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The Use of *Eucalyptus Grandis* Bark and Root as Raw Material in Pulp and Paper Production

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The suitability of *Eucalyptus grandis* bark and root for the pulp and paper industry was investigated. The bark of *E. grandis* was cooked using sodium borohydride ($NaBH_4$) in the kraft process , while the root of *E. grandis* was cooked using the soda-anthraquinone (AQ) process. Four different charges (0.0, 0.3, 0.5, 0.7%) of $NaBH_4$ and AQ used as catalysts were added to the cooking liquor used in the processes. The chemical, mechanical and optical properties of the produced pulps were investigated and characterized. The yields, viscosity values, kappa numbers, as well as the brightness, tensile, burst and tear indices of the pulps were determined. The yield (RP: 39.1%, BP: 36.8%), viscosity value (RP: 897 cm^3/g, BP: 650 cm^3/g) and the kappa numbers (RP: 90, BP: 50) of the pulps produced from the root (RP) were higher than those of the pulps produced from bark (BP). The catalysts generally affected all the pulp properties, improving the properties of BP and RP. It was concluded that *E. grandis* bark and root can be suitable for the pulp and paper industry.

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Introduction

The world population is growing rapidly, leading to higher product and resource consumption. This increases the demand for wood as a raw material. Using real wood to make fiber-based products can raise the cost of the product. Consequently, it would be more cost-effective to use as many non-wood or waste products as possible in the paper industry. Waste materials utilized in the forest sector include lignocellulosic and agricultural wastes. Grain stalks, straws, bushes, and wood bark are the most common non-wood elements used in the manufacture of pulp and paper [Gencer 2015]. The utilization of bark as a biomass for steam energy generation in factories is a prevalent practice, albeit accompanied by the undesirable consequence of air pollution during its combustion process. Much research has been done in this area to ascertain if using lignocellulosic waste is more cost-effective and environmentally friendly than disposing of the waste [Reis et al. 1987]. The wood from roots (known as briar root) has a high economic value, Furthermore, considering the fact that about 25% of all biomass comes from roots, one can see how

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important this output could be, especially when using economic allocation techniques [Cambria and Pierangeli 2012].

Roots are sections of trees that are often found deep underground. Their main functions are to anchor the tree to the ground as well as to absorb water and nutrients. The stump is the above-ground section of a tree trunk that is attached to the tree's underground roots [Foelkel 2014]. After forest harvest, the roots and stumps remain in the harvested region and create a physical obstacle to mechanical soil preparation for the next harvest [Pereira et al. 2013]. Forest reform consists of two main measures: trunk removal and tree stump removal [Segura et al. 2017]. The stumps and roots can be removed to enable future planting. Several studies have been carried out to develop stump/root harvesting methods and investigate the potential of this biomass, which is lignocellulosic material that is removed from the soil. A study of biomass production on eucalyptus plantations found that underground biomass accounted for about 13% of the total biomass produced at a site [Reis et al. 1987]. Forest leftovers have a high potential for energy generation; hence, roots and stumps taken from the soil are generally burned for energy production in mills that produce pulp, paper, particleboard, fiberboard, etc.

Depending on the type and diameter of the trunk, the prevalence of bark with wood is between 10% and 20% [Ferreira et al. 2015, Ogunwusi 2013]. The bark is extremely varied, both morphologically and chemically, and varies widely across species and within particular hardwood and softwood species [Argyropoulos 2001, Hemingway 1981]. The bark is a tissue complex outside the vascular cambium, composed mainly of phloem, periderm, and rhytidome, and normally contains both living and dead cells produced by the cambia [Ferreira et al. 2015]. Bark has more ash and longer fibers than wood, and is reported to have ten times the mineral content of wood [Jorge et al. 2000]. It is assumed that the cellulose and hemicellulose content of bark fibers corresponds to that of bark-free wood [Ogunwusi 2013]. Bark contains more extractives and fewer polyphenols, suberin and polysaccharides than wood [Ogunwusi 2013, Rosdiana et al. 2017]. For chemical or mechanical pulp, wood-based pulp and paper mills typically use bark-free hardwood or softwood. Manual or automatic debarking machines are used to remove the bark from the log. According to researchers, the debarking technique does not always remove the bark properly, and some bark remains on the tree trunk after debarking (Isokangas & Leiviska, 2005, Tripathi et al., 2020). Compared to identical pulps made from wood chips, the bark-derived pulp has a higher kappa number, higher drainage resistance, and higher consumption of bleaching chemicals, according to researchers [Fernandes et al. 2014]. It was discovered that wood chips with a higher proportion of bark have greater pulp strength and brightness [Brill 1985].

