Examining the Pulp Production Compatibility of Earlywood and Latewood in Willow (*Salix excelsa*) Clones in Terms of Fiber Morphology

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Pulp production suitability of earlywood and latewood of *Salix excelsa* 64/12 and 84/28 clones that grow in the experimental sites of the Poplar and Fast Growing Forest Trees Research Institute of Turkey has been discussed as the result of a preliminary study based on their fiber dimensions and how they relate to each other. Fiber slides were prepared according to Franklin's (1954) maceration method for the microscopic measurement of fiber dimensions defined as fiber length, fiber width, lumen width, and cell wall thickness. Felting power, elasticity, rigidity coefficient, Runkel classification, Muhlsteph ratio, and F-factor parameters that demonstrate the fiber dimensions and the relationship between them have been calculated. The effect of these variables on paper strength properties were evaluated, and fiber dimensions and the relationship between them were statistically analyzed. According to the fiber dimensions of *S. excelsa* 64/12 and 84/28 clones and the results of relationships such as Muhlsteph, Runkel, F-factor elasticity, it has been decided that clones can be used in paperboard and corrugated board production as well as in furnish when mixed with long fibers.

Keywords: Salix excelsa S. G. Gmelin; Willow; Fiber morphology; Fiber dimension

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INTRODUCTION

Willow is one of the most common types of trees with species of about 300 across the world and 23 in Turkey. It naturally grows in the temperate zones and some types of it in the cold sections of the Northern Hemisphere (Tunçtaner 1990-2; Küçükgöksel 2010). The most essential species found in natural tree form in Turkey are *S. alba*, *S. fragilis*, *S. excelsa* S. G. Gmelin, and *S. amygdalina* L. (Yaltırık and Efe 1994; Tunçtaner 1990-2).

It has been noted that willow species that are easily grown, and fast-growing species with short management periods can be effective in meeting the country's wood raw material needs. The forest tree species developing in a rapid manner in order to meet the raw material demand of the pulp and paper industry and dealing with the extensive industrial plantation forestry for the increase of the wood production play a vital role (Semen *et al.* 2001). According to research conducted by the Poplar and Fast Growing Forest Trees Research Instutue (İzmit, Turkey), the following are the specifications of two-year-old clones of 53 willow trees grown in experimental sites in terms of their usage as industrial raw materials in the production of cellulose, energy, chemical substances, and nutrient goods; basic density values were found to be between 0.299 and 0.409 g·cm^{-3} with a mean of 0.344 g.cm^{-3}, production of dry goods that varies between 80 and 23.06 tons in a hectar of land, holocellulose amounts in a hectar of land vary in the range 0.92 to 16.90

tons and 67.11 to 79.40%, low-high calorie averages of their clones demonstrated the values of 4199 to 4266 cal.g^{-1}, ash amount was reported to be between 0.405 and 0.974% with a mean of 0.691% (Tunçtaner 1990-2). Additionally, they have been reported as an essential source of production for animal feed with average protein amounts of 10.85% (Tunçtaner 1990-2).

Basic density is the most important factor in determining the usability of wood in fiber production for fiberboard and paper industries (Bozkurt 1979; Kurtoğlu 1984; Tunçtaner 1990-2). The basic densities of willow clones have been reported as 0.276 g·cm^{-3} to 0.409 g·cm^{-3} in Yugoslavia and 0.360 g·cm^{-3} in Italy (DeBell *et al.* 2002; Kuzovkina and Quigley 2005; Oktaee *et al*. 2017). Approximately 4 million cubic meters of willow and poplar wood are produced in Turkey (Akgül and Tunçtaner 2011). Therefore, the willow clones selected for wood production are in short rotation plantings in Turkey (Cobas *et al.* 2013).

According to Turkish standards TS EN 1015-6 (2000) willow wood which is in the hardwood group of trees is classified as pulp wood (Kıcı 2003).

There are differences between earlywood and latewood sections in terms of fiber properties (Kırcı 2000). Morphological fiber dimensions are length, width, cell wall width, and lumen radius. Fiber dimension and relationships are assessed in terms of the felting rate, elasticity coefficient, Runkel ratio, Muhlsteph Index, rigidity coefficient, and flexibility (F-factor). Fiber dimensions and the relationships between them act as indicators providing essential preliminary information about suitability for pulp production and usability as wood raw material (Dinwoodie 1965; Bostancı 1987; İstek 2008).

In the study, suitability for pulp production has been interpreted through an analysis of results obtained from the preliminary evaluation study of fiber morphology that consists of the earlywood and latewood fiber dimensions and the fiber dimension relationships of *Salix excelsa* 64/12 and 84/28 clones that grow in the experimental sites of the Poplar and Fast-Growing Forest Trees Research Institute of Turkey.

EXPERIMENTAL

Material

Clones 64/12 and 84/28 came from plantation areas (Izmit and Çarşamba-Ordu, of Turkey) of The Poplar and Fast Growing Forest Trees Research Institute. The specimens of 3-year-old 84/28 and 64/12 clones had average diameters of 5.8 cm and 5.2 cm, respectively, and were 60 cm in length. The average diameters of the 84/28 and 64/12 willow clone discs are shown with bark and without bark in Fig. 1.

