

## Original Research Article

# Papermaking using Willow (*Salix dasicladors*) as a Hardwood Source – A Handsheet and Pilot Paper Machine Study

### ABSTRACT

This laboratory handsheet and Fourdrinier pilot paper machine study examines the suitability of willow as a commercial hardwood pulp replacement including utilizing recycled pulp for paper production. The handsheet study contains 16 different and the pilot paper machine study eight different furnish mixtures. For both studies, the base sheet consists of a 40% softwood and 60% hardwood commercial pulp mixture, followed by replacing the hardwood with bleached willow Kraft pulp. Deinked pulp, as recycled fibers, at 5% increments up to 100% for the handsheet study and at 10%, 20%, 30%, 50%, 70% and 90% for the pilot paper machine study is added to the base pulp mixture. Both the handsheet and the FPPM study revealed that willow Kraft pulp can replace commercial hardwood pulp fibers. Basis weight, caliper and stiffness as related properties showed comparable results for the handsheet study. Willow Kraft fibers tend to be **bulkier and denser than** the used hardwood fibers.

Tensile and tear index show comparable properties for the handsheet and pilot paper machine study, except for the 50%, 70% and 90% DIP containing paper sheets.

Porosity values for the handsheet study showed lower results than the base sheet, whereas the FPPM study showed higher results except for the 70% and 90% DIP containing handsheet. Comparable results are shown for opacity. Brightness levels were significant lower due to the lower brightness of the manufactured willow Kraft and deinked pulp fiber furnish.

Despite the opportunity of replacing commercial hardwood with willow, more research needed to optimize willow into pulp fibers and preparation of the fibers for papermaking.

*Keywords: Willow, Kraft pulping, bleaching, papermaking, deinked pulp, paper properties, recycling*

### 1. INTRODUCTION

Today's pulp and paper industry around the world faces more rigid environmental regulations, globalization, high competitiveness, pressure on profit margins, threats by digital media replacing paper products, and the need to find alternative materials to sustain global competitiveness [1, 2,]. Despite that, the demand for pulp and paper product is still growing and our society is far away from becoming paperless.

One of this alternative material for the production of paper products is Willow (*Salix dasicladors*) that might **replace** existing hardwood fiber sources.

The use of willow dates back to the second century BC [3]. In North America Native Americans and European immigrants used willow for similar applications. In New York State and Pennsylvania willow cultivation was a significant income source by the late 1800s till the

29 1930s when other materials and competition from overseas resulted that only a few willow  
30 **cultivations** remained [3, 4, 5]. Willow has been investigated as a Hardwood (HW) crop for  
31 biomass applications since the mid-1980s at the State University of New York College of  
32 Environmental Science and Forestry (SUNY-ESF) [6] and is available for research purpose  
33 in sufficient quantities.

34 The willow fiber material is composed of cellulose, hemi-cellulose, lignin, extractives and ash  
35 as shown in Table 1. The principal constituents of willow compared to hardwood and  
36 softwood, the major wood material used for the production of paper all over the world is are  
37 present in nearly equal amounts [7]. There can be major variances between different wood  
38 species, dependent on their water content and growing conditions [8].

39 **Table 1: Gross chemical composition of wood for paper production**

<b>Component</b>	<b>Willow [%] [11, 12, 13]</b>	<b>Hardwood [%] [9,10]</b>	<b>Softwood [%] [9, 10]</b>
Cellulose	45-56	42-49	41-46
Hemi-cellulose	13-22	23-34	25-32
Lignin	13-26	20-26	26-31
Extractives	2-3.5	3-8	10-25
Ash	1.3-1.4	0.2-0.8	0.2-0.4

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41 This study investigates the suitability of willow for paper production including fiber  
42 preparation, and analysis of mechanical and physical paper properties incorporated into a  
43 handsheet and small laboratory pilot paper machine study.

## 44 45 **2. MATERIALS AND METHODS EXPERIMENTAL DETAILS**

46  
47 This section describes the materials, standardized TAPPI test methods, and procedures,  
48 used for this study. Repeatability of the results stayed in between the allowable margins of  
49 the TAPPI testing standards.

### 50 51 **2.1 Experimental Regime**

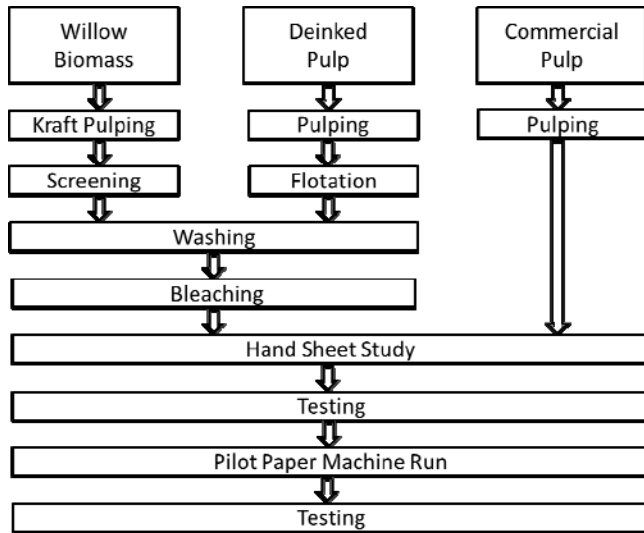
52 In this study, the process sequence illustrated in Figure a is carried out. A 3 cubic foot  
53 laboratory Digester is used to produce the Willow Kraft Pulp (WKP). After Kraft cooking the  
54 WKP is washed and disintegrated with a laboratory disperser followed by a screening  
55 treatment using a Valley type screen with a slot width of 350 microns to separate out usable  
56 fibers. Hydro pulping, followed by a flotation process sequence, produces the Deinked Pulp  
57 (DIP).