Eucalyptus wood is utilized in various sectors including greenhouse cultivation, the packaging industry, construction industry, pole constructions at piers, firewood and charcoal production, boat keels, plywood manufacturing, particleboard production, fiberboard construction, and lastly, in the pulp and paper industry [Özdemir et al. 2019, Özkurt 2002]. Eucalyptus, which has such a wide range of uses in industry, naturally has a large amount of waste such as bark and roots.

Because of the fast rise in paper consumption and demand, chemical pulp mills are trying to identify novel lignocellulosic raw materials for pulp production or to increase pulp output through the use of different cooking liquor additives [Kaur et al. 2018]. Cooking chemicals ($NaOH$, Na_2S, etc.) at a temperature of around 170 °C break down the lignin in the chemical pulping process. During the cooking process, there are carbohydrate losses that lead to a reduction in pulp yield. However, these losses can be prevented by oxidizing the reducing end of carbohydrates. In terms of cost and economy, increasing the yield in pulp production is of crucial importance for the pulp and paper sector. An increase in pulp yield can be achieved by modifying the kraft cooking process, for example, by adding anthraquinone (AQ) and boron compounds ($NaBH_4$, KBH_4). By utilizing these additives as a catalyst in cooking experiments, the end groups of carbohydrates are preserved from peeling reactions [Akgül et al. 2007, Akgül et al. 2018, Comlekcioglu et al. 2016, Copur and Tozluoglu 2008, Ersisir et al. 2015, Gulsoy and Eroglu 2011, Istek and Gonteki 2009, Tutus and Cicekler 2016].

The aim of this study was to investigate the suitability of *E. grandis* bark and roots, which are generally disposed of by incineration or rotting in the soil, as raw materials for pulp and paper manufacture. A secondary aim of this study was to evaluate the effects of sodium borohydride ($NaBH_4$) and AQ on the chemical, mechanical and optical properties of pulp and paper produced *E. grandis* bark and roots.

Material and methods

This study was conducted at Kahramanmaras Sütçü İmam University, Faculty of Forestry, Paper and Board Production Laboratory. The *Eucalyptus grandis* bark and root used in the study were obtained from study areas of the Eastern Mediterranean Forestry Research Institute. We recently conducted a study investigating the chemical components and fiber morphological properties of *Eucalyptus grandis* bark [Özdemir et al. 2019] and root given in Table 1. The chemical components and fiber morphological properties of the *Eucalyptus grandis* root were determined according to the standards mentioned in a previous study.

Table 1. Some chemical components and fiber morphological properties of *E. grandis* bark and root

Species / Chemical components	*E. grandis* bark [Özdemir et al. 2019]	*E. grandis* root	*E. grandis* wood [Gültekin 2014]	Hardwoods [Atchison 1987, Kırcı 2006]	Softwoods [Atchison 1987, Kırcı 2006]
Extractives (%)	12.4	5.26	3.30	1-6.2	1-5.8
%1 NaOH solubility (%)	21.1	22.4	14.9	12-25	8-10
Cold water solubility (%)	4.28	4.06	4.00	0.2-4	0.5-4
Hot water solubility (%)	9.34	7.98	5.60	1-8	1-5
Holocellulose content (%)	76.3	74.9	74.4	72-82	63-74
Cellulose content (%)	45.6	46.7	55.6	38-55	55-61
A-cellulose content (%)	38.6	44.3	49.8	-	-
Lignin content (%)	25.9	35.2	26.7	18-26	25-32
Ash content (%)	4.09	0.46	0.40	0.2-0.7	0.2-0.5
Fiber length (mm)	0.92	0.98	1.31	0.7-1.6	2.7-4.6
Fiber width (μm)	18.4	13.1	27.7	20-40	32.-43
Lumen diameter (μm)	4.10	4.13	12.9	-	-

The pulps were made by cooking *E. grandis* bark using $NaBH_4$ in the kraft process and *E. grandis* root using the soda-AQ process. The cooking conditions for the bark and root are given in Table 2. 500 g of oven dried raw material was used for each cooking experiment.