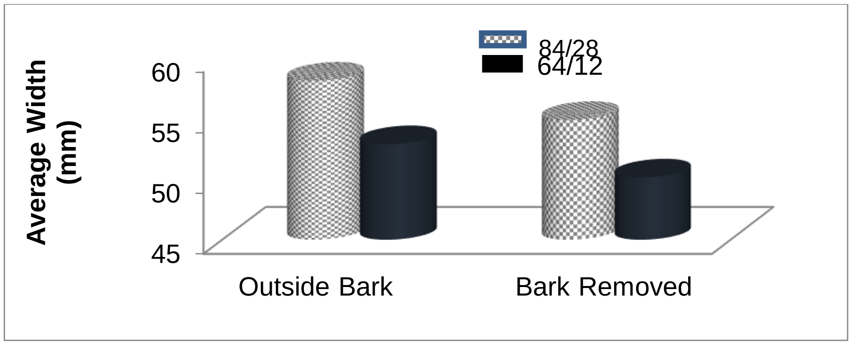

Fig. 1. The average diameters of the 84/28 and 64/12 willow clones

Properties of 64/12 and 84/28 willow clones are illustrated in Table 1.

Table 1. The Properties of 64/12 and 84/28 Willow Clones (Tunçtaner 1990-2)

Clone	Dbh (cm)	Heig ht (m)	Tree vol. (m^3)	Volume (m^3/ha)	Increament (m^3/ha)	Density g.cm^{-3}	Cal.val. cal/g
84/28	4.30	6.28	0.00595	66.11	33.06	0.330	4277
64/12	3.95	5.50	0.00400	44.44	22.22	0.353	4359

Dbh: Diameter at breast height; Tree vol:Tree volume

Method

Samples of 3-year-old *S. excelsa* wood were cut into discs with an approximate thickness of 40 mm and each by annual ring earlywood (EW), latewood (LW). The samples were labeled: first earlywood (1EW), first latewood (1LW), second earlywood (2EW), second latewood (2LW), third earlywood (3EW), and third latewood (3LW). The specimens were subject to morphology analysis. The fibering process was performed using the Franklin (1954) method. After being washed thoroughly, fibers are blended for 3 min. in a mixer. The obtained fiber suspension was strained in a Büchner funnel using a strainer with small holes. Then the fibers that remained on the strainer were placed in small-sized tubes to be preserved by adding glycerin on them in order to prepare the microscope slides. While preparing the microscope slides, it is essential to pay attention to the cleanliness of lam and lamellae and to the prevention of air bubbles while the sample is being placed on the lam. The mixture that consists of fiber and glycerin is dripped on the lam while being spread in a homogeneous manner, the lamella is placed above it and stabilized with varnish.

The morphological properties of the prepared fibers were measured using an optical microscope (Leica DM2500, İstanbul, Turkey) in accordance with TAPPI T232 cm-85 (1985) standards. In order to determine the earlywood and latewood fiber properties of each annual ring, 50 units of fiber length and 100 units of fiber width and cell wall of samples representing the same population were measured as fiber dimensions and their average was calculated. The criterion used in determining the suitability of fiber morphological properties for pulp production are defined below.

- Elasticity ratio: (Lumen radius/fiber width) x 100
- Fibering ratio: Fiber length/fiber width
- Runkel classification: Fiber membrane width/lumen radius

- Rigidity coefficient: (Cell wall thickness/fiber radius)x100
- F-factor: (Fiber length/cell wall thickness) x 100
- Muhlstep classification: (Cell wall thickness area/fiber cross-sectional area) x100

Statistical Analysis

In this study, statistical minimum, maximum mean (μ), and standard deviation (σ) values of fiber dimension parameters measured as fiber length, fiber width, and cell wall thickness are calculated and their data mean is expressed as $\mu \pm \sigma$ (n= 50). In most studies, statistical evaluations are mostly done with 30 units of data number (n). This study was conducted with fiber length (n=50), fiber width (n=100), and cell wall thickness (n=100) units of data.

RESULTS AND DISCUSSION

Fiber Morphology of Willow Clones

The values fiber dimentions are indicated in Table 2.

Table 2. The Values Fiber Dimensions

Species	Length (µm)	Width (µm)	Cell wall thickness (µm)	
S. excelsa (84/28)	776.2±98.3 (n=300)	18.9±3.1 (n=600)	3.3±1.1 (n=600)	Current
S. excelsa (64/12)	853.9±126.4 (n=300)	20.8±3.7 (n=600)	3.7±1.1 (n=600)	Current
S. alba	918	20.8	5.0	Eroğlu and Usta 2004
S. caprea	880	19.0	4.5	Eroğlu and Usta 2004
P. tremula	1413.7	24.8	3.4	Atik 1995
P. nigra	1250	27.2	5.0	Alkan *et al.* 2003
F. orientalis	1655	19.5	7.3	Tank 1978
F. orientalis	670	17.9	4.6	Akgül and Tozluoğlu 2009
A. platanoides	260	21.4	3.5	Durmaz and Ateş 2016

Increments of the 84/28 and 64/12 willow clones in were at minimum 0.81mm and 0.82 mm for 1LW, at maximum 11.80 mm and 10.24 mm for 3EW, and 3.88 mm to 4.68 mm on average (Fig. 2).

