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Utilization of the pomegranate tree (*Punica granatum* L.) in the paper industry

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Abstract: In this study, the chemical composition, fiber morphology, pulp, and paper properties of the pomegranate tree (*Punica granatum* L.) were determined. Stem and branch wood were analyzed separately. Kraft and kraft- anthraquinone (AQ) methods were used for the pulping processes. Stem wood showed superiority to branch wood with longer, wider, thicker-walled fibers. The holocellulose, α-cellulose, and lignin contents of branch and stem wood were 72.98%–73.50%, 38.37%–39.92%, and 21.04%–25.29%, respectively. In addition, the yield of kraft pulp made from stem wood was higher than that of branch wood. However, branch wood handsheets had higher strength properties and brightness than those of stem wood handsheets.

Key words: Anthraquinone, branch wood, kraft pulp, *Punica granatum* L., paper properties, stem wood

1. Introduction

Pomegranate (*Punica granatum* L.), a small tree with a height of 2–5 m, is native to Central Asia and has been cultivated for centuries in the Middle East, Asia, the Mediterranean, the United States, and South and Central America (Currò et al., 2010). India, Iran, China, and Turkey are the major producers of pomegranate fruit (Kurt and Şahin, 2013). In 2013, pomegranate fruit production in Turkey was estimated to be 383,085 t and the number of pomegranate trees 16,176,000 (http://www.tuik.gov.tr/). The red pomegranate fruit is mainly consumed fresh or as juice, wines, and extract (Mertens-Talcott et al., 2006). Interest in the pomegranate has been increasing because of its antioxidant, antitumor, antibacterial, and antidiarrheal properties. Not only the fruit but also the flowers have been reported as having astringent, hemostatic, antidiabetic, antioxidant, and hepatoprotective effects (Bektaş and Öztürk, 2007; Özgen et al., 2008). Each year pomegranate orchard branches are pruned and, after a few years, pomegranate trees are replaced by younger ones. However, these materials are not utilized commercially and are usually burned as an energy source. In the paper industry, utilization of orange and olive tree prunings was previously evaluated by Gonzaléz et al. (2011) and Requejo et al. (2012), respectively. This study aimed to analyze the utilization of pruned pomegranate branches in the pulp and paper industry. Global paper consumption is increasing yearly, and, consequently, forest resources are being reduced. In order to overcome the future demand for wood (Samariha et al., 2011), many studies have focused on the utilization of alternative resources in the pulping industry (Atik, 2002; Deniz et al., 2004; Shatalov and Pereira, 2006; Çöpür et al., 2007; Gümüşkaya et al., 2007; Akgül and Tozluoğlu, 2009; Khiari et al., 2010; Şahin, 2012). The present study proposed to answer the following question: Can pomegranate prunings be utilized as an alternative resource in the pulping industry?

In this study, the chemical composition and fiber morphology of stem and branch wood of the pomegranate tree (*Punica granatum* L.) were determined. Furthermore, the kraft and kraft- anthraquinone (AQ) pulp properties of both branch and stem woods were evaluated.

2. Materials and methods

2.1. Plant material and sample preparation

Stem and branch samples of pomegranate (*Punica granatum* L.) were collected from Antalya Province, Turkey. The stem and branch samples averaged 4.2 cm and 1.4 cm in diameter, respectively. All wood samples were debarked and subdivided into 4 disks of 25 mm in height. A chisel was used to manually chip each disk as homogeneously as possible to the size of 25 × 14 × 5 mm for pulping. The wood chips were air-dried and stored in dry conditions.

2.2. Chemical analysis

The standard methods used in the main chemical analyses of both stem and branch woods are given in Table 1. Three repetitions were carried out for all experiments.

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Table 1. The standard methods of chemical analysis using in this study.

Experiment	Standard method
Sample preparation	TAPPI T 257
Holocellulose	Wise and Karl (1962)
α-Cellulose	TAPPI T 203
Klason lignin	TAPPI T 222
Acetone-water solubility	TAPPI T 204
Cold and hot water solubility	TAPPI T 207
1% NaOH solubility	TAPPI T 212

2.3. Fiber analysis

The maceration with chlorite method (Spearin and Isenberg, 1947) was applied and the samples were then agitated to obtain the individual fibers (Berlyn and Miksche, 1976). Before storing them in glycerin, the samples were dehydrated with ethyl alcohol. The fiber length, fiber width, lumen width, and cell wall thickness of 50 randomly selected fibers were measured. In addition, the length of the vessel elements was detected and the slenderness ratio (fiber length/fiber width), Runkel ratio [(2 × cell – wall thickness)/lumen width], and flexibility ratio [(lumen width/fiber width) × 100] were calculated.