Table 2. Cooking conditions for *E. grandis* bark and root

Pulping conditions for bark (Kraft-$NaBH_4$)	**Unit**	**Value**
Active alkali	%	20
Sulfidity	%	25
$NaBH_4$ charge	%	0, 0.3, 0.5, 0.7
Cooking temperature	°C	160
Time to maximum temperature	min.	40
Time at maximum temperature	min.	90
Liquor to raw material ratio	L/kg	5/1
Pulping condition for root (Soda-AQ)	**Unit**	**Value**
NaOH charge	%	23
AQ charge	%	0, 0.3, 0.5, 0.7
Cooking temperature	°C	150
Time to maximum temperature	min.	40
Time at maximum temperature	min.	100
Liquor to raw material ratio	L/kg	5/1

The cooking processes were carried out in an electrically heated, high-temperature and pressure-resistant rotary cooker. At the end of cooking, the pulp discharged from the digester was washed in a 200-mesh screen with plenty of water until the black solution was removed. After separation into fibers in a disintegrator, non-fibrous structures were removed by a vibrating screen with a 0.15 mm slot. The screened pulps were brought to 20-25% dryness and stored in polyethylene bags at +4 °C.

The pulps were beaten by means of a laboratory-type Hollander to a freeness level of 35 ± 2 SR° (Schopper Riegler), then the freeness level and drainability were measured using the ISO 5267-1 standard.

Test sheets with grammages (basis weight) of 80 ± 2 g/m^2 were produced using a Rapid Köthen semi-automatic paper machine (RK-21) in accordance with the ISO 5269-2 standard. The sheets were then conditioned in a chamber at 23 ± 1 °C and 50% relative humidity for 24 hours according to the ISO 187 standard.

Selected mechanical and optical parameters of the sheets, including tensile, burst, tear strengths (expressed as an index) and brightness were measured using ISO standards 1924-2 (2008), 2758 (2014), 1974 (2012) and ISO 2470-1 (2016), respectively. All the measurements were repeated at least five times and mean values were used.

A one-way analysis of variance (SPSS for Windows) was used to ascertain if there was a statistically significant difference between the means of the independent groups. The Duncan test was then employed to assess the effect of AQ and $NaBH_4$ on the paper characteristics. Pearson's correlation coefficient tests (at the 0.05 significance level, Minitab for Windows) were performed to determine if a linear relationship existed.

Results and discussion

Many substances are dissolved during cooking processes, such as carbohydrates (cellulose and hemicelluloses), lignin and extractives [Dong et al. 2015]. Therefore, more than half of the raw material used (bark and root) was dissolved in the black liquor (the solution obtained after cooking). However, the addition of sodium borohydride ($NaBH_4$) and anthraquinone (AQ) to the digester prevented carbohydrate loss caused by peeling reactions and increased the pulp yield, as can be seen from Table 3.