Fiber tracheids of *S. excelsa* are shown in Fig. 3. When the various properties of *S. excelsa* clones are taken into consideration, the possibility of their use at the pulp industry as a raw material was determined thoroughly.

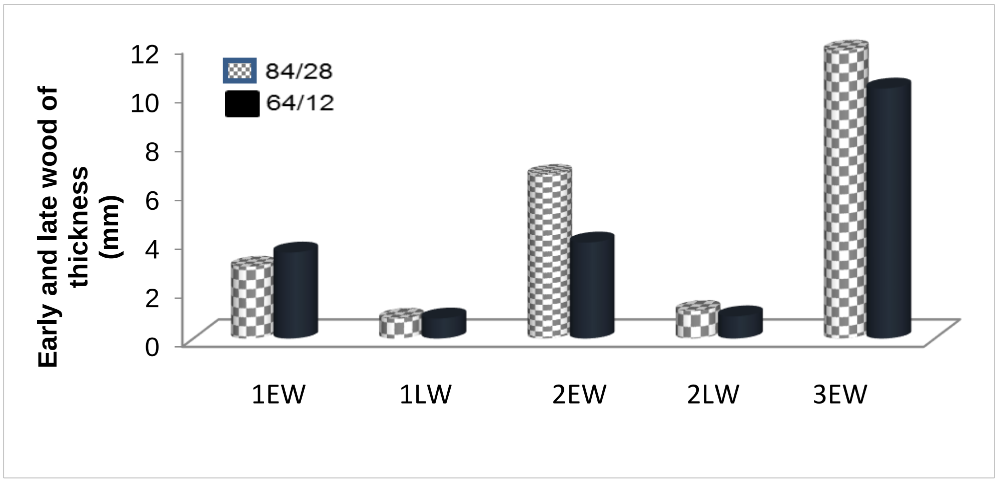

Fig. 2. Increments of 84/28 and 64/12 willow clones of early and late wood in every ring. The average width of 84/28 clone are 9.9% wider than 64/12 clone.

The average fiber length of the 84/28 clone was 776.2±98.29 µm (n=50), and between 920.9±115.5 µm (n=50) and 694.6±103.7 µm (n=50) for 3LW and 1EW. The average fiber length of the 64/12 clone, was 853.9±126.41 µm (n=50), and between 1069.4±181.4 µm (n=50) and 722.8±113.9 µm (n=50), for 3LW and 1EW.

