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PRESERVATIVE TREATMENT OF WILLOW WOOD (*SALIX ALBA*): PRODUCT RETENTION AND SPATIAL DISTRIBUTION.

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ABSTRACT

The aim of this research was to evaluate the preservative treatment of willow wood in order to increase its service life in outdoor commodities (use class 3 by EN 335-1). Treated willow could by used as garden wood (furniture, fencing,…) or as more technical building products such as cladding. An evaluation of the product retention was performed as well as of the penetration profile of the preservatives.

For this study eight trees of white willow (*Salix alba*), age 27, were selected in a homogeneous stand in Bree, Belgium. The sampling contained stem pieces taken at four different height levels. Based on the treatment data, it can be concluded that treatability of willow wood with a CCA preservative following a Lowry process is not just based on one single parameter. Significant intraclonal differences are noticed. Product retention and penetration levels are found to be much lower for heartwood and transition wood in comparison with sapwood. The penetration in radial direction develops homogenously whereas the penetration in tangential direction is highly variable. Although a significant correlation was found between wood density and product retention, density remains a weak parameter in predicting preservative treatability.

KEY WORDS : *Salix alba*, Lowry process, retention, spatial distribution, CCA

INTRODUCTION

The still increasing wood consumption leads to more emphasis on man-made forests using fast growing species. Unfortunately, many of these species have rather low natural durability with regard to wood decay agents and therefore their use requires a protective fungicidal and insecticidal treatment to lengthen their service life. After treatment willow wood can become applicable in garden wood applications (outdoor furniture, fencing) or technical building products as cladding. Consequently, a more profound knowledge must be attained regarding permeability and resulting treatability (Troya et al. 1995). Although, these low density wood species are considered to be permeable, differences in density, fibre collapse during drying, occurrence of tension wood, wood anatomical and pit membrane structure may influence their impregnability with wood preservatives (Baines et al. 1983, Baonza et al. 1992, Bauch et al. 1976, Singh et al. 1999).

Members of the *Salicaceae* family, both poplar and willow, have considerable importance as fast-growing plantation species in a number of countries or regions throughout the world. Some research has been done to characterize the treatability of poplar wood, but few experiments are reported considering willow wood (Murphy et al. 1991, Van Acker et al. 1990, Van Acker and Stevens 1995, Cooper 1976, Marcia Vasquez and Fabian Peredo 2005, Troya et al. 1995). Poplar has been reported as being not easy treatable. However, it is inaccurate to denominate poplar a refractory species. Practical experiences show an irregular impregnability due to an heterogeneous penetration of preservatives. Van Acker et al. (1990) reported significant differences in treatability depending on the poplar clone or hybrid considered. In addition, the presence of a transition zone at the heartwood / sapwood boundary, which shows refractory properties, was observed (Murphy et al. 1992, Van Acker et al. 1990). The flow rate of liquids through the wood structure increases when pit membranes are destroyed or broken. Poplars are well known to be highly susceptible to bacterial wetwood. The bacteria causing wetwood may dramatically increase the permeability of the timber by degrading the pits (Clausen and Kaufert 1952, Knuth 1964). They can also form the basis for a refractory zone corresponding with the transition zone between sapwood and heartwood (Van Acker et al. 1990).

Flanders, the northern region of Belgium, has a large genetic pool of indigenous willow species (De Cock et al. 2003). This allows the selection of specific willow hybrids for the establishment of man-made forests as alternative high yield forest in addition to native mixed broadleaved forests. Willow, based on its indigenous character, may become more important in the future in comparison to the more exotic inter-American poplar clones planted today.

The aim of this research was to evaluate the treatability of willow wood in order to increase its service life in outdoor products and its application in light construction (use class 3). Furthermore, variation in product retention and the penetration profile of the CCA-preservative applied, were evaluated.

MATERIAL AND METHODS

For this investigation eight trees of white willow (*Salix alba*), age 27, were felled within a homogeneous stand in Bree, Belgium. Using Amplified Fragment Length Polymorphism (AFLP), a multi locus DNA fingerprint proofed that all trees were genetically identical. The total tree height ranged from 18 to 21.8 meter and the circumference at breast height showed values between 80 and 122 cm. The very homogeneous habitat had a sandy loam soil and was used earlier as a regular natural irrigated meadow.