Table 3. Some chemical and yield properties of pulps made from *E. grandis* bark and root

Pulp yield and chemical properties	Bark				Root			
	$NaBH_4$ charge (%)				AQ charge (%)			
	0.0	0.3	0.5	0.7	0.0	0.3	0.5	0.7
Screened pulp yield (%)	36.8^c	37.7^b	38.3^b	40.5^a	39.1^c	40.9^b	40.5^b	41.8^a
Screen rejects (%)	0.45^b	0.00^a	0.00^a	0.00^a	0.65^a	0.90^{ab}	1.48^c	1.24^b
Total pulp yield (%)	37.3^c	37.7^b	38.3^b	40.5^a	39.8^c	41.8^b	41.9^b	43.0^a
Kappa number	50^a	50^a	48^a	48^a	90^c	78^b	77^b	67^a
Viscosity (cm^3/g)	650^c	659^b	685^a	690^a	897^c	988^b	998^b	1029^a
DP	933^c	948^b	990^a	997^a	1332^c	1483^b	1499^b	1551^a

*DP refers to degree of polymerization of cellulose. Means with same lowercase letters are not significantly different at 95% confidence level according to Duncan's multiple range test.

Pulp yield is an important parameter for manufacturers and it is known that the higher it is, the more economical and beneficial it is. In general, in pulp production, it is undesirable for the pulp yield to fall below 35-40% in terms of the cost and production capacity [Çiçekler 2019]. In this study, the total pulp yield of BP and RP was over 35% and the highest yield was obtained from the root (39.1%) by the catalyst-free cooking experiment. It is believed that the reason why the yields of pulp obtained from the bark are lower than those obtained from the root is due to the fact that the extractive substance and ash content of the bark is higher than that of the root (Table 1). According to the table, it was found that there is an increase in pulp yield by using $NaBH_4$ and AQ as catalysts in bark and root cooking. Since $NaBH_4$ has a protective effect on cellulose and hemicelluloses in the cooking environment, it increases pulp yield. Studies have shown that pulp yield improvement can be achieved by using $NaBH_4$ as a catalyst [Deniz et al. 2017, Istek and Gonteki 2009, Saraçbasi et al. 2016, Tutus and Cicekler 2016]. The total pulp yield increased by 10.1% when $NaBH_4$ (0.7%) was added to the cooking medium during bark cooking. The researchers worked tirelessly to evaluate the possible industrial applications for AQ. It was concluded that AQ could be utilized to augment the pulp yield [Bhardwaj et al. 2005, Jahan and Mun 2004, Melesse et al. 2022, Nagpal et al. 2021, Utami et al. 2021]. In this study, the addition of 0.7% AQ to the cooking liquor raised the pulp yield by approximately 2.7 units.

While the kappa number is used to estimate the amount of lignin in the pulp, it also indicates the degree of pulp bleachability [Costa and Colodette 2007, Tutus and Cicekler 2016]. The kappa number of all BP obtained from different cooking methods exhibits a lower value compared to that of RP. As a result of the cooking experiments using no catalyst, the kappa numbers of BP and RP were found to be 50 and 98, respectively. As can be seen in Table 1, the fact that the lignin content of the root is higher than that of the bark affects the amount of lignin that is dissolved during cooking, and the lignin content of RP is higher than that of BP, and this also explains why BP is easier to bleach compared to RP. AQ used as a cooking additive had a significant effect ($p<0.000$, Duncan) on the kappa numbers of RP, reducing these values from 90 to 67 with a 0.7% AQ addition to the cooking liquor. Nevertheless, the addition of $NaBH_4$ to the cooking liquor had no effect ($p<0.349$, Duncan) on the kappa number of BP. Holton reported the application of AQ as an efficient pulping catalyst for the first time in the 1970s [Holton and Chapman 1977, Saka et al. 1982]. Under otherwise equal cooking circumstances, Holton claimed that AQ may be utilized to lower the kappa number. In addition, it has been also reported that AQ not only quickened the kraft pulping process, but it also controlled or maintained pulp yield [Ghazy et al. 2014, Haddad et al. 2009, Hart and Rudie 2014]. The pulp viscosity indicates the average degree of polymerization of the cellulose. As a result, such a test provides a relative indicator of the degradation (reduction in the molecular weight of the cellulose) caused by the pulping and/or bleaching processes [Çiçekler et al. 2021, Tappi 2013b]. When the viscosity values of BP and RP from the cooking experiments without additives were compared, the viscosity (879 cm^3/g) and degree of polymerization (DP) (1132) of RP were higher than those of BP (650 cm^3/g) and DP (933). The molecular weight of cellulose varies according to plants and plant parts (Broxterman & Schols 2018, Evans & Wallis 1989), and the molecular weight and chain length of the cellulose of the bark is lower than that of the root (Table 3). $NaBH_4$ added to the cooking liquor as a catalyst had a positive effect on the viscosity ($p<0.921$, Pearson) and DP values ($p<0.921$, Pearson) of BP as it prevented the peeling reactions occurring on carbohydrates during the cooking process [İstek and Özkan 2008, Tutus and Cicekler 2016]. The addition of 0.7% $NaBH_4$ to white liquor (the solution used in cooking) increased the viscosity and DP by 6.2% and 6.9%, respectively. $NaBH_4$ affects the viscosity and DP because it can stop peeling processes in cellulose chains. The AQ addition to white liquor during root cooking also increased the viscosity ($p<0.316$, Pearson) and DP values ($p<0.315$, Pearson) of the RP. With the 0.7% AQ addition to the digester, the viscosity and DP values of RP increased by 14.7% and 16.4%, respectively. A possible explanation for these increases is that AQ oxidizes the reducing end of polysaccharides in the pulp and protects them from alkaline degradation [Akgül et al. 2007; Akgül and Tozluoglu 2009; Dimmel 1985; Tutus et al. 2015].