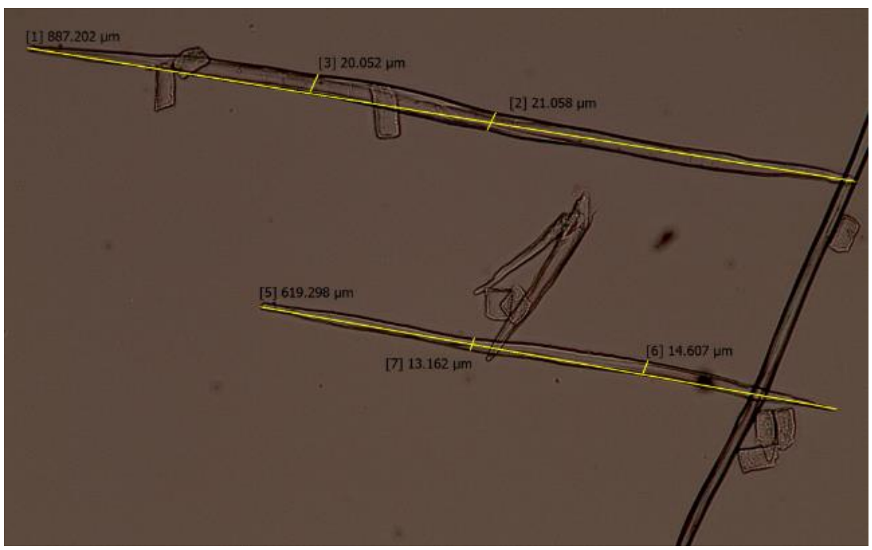

Fig. 3. Fiber traheids 3EW of 64/12 clone

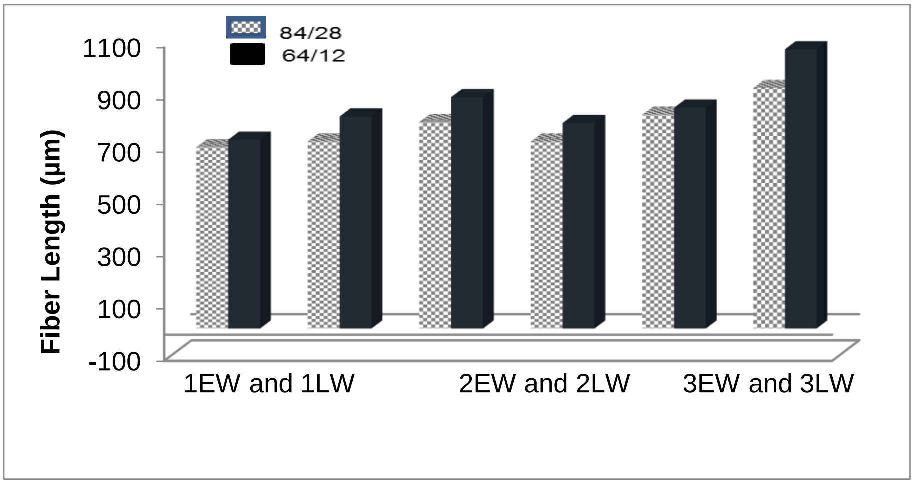

Fig. 4. Distribution of fiber lengths of willow clones

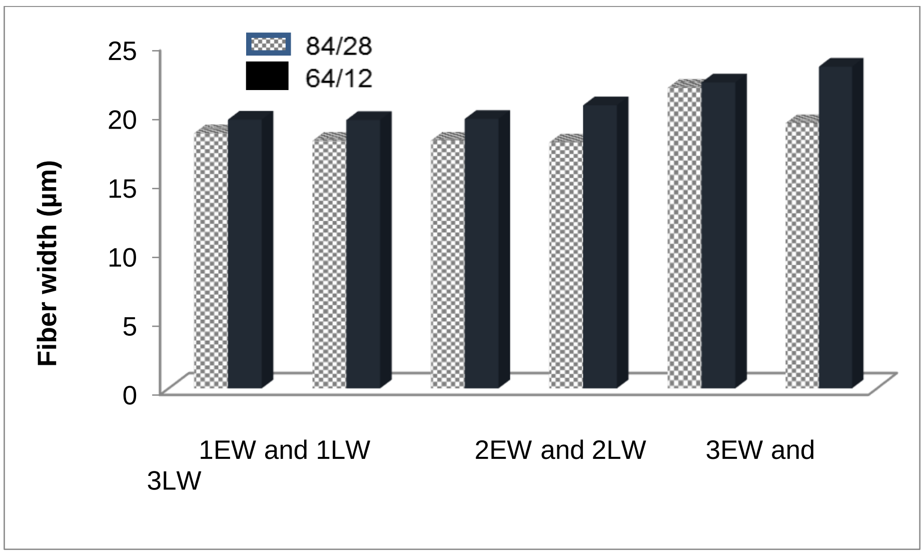

Fig. 5. Distribution of the fiber width of willow clones

The fiber length of both clones was typically in the middle classification of the short fiber sequence of hardwood species. Although the fiber length of *S. exelsa* 64/12 clone was larger than that of 84/28 clone, its fiber distribution demonstrated 22% more deviation while 84/28 clone demonstrated a more homogeneous distribution (Fig. 4). Both clones seemed to fit within their own species and among other broad-leaved trees, except *F. orientalis* and *A. platanoides* (Table 2).

In the 84/28 clone, the fiber width values were between 17.8±3.22 µm (n=100), and 21.8±3.19 µm (n=100), for 2LW and 3EW, respectively, and was 18.9±3.1 µm (n=100) on average. The 64/12 clone was between 19.5±3.68 µm (n=100), and 23.4±5.36 µm (n=100), for 1EW and 3LW, respectively, and was 20.8±3.7 µm (n=100), on average.

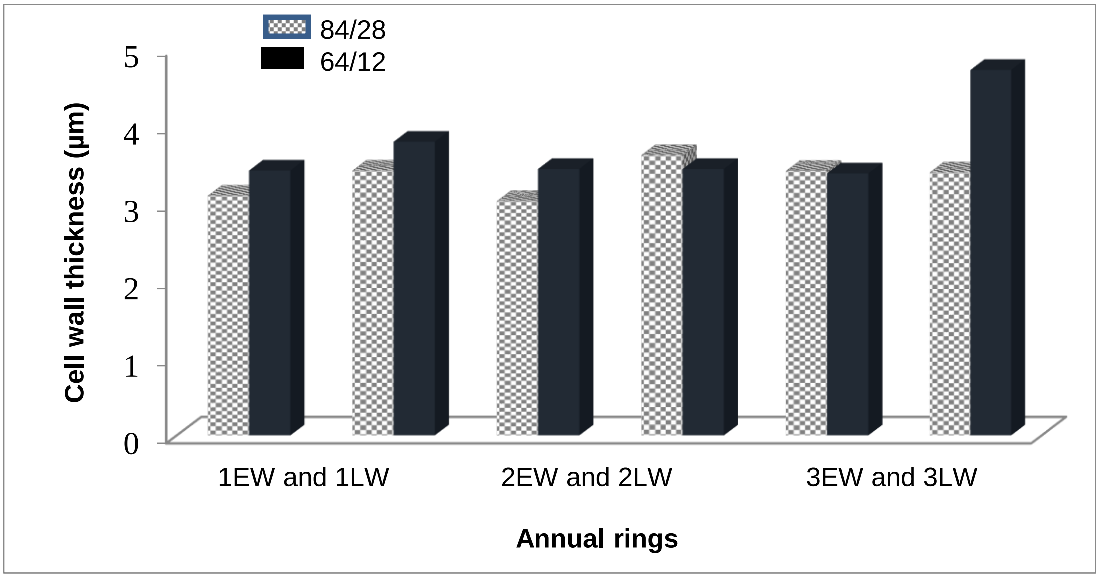

Fig. 6. Distrubuation of the cell wall thickness of willow clones

Cell wall thickness influences the strength of individual fibers. The tearing resistance of paper made from very thin-walled fibers is quite low. Papers produced from extremely thick-walled fibers exhibit low resistance and bulk due to insufficient flattening during paper formation (Kırcı 2003). The cell wall thickness of the 64/12 clone was found to be between 3.4±1.11µm (*n*=100) and 4.7±1.48 µm (*n*=100) for 3EW and 3LW and was 3.7±1.13 µm (*n*=100) on average. The 84/28 clone was between 3.6±0.89 µm (*n*=100) and 3.0±1.22µm (*n*=100) for 2EW and 2LW and was 3.3±1.13 µm (*n*=100) on average (Fig. 6). It can be said that both clones have a linear relationship between age and length and width of fiber.

