaching process. The optical properties of the pulps, including brightness, whiteness, and yellowness, were also improved by the addition of $NaBH_4$. The increase in brightness and whiteness is attributed to the reduction in residual hydrogen peroxide and the removal of metal ions, while the decrease in yellowness is attributed to the removal of metal ions that can cause oxidative degradation of the pulp. Overall, the use of $NaBH_4$ as an additive in peroxide bleaching is a promising approach to improve the mechanical and optical properties of *P. brutia* and wheat straw pulps. This study provides valuable insights into the potential benefits of adding $NaBH_4$ peroxide bleaching, which can be further explored in future research.

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It is with heavy hearts that we acknowledge the passing of Ayşe Özdemir in Kahramanmaraş mega-earthquakes on February 6th, 2023. Ayşe was a respected and valued member of our team, and her contributions to our laboratory investigations will be deeply missed. Her passion for research and dedication to advancing our understanding of the field was evident in her work, and her positive energy and warm personality made her a pleasure to work with. We extend our heartfelt condolences to Ayşe's family and loved ones during this difficult time. Our thoughts and prayers are with them, as well as with all those affected by the earthquakes. We are grateful for the time we were able to spend working with Ayşe and will always remember her fondly.

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