Data resulting from the mechanical and optical testing of the papers manufactured from the bark (BP) and root (RP) pulps are illustrated in Fig. 1.

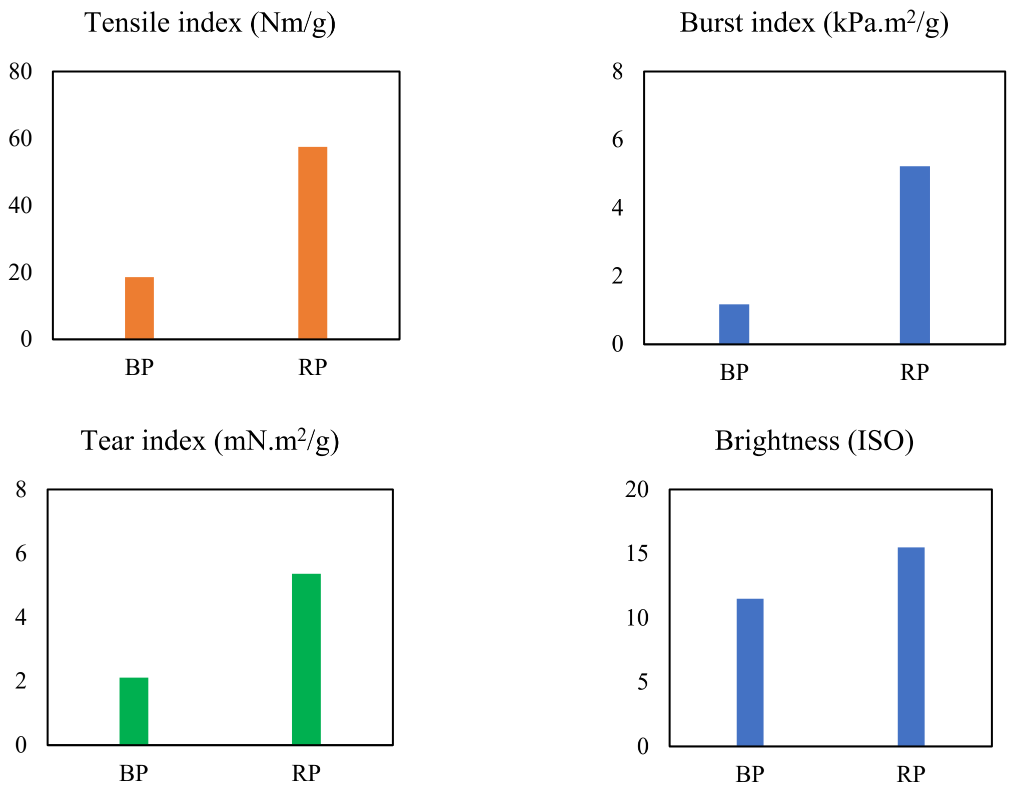

Fig. 1. Mechanical and optical properties of pulps made from *E. grandis* bark (BP) and root (RP) by EDS method by EDS method