It has been determined that the 64/12 clone has a wide fiber radius and wide lumen and thick cell wall. The thin wall of the 84/28 clone makes it more favourable for paper production (Table 2; Fig. 6 and 7). The 84/28 clone has a thinner wall in comparison to *P. tremula* and *A. platanoides* among other hardwood species (Table 2).

Pulps are generally produced from hardwood (short fibers) and softwood (long fibers). Short fibers usually range from 0.7 mm to 1.6 mm, with an average fiber length of 1 mm. The long fibers typically range from 2.7 mm to 4.6 mm (Ateş *et al.* 2008).

Fast growing *S. excelsa* revealed itself as close to the values of species such as *S. alba* and *S. caprea*, and may be used in corrugating medium, newsprint (paper), and in mixtures of other long fibers for specific blends (Table 2).

The Relationships between Fiber Dimensions

Relationships between the fiber dimensions provide an important insight into the evaluation of raw material in assessing its suitability for use in paper production. Some relationships between the fiber dimensions impact the forming of the pulp, and as a consequence the properties of the paper are impacted as well. This is because fiber length affects the strength properties of the paper positively, and cell wall thickness has an effect on the paper's strength, especially tearing resistance of the paper (Kırcı 2003). The relationships between the fiber dimension of *Salix excelsa* and age of the wood are shown in the Figs. 7 through 12.

Felting power

Felting power is one of the criteria that indicates the suitability of wood fibers to papermaking. This value affects the durability, breaking length, tearing, bursting, and double-fold (MIT test) strength of paper. The optimum felting power for softwoods is 70. This number is lower for hardwoods. It is known that this felting power has a systemic relationship, especially over the tear strength of paper (Kırcı 2003).

Felting power of the 84/28 and 64/12 clones are at a minimum of in the 2EW sections and include a maximum of 47.9 to 45.8 values respectively in the 3LW section and the clone averages are at the values of 41.1±3.7 and 41.1±3.5 (n=6) (Fig. 7).

The felting coefficient of both clones was slightly higher than hybrid poplar, *P. alba*, *E. camaldulensis*, and *F. orientalis* specimens except other hardwoods (Table 3). According to Ateş *et al*. (2008), strength properties are not only dependent on the felting coefficient, but also on the cell wall thickness. Even though it demonstrates an undesireable situation in terms of felting power, it can be said the thinness of the cell wall makes it suitable for the production of second grade paper.

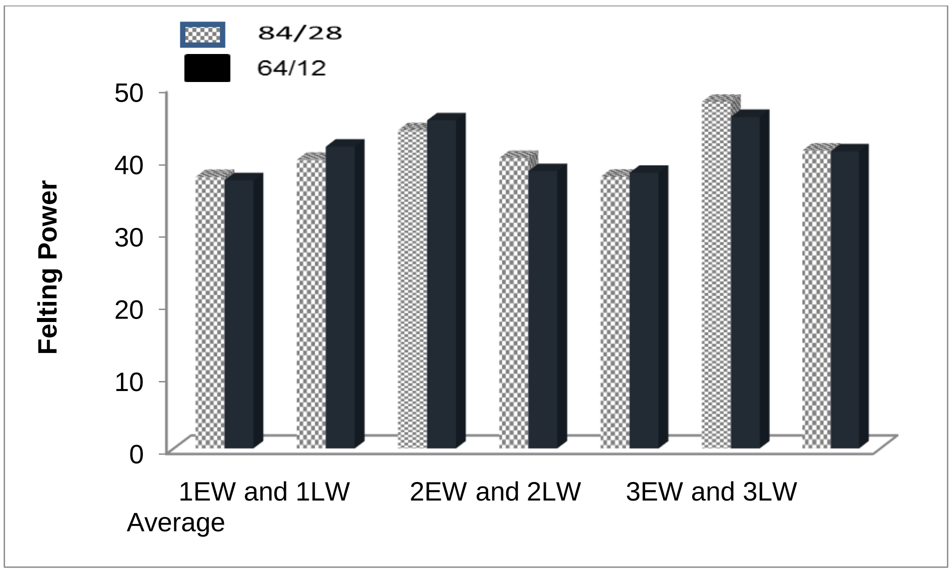

Fig. 7. Felting coefficient of willow clones

Elasticity coefficient

The elasticity coefficient describes the density of the hardwoods, and the classification was evaluated according to Istas *et al.* (1954). The elasticity coefficient of the 84/28 clone was between 59.4 and 68.7 for 2LW and 3EW, with a value of 64.6±3.1 (n=6) on average. The 64/12 clone was between 59.6 and 69.5 for 3LW and 3EW, with a value of 64.4±3.3 (n=6) on average. In terms of elasticity coefficient, it has been determined that the 84/28 clone is a bit more elastic when compared to the 64/12 clone (Fig. 8). According to Istas *et al.* (1954), wood fibers with an elasticity between 50 and 70 are classified as the second group. Being relatively thick walled when compared to hardwood species while having enough elasticity capacity to flatten, it can be said to be capable of producing rather durable paper. Elasticities of both clones are lower than the *P. tremulai* and *A. platanoides* woods and higher than the other hardwoods (Table 3).