2.4. Pulping and handsheet properties

The kraft pulps made from pomegranate stem and branch woods were prepared under the following conditions: 20% active alkali as Na_2O; sulfidity 25%; liquor/wood ratio 4:1; 170 °C cooking temperature; time schedule of 90 min until 170 °C and 75 min at this temperature. The same cooking conditions were applied to the samples with 0.1% AQ added. The air-dried chips were cooked in a laboratory-type 15-L electrically heated rotary digester. In order to remove the black liquor, the pulps were washed and disintegrated before screening with a Somerville-type pulp screener having 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 under the same conditions. The kappa number, screened yield, and freeness levels of all the pomegranate wood 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 (TAPPI T 494), burst index (TAPPI T 403), tear index (TAPPI T 414), brightness (TAPPI T 525), and opacity (TAPPI T 519) of the handsheets were measured using the relevant standard methods.

2.5. Statistical analysis

All data were analyzed using SPSS. The data related to fiber morphology and chemical composition of the stem and branch woods were analyzed statistically using the independent t-test ($P < 0.05$). The data belonging to the kraft and kraft-AQ pulp properties of the stem and branch woods were analyzed with analysis of variance (ANOVA). The dual factors of tree compartments (stem and branch) and AQ (AQ-free and AQ-added) were used. The effects of tree compartment, AQ, and the interactions of both factors on paper properties were evaluated statistically. All pairwise multiple comparison procedures were performed using Duncan's test ($P < 0.05$). The same letter within a column in Tables 2 and 3 denotes that there were no statistically significant differences between the groups in question.

3. Results and discussion

3.1. Chemical composition

The chemical compositions of the stem and branch wood of the pomegranate tree are shown in Table 2. The content of main compounds like holocellulose, Klason lignin, and α-cellulose content was found to be higher in the stem wood than in the branch wood. On the contrary, solubility values including acetone-water, hot water, and cold water were higher in the branch wood.

Goulart et al. (2012) noted that holocellulose, lignin, and extractive content differences between the stem and branch wood of *Stryphnodendron adstringens* were statistically insignificant. However, Jahan et al. (2010)

Table 2. Chemical composition of stem and branch wood of pomegranate.

Experiments	Stem	Branch
Holocellulose (%)	73.50 ± 0.77a	72.98 ± 0.08a
α-Cellulose (%)	39.92 ± 0.72a	38.37 ± 0.17b
Klason lignin (%)	25.29 ± 0.12a	21.04 ± 0.24b
Acetone-water solubility (95:5 w:w)	2.53 ± 0.24a	4.17 ± 0.23b
Hot water solubility (%)	6.90 ± 0.08a	11.29 ± 0.12b
Cold water solubility (%)	4.53 ± 0.10a	10.18 ± 0.14b

Table 3. The fiber morphology of stem and branch wood of pomegranate.

Fiber properties	Stem	Branch
Fiber length (μm)	745.40 ± 74.51a	702.80 ± 72.96b
Vessel element length (mm)	591.48 ± 53.54a	537.20 ± 70.21b
Fiber width (μm)	20.95 ± 1.88a	19.20 ± 2.23b
Lumen width (μm)	11.65 ± 1.29a	11.50 ± 1.67a
Double wall thickness (μm)	9.30 ± 1.24a	7.70 ± 0.98b
Flexibility ratio	55.61	59.90
Slenderness ratio	35.58	36.60
Runkel ratio	1.60	1.34

reported that stem wood of *Trema orientalis* had a higher α-cellulose and lower lignin content. In addition, the hot and cold water, acetone, and 1% NaOH solubility values of *Trema orientalis* branch wood were higher compared to stem wood.

3.2. Fiber morphology

As seen in Table 3, the pomegranate stem wood had longer and wider fibers than the branch wood. Furthermore, it had a thicker wall and the vessel element length was longer. The branch wood fibers were found to have a higher flexibility and slenderness ratio than the stem wood, while the Runkel ratio was lower. These results are compatible with the literature. It was shown that fiber length in the branches of hardwood species is significantly shorter than in the stem fibers (Manwiller, 1974; Taylor, 1977; Bhat and Kärkkäinen, 1981; Bhat et al., 1989). Longer and wider stem wood fibers were reported by Samariha et al. (2011) and Jahan et al. (2010) in *Ailanthus altissima* and *Trema orientalis*, respectively. The high amount of α-cellulose and lignin content in the stem wood can be explained by the high double-wall fiber width (Sjöström, 1993).

3.3. Pulp and paper properties

The kraft and kraft-AQ pulp yields of the pomegranate stem and branch wood are given in the Figure. The total yield of the stem wood kraft pulp was found to be higher than that of the branch wood. This result could be ascribed to the high α-cellulose content of the stem wood compared to the branch wood (Table 2). Law and Lapointe (1983) noted that the branches of *Picea glauca*, *Betula papyrifera*, and *Populus tremuloides* produced 5%–7% less pulp than the stem wood. A high pulp yield was also obtained from the stem wood of *Trema orientalis* (Jahan et al., 2010). The addition of 0.1% AQ increased both the screened yield and total yield of the stem and branch wood pulps. Similar results have been reported by several authors (Jiang, 1995; Çöpür et al., 2007; Akgül and Tozluoğlu, 2009; Biswas et al., 2011). This result could be attributed to carbohydrate retention increases with the addition of AQ (Li et al., 1998; Vaaler and Moe, 2001). The stem wood pulp had a higher kappa number than the branch wood pulp (Figure). This finding could be explained by higher lignin content of the stem wood compared to the branch wood (Table 2). Furthermore, the AQ-added pulps had a lower kappa number than the AQ-free pulps (Figure). This result could be due to the acceleration of delignification rate with the addition of AQ. The reject ratios of stem and branch pulp were 0.41% and 0.07%, respectively. The reject ratio decreased slightly with the addition of AQ.