Tensile strength is the maximum tensile force generated in a specimen before rupture during a tensile test conducted under specified conditions. The tensile index is the tensile strength in N/m divided by the grammage [Tappi 2013a]. Tensile strength depends on the fiber length and the fiber-to-fiber bonding ratio in the fibers, and as this ratio increases, the tensile strength of paper also grows. The tensile strength or index is often used to represent the impact of applications on paper strength, which is derived from factors such as fiber strength and bonding [Zhai and Zhou 2014]. In Fig. 1, the tensile indices of BP and RP obtained from the catalyst-free cooking trials were found to be 18.55 and 57.46 Nm/g, respectively. This demonstrates that the fiber strength and bondability of RP fiber is better than that of BP.

One of the factors affecting burst strength is fiber length and the other is internal bonding [Clark 1978]. As with the burst index, the burst strength of RP (5.22 $kPa.m^2/g$) was found to be higher than that of BP (1.17 $kPa.m^2/g$) because RP fibers are longer than BP fibers (Table 1) and have a higher internal bonding potential. A greater fiber length has been reported to increase tenacity, particularly at reduced bond levels [Seth 1990]. Furthermore, coarser fibers produced papers with a higher tear index than finer fibers [Seth and Page 1988]. Consistent with this information, the tear strength of RP (5.36 $mN.m^2/g$) was higher because the fiber length and coarseness were higher than BP (2.11 $mN.m^2/g$).

Pulp brightness is defined as the diffuse reflection of blue light from pulp sheets at a notional wavelength of 457 nm and one of the most critical quality indicators in term of print quality [Hubbe et al. 2008, Li et al. 2014]. The brightness values of BP and RP were determined to be 11.5 ISO% and 15.5 ISO%, respectively. The reason BP brightness is lower than RP is because it is made using the kraft process. The dark color of kraft pulp has been linked to double bonds conjugated to aromatic rings as well as quinones and quinone methides [Hubbe et al. 2008]. In addition, it has been experimentally proven that the brightness is reduced in wood chips with a larger proportion of bark [Tripathi et al. 2020].

Figs. 2 and 3 illustrate the effects of $NaBH_4$ and AQ on the mechanical properties of papers manufactured from BP and RP. It has been reported that $NaBH_4$ and AQ added to the cooking liquor improves the mechanical properties of papers, with the exception of tear strength [Birinci et al. 2020, Gülsoy and Simsir 2018, Hassan et al. 2020, Melesse et al. 2022, Sjöberg et al. 2004, Tutus and Cicekler 2016]. It has been stated in many studies that boron compounds and AQ prevent the peeling reaction during cooking and are less damaging to carbohydrates, and therefore the mechanical properties of the produced papers are improved [Akgül et al. 2007, Copur and Tozluoglu 2008, Erisir et al. 2015, Hart and Rudie 2014, Hedjazi et al. 2009, Istek and Gonteki 2009]. The pulps obtained from the cooking experiment with the addition of 0.7% $NaBH_4$ and 0.7% AQ exhibited the highest tensile and burst index values. With the addition of 0.7% $NaBH_4$ to the white liquor, the tensile index increased from 18.6 Nm/g to 20.9 Nm/g, while the burst index rose from 1.17 $kPa.m^2/g$ to 1.50 $kPa.m^2/g$. With the addition of AQ, the tensile and burst indices grew by 20.1% and 15.4%, respectively. As previously mentioned, the mechanical properties of the pulps obtained from the cooking experiments using $NaBH_4$ and AQ are higher since The utilization of $NaBH_4$ and AQ helps prevent the degradation of carbohydrates, such as cellulose and hemicellulose, which significantly influences the mechanical properties of paper [Akgül and Tozluoglu 2009, Erisir et al. 2015, Hedjazi et al. 2009, Istek and Gonteki 2009, Molin and Teder 2002]. In terms of tear strength, the use of $NaBH_4$ and AQ as catalysts in the cooking process proved to be less effective compared to other mechanical properties (Figs. 2 and 3).