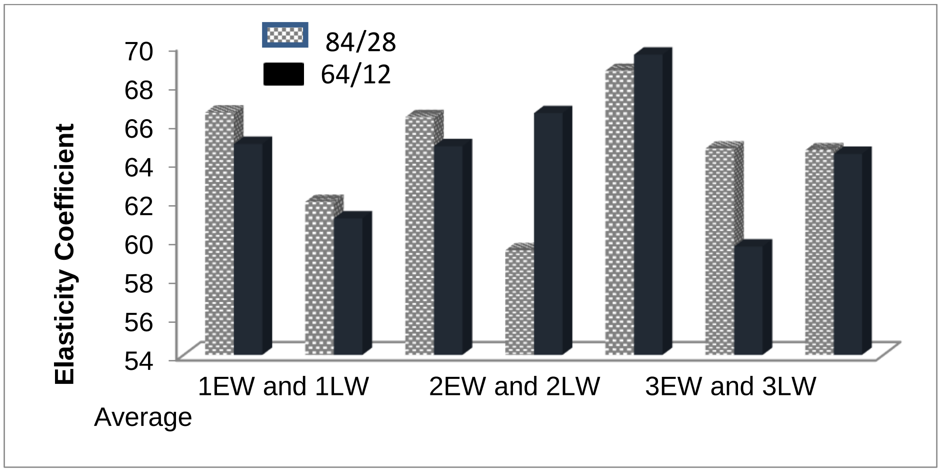

Fig. 8. Elasticity coefficient of willow clones

Runkel ratio

The Runkel ratio is calculated by dividing the double cell wall thickness by the lumen width. The ideal result is smaller than 1. This variable impacts the physical strength properties of paper such as: tearing, bursting, and breaking length. Additionally, the Runkel ratio is used to determine the suitability of the fine cell wall thickness of materials used in papermaking.

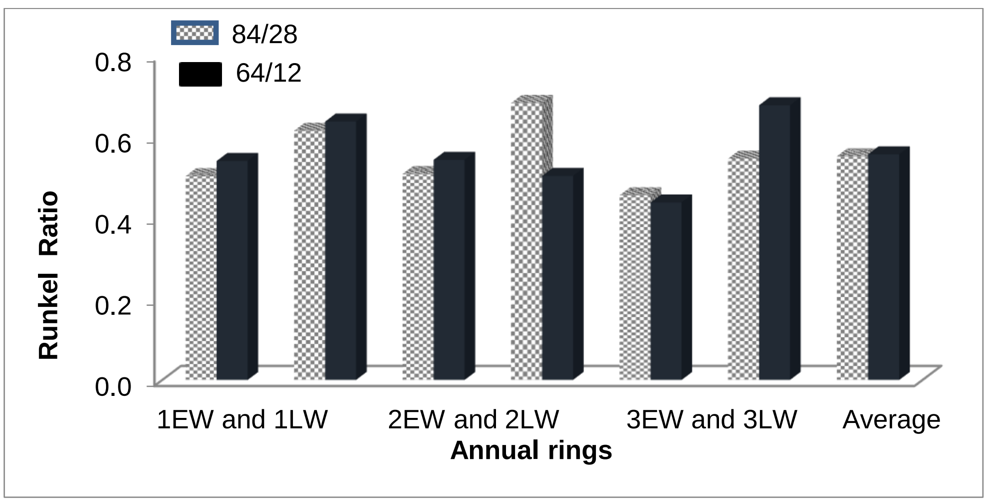

Fig. 9. Runkel ratio of willow clones

The Runkel ratios of 84/28 ve 64/12 clones were determined to be between the minimum values of 0.46 and 0.44 in the 3EW sections. The 84/12 clone in the 2LW sections and the 64/12 clone in the 3LW sections covers the same maximum value of 0.68. The averages of the clones are at the values of 41.1±3.7 ve 41.1±3.5 (n=6), respectively (Fig. 7). The Runkel ratio of clones are in similar values while the 64/12 clone can be said to be a bit more suitable for paper production (Table 3 and Fig. 9).

Rigidity coefficient

The rigidity coefficient depends on the physical and chemical properties of the cell wall material as a measure of fiber conformability. There is an inverse relationship between the rigidity coefficient and paper resistance properties (Bektaş *et al.* 1999). The rigidity coefficient of the 84/28 clone was between 15.7 and 20.3 for 3EW and 2LW, with a coefficient of 17.7±1.5 (n=6) on average. The 64/12 clone was between 15.2 and 20.2 for 3EW and 3LW, with a coefficient of 17.8±1.7 (n=6) on average (Fig. 10). According to Ateş *et al.* (2008), elasticity having a second class (65) shows the inverse relationship with the rigidity for 84/28 (17.7) coefficient. This is supported by the fact that it reflects rigidity properties similar to elasticity resistance properties. It has been clearly understood that the rigidity coefficient of the 84/28 clone favours the elasticity properties in paper production compatibility.