The sampling contained stem pieces of 50 cm taken at four different height levels (breast height (1.3 m), 3.6 m, 7.2 m and 10.8 m). They were divided into battens of 50 x 50 x 400 mm according to the grid shown in Fig. 1.

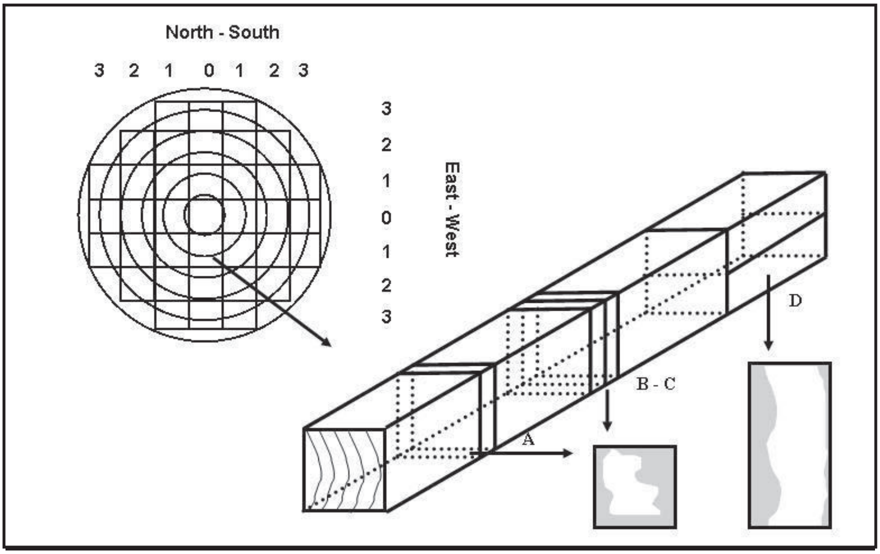

Fig. 1: Partitioning of stem pieces into battens of 400 x 50 x 50 mm at known positions, as well as the further dividing in order to evaluate the uptake of chemicals (A and B) and their spatial distribution (C and D)

All samples used were defect free, excluding impact on retention and penetration. For the same reason they were dried very carefully for several weeks at low temperature (40 ˚C) to an approximate final moisture content of 12-14 %. After drying they were conditioned at 60% relative air humidity and 20 ˚C to ensure an equal moisture distribution throughout the different samples. Every batten received a unique code, so the exact position within the stem volume remained known during the whole experiment (Fig. 1). The readily visible darkened heartwood was marked before dividing the stem pieces. Every sample that was cut out within 5 cm of this marked border, was considered to belong to a transition zone. The other samples were marked as clearly heartwood or sapwood samples.

Tab. 1 gives an overview of the number of samples per tree at every height level.

Tab. 1: Number of battens used for every tree at four different heights

Height	Tree 1	Tree 2	Tree 3	Tree 4	Tree 5	Tree 6	Tree 7	Tree 8
1.3 m	16	25	13	17	19	11	12	8
3.6 m	10	8	6	19	13	9	9	3
7.2 m	8	7	8	6	15	8	8	3
10.8 m	4	8	4	5	3	3	8	3
Sum	38	48	31	47	50	31	37	17

Penetration was only allowed in radial and tangential direction, since the transversal sections were sealed with a paraffin wax. This was done to exclude longitudinal penetration and allows to simulate longer sawn wood. The final step before treatment was the determination of mass and dimensions of the samples.

For the purpose of comparison a treatment with a CCA preservative (Copper, Chromium, Arsenate) was chosen. CCA was preferred based on reference literature for poplar wood and the easy way of visualising the active components based on staining. The composition of the 3% CCA solution is specified in Tab. 2.

Tab. 2: Composition of the CCA used (Copper – Chromium – Arsenate)

Chemical Components	Content of oxides [%]	In 3% solution [%]	Active element proportion [%]	Concentration of active element [kg/100 kg solution]
Copper oxide (CuO)	10.8	0.324	80.0	0.260
Chromium trioxide (CrO_3)	31.0	0.930	52.0	0.484
Arsenic pentoxide (As_2O_5)	17.1	0.513	32.6	0.167
Sum	58.9	1.767		

To avoid excessive product uptake, a Lowry impregnation process was used at low pressure (5 bar). The process started with a pre-vacuum for 10 minutes, followed by the pressure phase that lasted for 40 minutes.