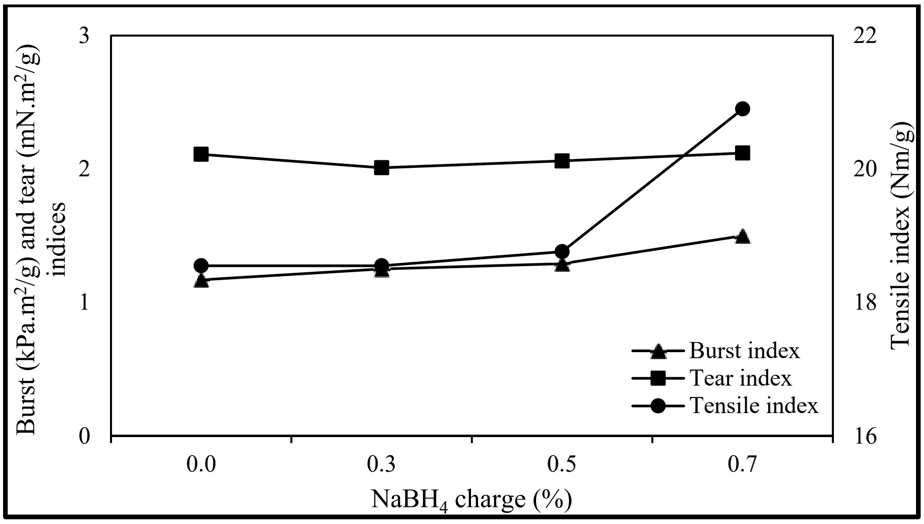

Fig. 2. Effects of $NaBH_4$ on mechanical properties of BP

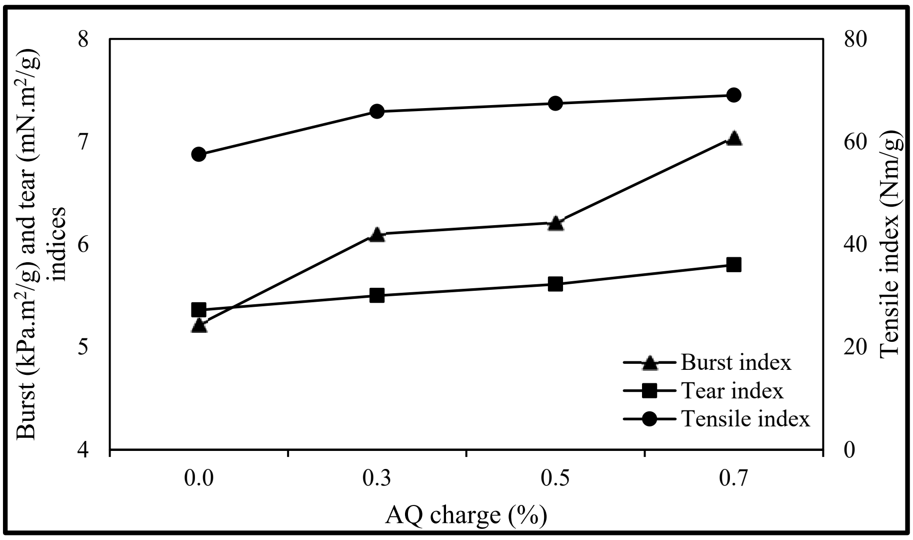

Fig. 3. Effects of AQ on mechanical properties of RP

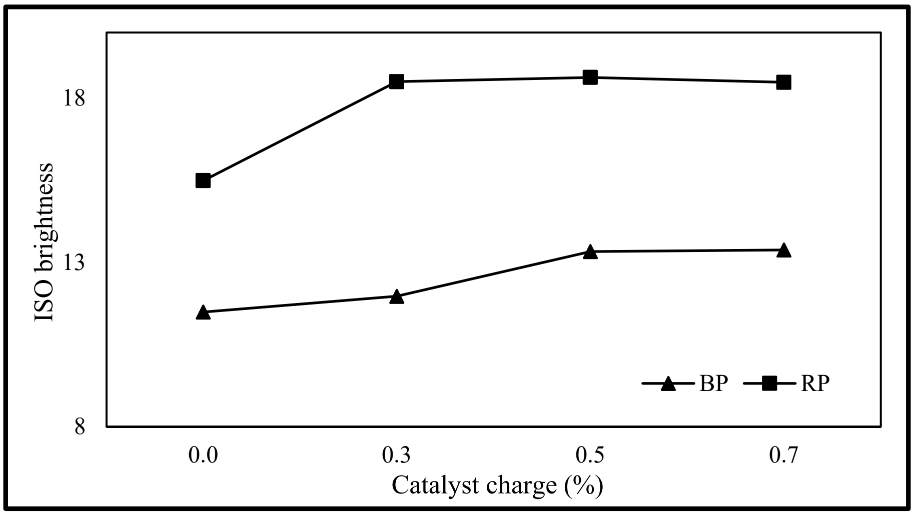

Fig. 4. Effects of $NaBH_4$ and AQ on brightness of BP and RP

Fig. 4 presents the effects of the catalysts ($NaBH_4$ and AQ) on the BP and RP brightness values. As can be seen in the figure, the brightness values of BP and RP obtained using $NaBH_4$ and AQ increased. As with the mechanical properties, the highest brightness values were obtained using 0.7% $NaBH_4$ (13.4 ISO%) and 0.7% AQ (18.5 ISO%). $NaBH_4$ has a bleaching effect [Agarwal and Atalla 1994, Tutus and Cicekler 2016], while AQ accelerates the dissolution of lignin [Masrol et al. 2014, Moradbak et al. 2016].

Conclusions

This study aimed to evaluate the suitability of the bark and root of *E. grandis* for the pulp and paper industry, to characterize them in terms of their chemical, mechanical and optical properties, as well as to determine the effects of $NaBH_4$ and AQ on the pulp properties. Below are the main conclusions that can be drawn:

The yields of the pulp obtained from the *E. grandis* bark (37.3%) and root (39.8%) were at the desired values for the pulp and paper industry. The kappa number of BP and RP was found to be 50 and 90, respectively. The respective viscosity values were determined to be 650 cm^3/g and 988 cm^3/g. By using $NaBH_4$ as a catalyst in the cooking experiments, the pulp yield and viscosity values increased by 8.6% and 6.2%, respectively, while the kappa number decreased by 4%. AQ added to white liquor also exhibited the same effects in root pulping. When comparing the pulp obtained from bark (BP) and root (RP), the yield and viscosity values of RP are better than BP, while