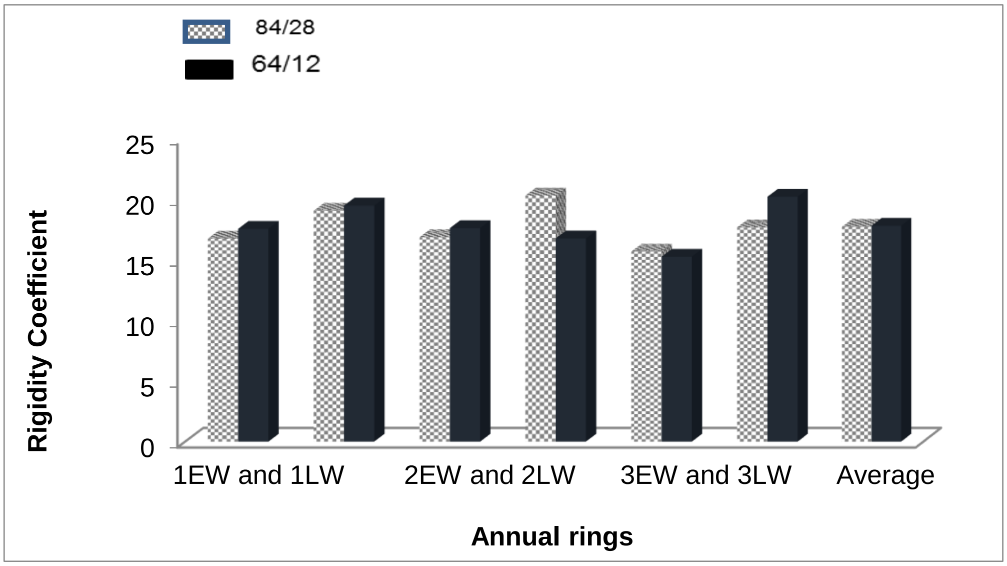

Fig. 10. Rigidity coefficient of willow clones

F-factor (flexibility)

F-factor shows flexibility of fibers. The F-factor of the 84/28 clone was determined to be between 209.5 and 271.0 for 1LW and 3LW, with a factor of 233.9±26.6 (n=6) on average. The 64/12 clone was between 211.3 and 257.5 for 1EW and 2EW, with a factor of 231.4±17.1 (n=6) on average (Fig. 11). Even though both clones demonstrate similar values, the 64/12 clone can be said to have a higher flexibility property since it demonstrates a more homogeneous distribution despite its slightly low F-factor (Akgül and Tozluoğlu 2009). Both clones have a higher F-factor than *P. tremula* L. (207) (Kar 2005) and *F. orientalis* (159.6), but lower than *P. orientalis* (251.8), *C. betulus* (256.1), *C. orientalis* (247.2), and *P. euramericana* I-214 (276.3) (Table 3). The high F-factor values of both clones place them well in the ranking at the upper limit of mid-level among hardwood species.

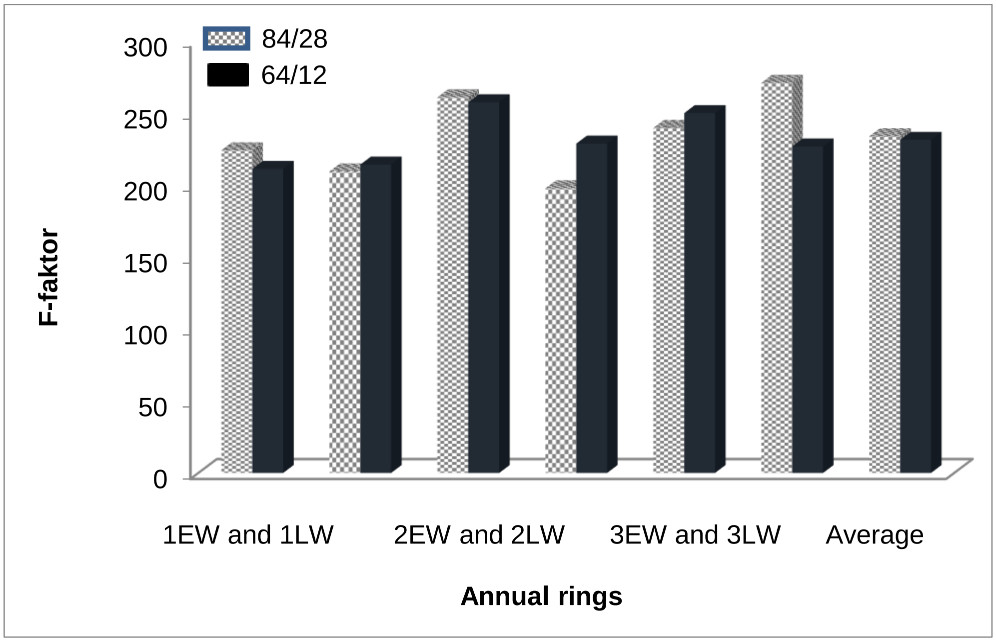

Fig. 11. F-factor of *Salix excelsa* clones

Muhlsteph ratio

Muhlsteph ratio varies depending on cell wall thickness and effects tearing endurance and tensile strength of paper. The Muhlsteph ratio of the 84/28 clone was determined to be between 52.2 and 64.7 for 3EW and 2LW, with a ratio of 58.2±4.0 (n=6) on average. The 64/12 clone was between 51.7 and 64.5 for 3EW and 3LW, with a ratio of 58.4±4.3 (n=6) on average. Even though the Muhlstep ratio of clones are at values very similar to each other, due to its more homogeneous fiber distributiton the 84/28 can be said to have higher resistance properties than the 64/12 clone (Fig. 12).