Weighing after treatment allowed assessing the retention levels. The treated battens were allowed to fixate during minimum three weeks under natural drying conditions.

To quantify the penetration, several sub samples were taken. At the centre of each batten two cross sections in the radial - tangential plane (RT plane) were cut out (Fig. 1). Sample B was used to determine the surface area impregnated using a planimeter. Sample C was used to determine the penetration depth by three evenly distributed measurements per side. At 10 cm from the sample end a second sample (A) was taken for the evaluation of the impregnated area. A large sample (100 x 50 mm) was used in the radial - longitudinal plane (RL plane) to describe specific radial penetration (sample D). This penetration was determined at four points evenly distributed per side.

To enhance the contrast between treated and untreated wood a chromium-azurol-S colouring agent was used to form a blue chemical complex with the copper present.

RESULTS AND DISCUSSION

CCA preservative retention

Weighing the battens before and after treatment allows determining several parameters to express retention. The increase in mass relatively to the mass before treatment or the volume of the batten provides a relative uptake of solution (%) or volumetric uptake (kg/m^3) respectively. The amount of absorbed preservative solution can also be expressed in function of the wood surface of the batten that was in contact with the preservative (kg/m^2). Knowing the exact composition of the solution (Tab. 2), the results can also be converted to a product concentration (kg/m^3).

Chandra and Gupta (1972) stated that 16 kg/m^3 of dry salt was necessary for the effective preservation of poplar poles in ground contact and 6.4 kg/m^3 for other outdoor applications without ground contact. Vasquez and Peredo (2005) used a Bethell impregnation process and obtained retention values of 6-7 kg/m^3 for poplar poles. They proved that these levels were sufficient for the use in ground contact. Van Acker (1995) reported preservative uptakes of 10-12 kg/m^3 applying a similar process. Earlier Van Acker (1990) did evaluate the use of a Lowry process and found retentions ranging from 2 to 10 kg/m^3.

All mean values for the obtained product retention parameters are given per tree in Tab. 3a, showing the Duncan multiple range test statistics as well. Dry salt concentrations range from 3.1 to 5.7 kg/m^3. Taking into account the envisaged process impact, average retention is acceptable for use class 3. The mean retentions were significantly different for tree number 4 in respect to the other trees. Intraclonal variations were not reported by Van Acker et al. (1990).

Tab. 3: Parameters expressing retention levels (uptake of CCA-salt solution [kg/m^3],[kg/m^2] and [%] as well as dry salt retention [kg/m^3]) and penetration (mean impregnated area [%] and mean penetration depth [mm] in the RT and RL plane) for the global set of samples for the individual trees as well as the results of the Duncan multiple range post hoc test

3(a) Retention

Parameter	Tree number								Duncan Test
	1	2	3	4	5	6	7	8	
Uptake solution [kg/m³]	154	128	123	190	177	126	137	103	aaabaaaa
Uptake solution [kg/m²]	1.82	1.50	1.44	2.24	2.08	1.48	1.16	1.21	aaabaaaa
Uptake solution [%]	35.7	31.2	28.1	45.3	40.3	28.4	30.7	23.6	aaabaaaa
Uptake dry salt [kg/m³]	4.63	3.83	3.68	5.70	5.31	3.77	4.11	3.09	aaabaaaa

3(b) Penetration

Parameter	Tree number								Duncan Test
	1	2	3	4	5	6	7	8	
Impregnated area RT plane [%]	35.0	30.0	27.9	45.9	42.4	31.0	34.0	21.7	aaabaaaa
Penetration depth RT plane [mm]	7.4	6.1	5.7	9.3	8.4	6.9	7.0	4.6	aaabaaaa
Impregnated area RL plane [%]	39.7	28.6	30.5	50.4	40.2	27.8	32.3	19.6	aaabaaac
Penetration depth RL plane [mm]	7.5	6.1	6.3	9.1	7.5	5.9	7.1	4.2	aaabaaaa

The relatively low pressure applied could be an influencing factor. The values do not correspond with results obtained when treating rubberwood with a similar methodology (Lowry process at 5 bar), leading to retentions expressed in product concentration of 17 kg/m^3 (Akhter 2005). The impregnability of willow is clearly lower.