the kappa values of BP are lower than those of RP. The optical and mechanical properties of RP occurred to be better than those of RP. The fact that the viscosity values of root cellulose are higher than that of bark cellulose directly affects the mechanical properties, and the higher the viscosity value, the better the mechanical properties. Since the bark contains more extractives and lignin than the root, the color of the produced pulp is darker compared to the root and the brightness values are therefore lower. The grades of the bark and root handsheets are acceptable for the manufacture of corrugated papers (fluting, testliner) with values similar to those in the recycled paper market, giving advantages in using virgin fibers over recycled fibers. The bark and root of *E. grandis* have a potential for use in the backing paper industry. Besides, unbleached and bleached short fibers obtained from BP and RP can be added to long fibers in certain proportions and used in all kinds of paper production.

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We express our deep sadness at the loss of Ayse Özdemir in the Kahramanmaras mega-earthquakes on February 6th, 2023. Ayse was a highly respected and valued member of our team, and we will greatly miss the contributions she made to our work. She showed a strong passion for research and was dedicated to advancing our understanding of the field. Ayse's warm personality and positive energy made her a pleasure to work with. Our thoughts and prayers are with Ayse's family and loved ones during this difficult time. We also extend our sympathies to all those affected by the earthquakes. We express our gratitude for the time we had the privilege of working with Ayse and will forever hold her memory dear.

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