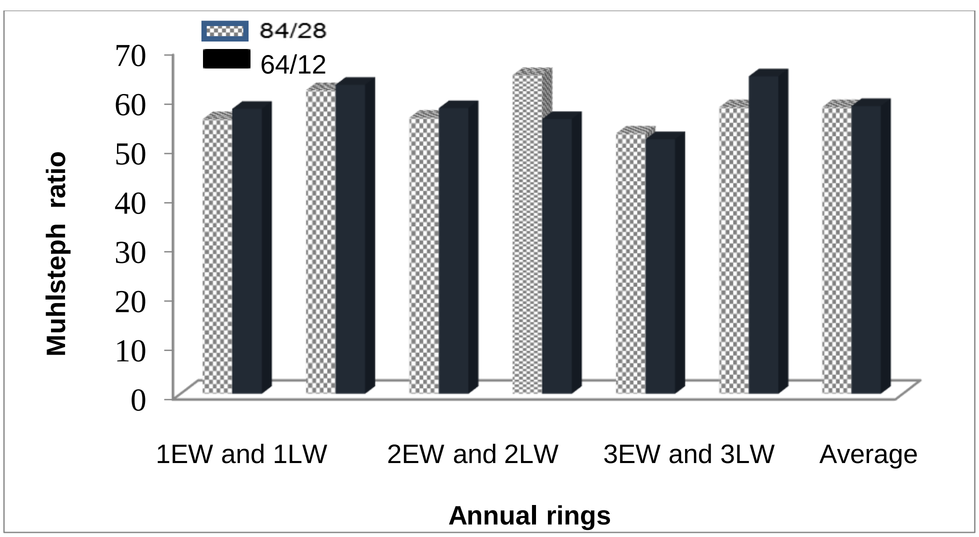

Fig. 12. Muhlsteph ratio of *Salix excelsa* clone

Both clones had higher Muhlstep values than *P. tremula* L. and *A. platanoides* L., but lower values than the others (Table 3). This ratio has indicated that use of clones is more suitable in board and corrugated board production rather than paper. Another aspect for papermaking would be blending of long fiber pulp with *S. excelsa*'s.

Table 3. The Relationships between Fiber Dimensions

Sample	Felt. Pow.	Elas Coff.	Runk. Ratio	Rig. coff.	F factor	Muh. Ratio	
84/28	41.1± 3.7	64.6± 3.1	0.55± 0.1	17.7± 1.5	233.9 ±26.3	58.2± 4.0	Current
64/12	41.1± 3.5	64.4± 3.3	0.56 ±0.1	17.8± 1.7	231.4 ±17.8	58.4 ±4.3	Current
Hybrid poplar	32.0	50.2	1	24.9	128.4	74.8	Madran 1996
E. camaldulensis	57.7	53.8	0.86	23.1	249.1	71.1	Huş *et.al.* 1975
P. tremula	57.0	72.5	0.4	13.7	415.1	47.4	Atik 1995
F. orientalis	59.7	27.6	2.87	37.0	159.6	93.0	Tank 1978
F. orientalis	37.2	48.3	1.1	25.9	140.4	76.7	Akgül and Tozluoğlu 2009
A. platanoides	12.1	71.9	0.8	16.6	141.7	48.4	Durmaz and Ateş 2016

Felt: Pow.; Felting Power; Elas: Elasticity; Runk: Runkel; Rigid: Rigidity; Muh: Muhlsteph; Coff.Coefficient

CONCLUSIONS

1. The more homogeneous fiber length distribution of the *S. exelsa* 84/28 clone indicates that it can produce slightly higher quality paper than 64/12.
2. 64/12 has been determined to have a wide fiber radius and wide lumen and thick cell wall. The 84/28 clone is at a more favourable position for pulp production in comparison to the other clone as well as among hardwood species due to its thin wall. It can be said that both clones have a linear relationship between age and length and width of fiber.
3. Even though it demonstrates an undesireable situation in terms of its felting power, it can be said to be suitable for second grade pulp production due to its thin cell wall.
4. The 84/28 has been determined to be slightly more elastic than the 64/12 clone. Having enough elasticity capability to flatten while being relatively thicker walled than hardwood species, it has been indicated to be capable of producing rather durable paper.
5. Even though the clones are suitable for paper production at similar values, in terms of its Runkel ratio the 64/12 clone has been determined to be suitable for slightly higher quality paper than the other clone.
6. It can be said to have a higher flexibility property due to 64/12 clone's homogeneous distribution. The clones are placed in the mid-level of the ranking among hardwood species due to their high F-factor values.
7. It is clearly understood that the rigidity coefficient of the 84/28 clone is aligned with the elasticity property required in paper production.

8. Muhlstep ratios has been indicated that the use of clones is more suitable for board and corrugated board production rather than paper. Another aspect of papermaking would be blending of long fiber pulp with *S. excelsa*.

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