The mean retention does not significantly change with height in the stem (values not shown, p=0.05). However, the radial position does show significant differences (Tab. 4a) going from pith to bark. Heartwood is the most difficult to treat. The transition wood shows somewhat higher retentions but they are not significantly different from the amounts found in the heartwood. Sapwood shows the highest retention and absorbs almost double the amount the heartwood does.

As retention levels of 6 kg/m^3 did comply with sufficient protection for poplar wood poles in ground contact (Marcia Vasquez and Peredo 2005), this value could be used as a general reference level of sufficient protection. However, the minimum retention level remains dependable on the commodity at hand. Taking 6 kg/m^3 of dry CCA salt as a reference for outdoor applications without ground contact, only the treated sapwood complies. Further durability tests are needed (EN 310 and EN 84) to evaluate lower retentions of the heartwood and transition wood in view of application in outdoor commodities without ground contact.

Tab. 4: Parameters expressing retention levels (uptake of CCA-salt solution [kg/m³],[kg/m²] and [%] as well as dry salt retention [kg/m³]) and penetration (mean impregnated area [%] and mean penetration depth [mm] in the RT and RL plane) specified for heartwood, transition wood and sapwood and the results of the Duncan multiple range post hoc test

4(a) Retention

Parameter	Heartwood	Transition wood	Sapwood	Duncan Test
Uptake solution [kg/m³]	124	141	278	aab
Uptake solution [kg/m²]	1.46	1.66	3.27	aab
Uptake solution [%]	28.4	32.5	69.0	aab
Uptake dry salt [kg/m³]	3.72	4.23	8.34	aab

4(b) Penetration

Parameter	Heartwood	Transition wood	Sapwood	Duncan Test
Impregnated area RT plane [%]	28.0	33.7	69.5	aab
Penetration depth RT plane [mm]	5.6	6.9	14.8	aab
Impregnated area RL plane [%]	28.1	33.8	72.0	aab
Penetration depth RL plane [mm]	4.9	6.9	14.4	aab

One can hardly state that willow wood is a refractory wood species, although there appeared to be zones in the wood structure with more difficult treatability, which complies with earlier research done on the very related poplar wood (Van Acker et al. 1990, Van Acker and Stevens 1995). Contrary to willow heartwood, poplar heartwood seems however easier to treat.

CCA preservative penetration

In addition to the product uptake level, the spatial distribution of the preservative product is of the utmost importance, as well. Sufficient retention is not always equal to a good protection and guaranteed service life.

On the cross sections A and B (Fig. 1) the impregnated surface was determined as a measure of penetration in the radial - tangential plane and expressed as a percentage of the total area. The same evaluation was made on the larger D samples characterizing the RL plane. The latter samples were also, together with the samples C examined on their actual penetration depth. All mean values per tree are given in Tab. 3b. Significant intraclonal differences are observed as pointed out by a multiple range Duncan statistic. The trees are grouped in the same way as they were for the retention values. Tree number 8 shows values being half of those of trees 4 and 5. Mean values of penetration properties were not different for the RT plane and the LR plane. Van Acker et al. (1990) reports penetration depths of 18 mm for the more treatable poplar clones, whereas the values here for willow wood remain below 10 mm, they range from 4 to 9 mm. The mean values for the various penetration parameters do not change significantly with height. In comparison with the retention features, a radial profile could also be discerned for the penetration potential of the preservative. Heartwood and transition wood (respectively ±5 mm and ±7 mm) resist harder to product penetration than the sapwood (±15 mm).

For a more in-depth evaluation of the data, two additional parameters were defined. First, the impregnated area as determined on sub samples B was recalculated to a uniform penetration depth using the following equation.

$$D_p = \frac{z - \sqrt{z^2 - S_p}}{2}$$

With D_p: calculated penetration depth [mm]
S_p: Measured impregnated area [mm^2]
z: length of the sample side [mm]

This penetration value was compared to the actual measured penetration value using a paired T-test. If the penetration front progresses homogeneously, both parameters should be equal. Statistics however, revealed significant differences for the wood in the transition zone and for the heartwood, pointing out that not all sides are equally penetrated.