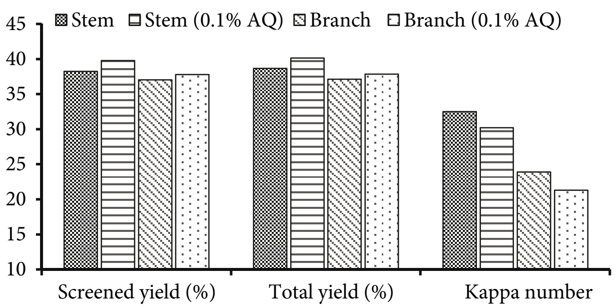

Figure. Some properties of pomegranate branch and stem pulps.

The kraft pulp of the branch wood had higher strength properties compared to the stem wood (Table 4). This result could be attributed to the higher fiber flexibility (Casey, 1961; Scott et al., 1995), higher bonding ability, higher slenderness ratio (Shakhes et al., 2011), and lower Runkel ratio (Nkaa et al., 2007) of the branch wood fibers (Table 3). Hakkila (1971) noted that branch pulp had more superior strength properties than stem pulp. However, Law and Lapointe (1983) and Jahan et al. (2010) reported weaker mechanical properties for the branch pulps of some softwood and hardwood species. On the other hand, compared to some hardwood species, such as eucalypts (Mardones et al., 2006) and aspen (Gülsoy and Tüfek, 2013), the strength properties of the pomegranate handsheets were lower, while they were similar to those

Table 4. The paper properties of kraft and kraft-AQ pulps of pomegranate stem and branch.

Paper properties	Stem	Stem - 0.1% AQ	Branch	Branch - 0.1% AQ	P-value
Tensile index (Nm/g)	38.85 ± 1.78a	40.66 ± 1.62b	42.88 ± 1.43c	41.74 ± 2.34bc	0.000
Stretch (%)	1.93 ± 0.18a	1.92 ± 0.20a	1.81 ± 0.18a	1.82 ± 0.24a	0.262
TEA (j/m^2)	41.25 ± 2.19a	40.74 ± 2.16a	43.05 ± 2.01b	41.71 ± 2.06ab	0.035
Burst index (kPa m^2/g)	1.76 ± 0.06a	1.84 ± 0.07b	1.80 ± 0.08ab	1.78 ± 0.11ab	0.051
Tear index (mN m^2/g)	1.70 ± 0.06a	1.71 ± 0.07a	1.82 ± 0.09b	1.87 ± 0.05b	0.000
Brightness (% ISO)	11.99 ± 0.06a	13.01 ± 0.06b	16.94 ± 0.15c	18.00 ± 0.06d	0.000
Opacity (%)	99.96 ± 0.06a	99.86 ± 0.21a	99.95 ± 0.09a	99.91 ± 0.11a	0.314

of olive (Lopez et al., 2000) and orange trees (González et al., 2013). Furthermore, the kraft pulp of the branch wood had a higher brightness than that of the stem wood. This finding could be explained by the lower kappa number of the branch pulp (Figure). Higher kraft pulp brightness in branch wood of *Trema orientalis* compared to stem wood was reported by Jahan et al. (2010). Except for the tensile and burst indices, no significant difference was observed in the strength properties of the AQ-added and AQ-free pulps of both stem and branch woods. AQ addition significantly increased the brightness, while opacity was not statistically affected by AQ (Table 4). Adding small amounts of AQ into cooking liquor has led to increases in lignin removal by promoting cleavage of β-O-4' linkages in the lignin that are not cleaved in the absence of AQ (Suckling, 1989; Venica et al., 1989). AQ is firstly reduced to anthrahydroquinone, and then it reacts with quinine methide intermediates of lignin, finally causing a reductive splitting of β-O-4' linkages. Thus, AQ accelerates the delignification rate (Liu et al., 2013). The increasing brightness could be attributed to the reduced kappa number with AQ addition (Sánchez et al., 2010).

The importance of utilizing waste material is gaining worldwide recognition. In light of this growing concern, the results of this study suggest an answer to the following question: Can pomegranate prunings be utilized as an alternative resource for the pulping industry? There is a statistical difference in chemical structure and fiber morphology between the stem and branch wood of the pomegranate tree. Although the total yield of the stem wood was higher, kraft pulp made from the branches had superior strength properties. The addition of AQ increased the pulp yield and lowered the kappa number in both. However, the fiber morphology and strength properties were not any lower than those of a number of hardwood species presently used in the pulping industry. Consequently, the stem and branch wood of pomegranate can be used as a fiber source for paper grades that do not require high strength.

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