Secondly, the penetration measurements on the D samples (Fig. 1) were evaluated separately for each side and again compared with a paired T-test. No significant differences were found, indicating that the penetration in the radial direction does progress homogeneously. Wood rays are mainly responsible for the transport in the radial direction and are numerous and homogeneously distributed in willow wood, supporting the findings of penetration potential. Both t-tests lead to conclude that penetration differences are mainly due to obstacles in tangential direction. In this direction transport of liquids occurs mainly through the intervascular pits. Several authors already mentioned inter- and intraspecific structural variations with regard to intervasicular pit membranes (Yazou 2005, Singh et al. 1999). Also the occurrence of aggregated lipids in areas with much larger pitting (intervasicular pits) could obstruct to waterborne preservatives (Murphy et al. 1991). These hypotheses remain subject to further research.

Table 5 shows that preservative retention is highly correlated with all penetration characteristics. Besides the classic parametric Pearson's correlation the non-parametric Kendall Tau-b correlation is shown. The latter correlation was determined because the restrictions of underlying normality and equal variances were most of the time not fulfilled. Nevertheless, the correlation is present in both cases, sometimes on a lower signification level (still $p > 0.05$).

Tab. 5: Correlation matrix for the parameters characterizing retention and penetration features with the parametric Pearson's and the non-parametric Kendall's Tau-b correlation

5(a) Pearson's correlation coefficient

Parameter correlated to:		(1)	(2)	(3)	(4)	(5)	(6)
Density [kg/m³]	(1)	1.00	-0.18**	-0.18**	-0.16**	-0.17**	-0.17**
Retention [kg/m³]	(2)		1.00	0.96**	0.91**	0.82**	0.77**
Impregnated area RT plane [%]	(3)			1.00	0.93**	0.78**	0.74**
Penetration depth RT plane [mm]	(4)				1.00	0.71**	0.71**
Impregnated area LT plane [%]	(5)					1.00	0.87**
Penetration depth LT plane [mm]	(6)						1.00

5(b) Kendall's Tau-b correlation coefficient

Parameter correlated to:		(1)	(2)	(3)	(4)	(5)	(6)
Density [kg/m³]	(1)	1.00	-0.08*	-0.09*	-0.06	-0.07	-0.07
Retention [kg/m³]	(2)		1.00	0.85**	0.77**	0.67**	0.65**
Impregnated area RT plane [%]	(3)			1.00	0.80**	0.61**	0.60**
Penetration depth RT plane [mm]	(4)				1.00	0.60**	0.57**
Impregnated area LT plane [%]	(5)					1.00	0.79**
Penetration depth LT plane [mm]	(6)						1.00

Density as a measure of porosity and treatability

In previous publications on poplar no correlation could be found between density and the uptake of preservative (Van Acker et al. 1990, Murphy et al. 1991). Here a weak negative, but significant correlation was found between the density and the impregnated area as well as the retention (Tab. 5). When using the non-parametric correlation statistic, no significant correlations could be found between density and penetration parameters. This is probably due to the fact that the radial penetration differs from the tangential penetration, resulting in larger variation coefficients on the penetration data compared to the impregnated area.

Fig. 2 shows the relationship between wood density and preservative retention expressed in kg/m^3 dry salt for the different trees at four height levels.

An increase in density with height, which has been reported earlier for poplar wood is clearly shown (Leclercq 1997, Yanchuk et al. 1983, Kroll et al. 1992, Pezlen 1998, Pilura et al. 2005, Klasnja et al. 2003). The retention tends to lower with height but stays mostly between 4 and 6 kg/m^3.

The radial density profile allows on other interpretation on the found negative correlation. Fig. 3 gives an overview of the mean product retention values against the changing radial density. The radial position mentioned in the graph corresponds to the radial distance to the pith of the middle of the considered batten. Only fully dimensioned battens were used (wane was not allowed).

The increasing density towards the bark is also been described in literature for the very similar poplar wood (Beaudoin et al. 1992, Debell et al. 2002, De Boever et al. 2007, Karki 2001). Taking into account the found correlation, sapwood should have the lowest retention values, whereas heartwood should have the highest product uptake. Fig. 3 clearly states the opposite. Only in the transition zone the correlation stated above can be withhold. In this zone we found increasing density, but still very low product retention due to its refractory character.

This clearly visualises the weak prediction potential of density as a measure for porosity and treatability of willow wood.

On the other hand, the conclusions of an overall correlation between density and retention could be wrongly interpreted by the specific penetration behaviour in the transition wood area. When sapwood and heartwood are evaluated separately, a weak significant correlation can be established between density and retention (p=0.10). More specific sampling is necessary to evaluate the latter hypothesis.

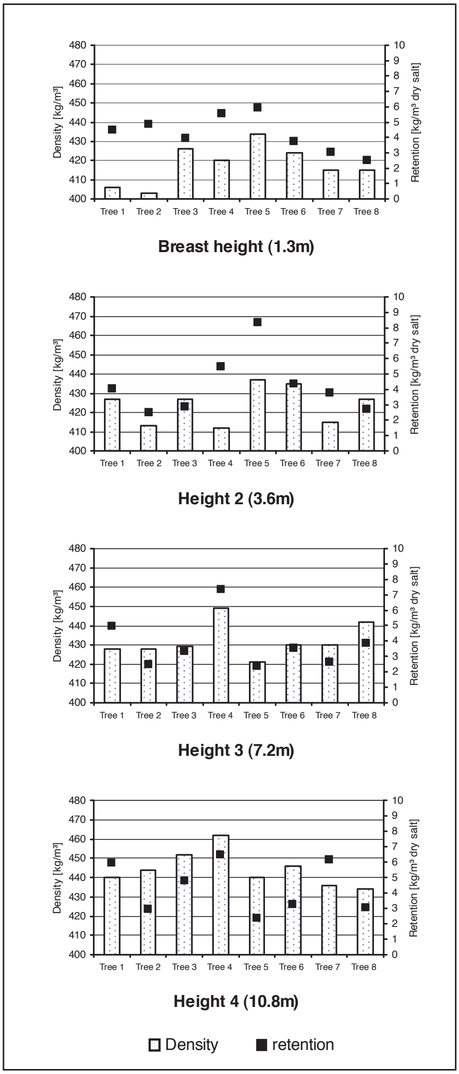

Fig. 2: Relation between wood density and the preservative retention expressed in kg/m³ dry salt for the different selected trees at four height levels

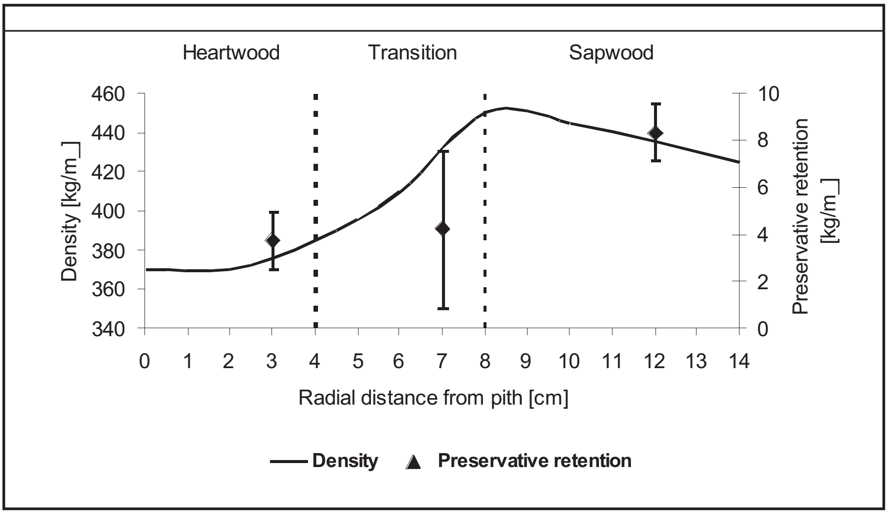

Fig. 3: Mean radial density profile for the selected trees at breast height, compared with mean preservative retention values for heartwood, transition wood and sapwood and their standard deviation

CONCLUSIONS

It can be concluded that treatability of willow wood with a CCA preservative following a Lowry process is not just based on one single parameter. Within one clone significant differences could be found between trees at one site. CCA-retention and penetration levels are much lower for heartwood and transition wood in comparison with the sapwood. Only the latter will be adequately protected in use class 3 for outdoor applications out of ground contact. The preservative penetration in radial direction progresses homogenously whereas the penetration in tangential direction is rendered more difficult. Although a significant correlation was found between density and product retention, this is only true for the transition zone with refractory characteristics. Density remains a weak parameter in predicting porosity and treatability of willow wood. In future experiments also the TL plane will be evaluated to furthermore explain the stated variability. Also different spatial positions of the transition border (sapwood-heartwood) within the batten could be evaluated to establish if the "refractory zone" occurs at the same place, irrespective of the followed pathway?

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