58 To improve the optical properties of the processed WKP and DIP pulp a hydrogen peroxide  
59 ( $H_2O_2$ ) bleaching process is performed, using a bag bleaching method in a hot water bath.  
60 After bleaching, samples are taken to measure Kappa number, and optical according to  
61 TAPPI standards.

62 To better design the 12" pilot paper machine run a handsheet study was performed with the  
63 produced WKP, DIP and CP pulp. Sixteen different furnishes were used to make  
64 handsheets, with all handsheets containing the same amount of filler (15%) and starch (1%).  
65 All produced handsheets were made they were tested according to TAPPI standards.

66 Based on the handsheets results a 12" Fourdrinier Pilot Paper Machine (FPPM) run is  
67 performed. A base sheet containing a 40%SW/60% HW CP mixture, and a base sheet  
68 containing a 40%SW/60% CP SW and WKP mixture was produced. Six base sheets that  
69 contain a DIP content of 10%, 20%, 30%, 50%, 70% and 90% were produced, whereas the  
70 percentage of DIP with the remaining furnish being a 40/60 ratio of CP SW to WKP.

71 The paper produced during the individual sequences is tested according to TAPPI  
 72 standards.  
 73



74  
 75 **Fig. a. Process study sequence**  
 76

77 **2.2 TAPPI Methods**

78 Pulp refining was done according to T 200 sp-06 “Laboratory beating of pulp (Valley beater  
 79 method) [14], Handsheets for physical testing were prepared accordance with T 205 sp-06  
 80 15], As was tested with T 211 0m-02, “Ash in wood pulp, paper and paperboard: combustion  
 81 at 525°C” [16],  
 82 Physical testing of handsheets was performed in accordance to T 220 sp-06, “Physical  
 83 testing of pulp handsheets” [17], the freeness of pulp was measured as Canadian Standard  
 84 Freeness (CSF) according to T 227 om-09 “Freeness of pulp (Canadian standard method)”  
 85 [18]. “Forming handsheets for physical tests of pulp”. Kappa number of the recycled pulp  
 86 was measured in accordance with T 236 om-06, “Kappa number of pulp” [19].  
 87 Screening of pulp was performed in accordance to T 274 sp-08, “Laboratory screening of  
 88 pulp (Master Screen-type instrument) [20], the instrument used was a Valley type Screen  
 89 with a 350 µm screen plate and a Voith Valley screen with 150µm screen plate. Conditioning  
 90 of the paper samples was done according to T 402 sp-08, “Standard conditioning and testing  
 91 atmospheres for paper, board, pulp handsheets, and related products” [21]. Tensile strength  
 92 was measured in accordance with T404 cm-92, “Tensile breaking strength and elongation of  
 93 paper and paperboard” [22]. Basis weight was measured with T 410 om-08. “Grammage of  
 94 Paper and Paperboard (weight per unit area)” [23]. The paper thickness was measured by T  
 95 411 om-10 “Thickness (caliper) of paper, paperboard, and combined board” [24]. Moisture  
 96 content of pulp was determined by T412 om-06 “Moisture in pulp, paper and paperboard”  
 97 [25]. The tear strength was done by following the T 414 om-12, “Internal tearing resistance of  
 98 paper (Elmendorf-type method)” [26]. Opacity of paper handsheets was performed according  
 99 to T 425 om-06, “Opacity of paper (15/d geometry, illuminant A/2°, 89% reflectance backing  
 100 and paper backing) [27]. Porosity of the paper samples was tested according to T 460 om-  
 101 06, “Air resistance of paper (Gurly method)” [28]. Brightness of pulp was measured  
 102 according to T 452 om-08, “Brightness of pulp, paper and paperboard (directional  
 103 reflectance at 457 nm)” [29]. Tensile strength was performed following T494 om-06, “Tensile  
 104 properties of paper and paperboard (using constant rate of elongation apparatus)” [30]

105  
 106 **2.3 Materials**

107 For the handsheet and small 12" pilot paper machine study the first fiber materials used was  
108 Plantation grown Willow (*Salix dasicladus*) was used. The willow was harvested at the  
109 plantation of the State University of New York, College of Environmental Science and  
110 Forestry (SUNY-ESF) in Tully, NY.  
111 The second material was DIP, produced from collected Old News Paper (ONP) and Old  
112 Magazines (OMG). The third material was CP produced from Peace River softwood, and  
113 eucalyptus hard wood.  
114 As filler material Ground Calcium Carbonate (GCC) was used.  
115 AKD cooked cationic starch was used at a consistency of 1.5% OD.  
116

## 117 2.3 Fiber Material Preparation

### 118 2.3.1. Willow Material preparation

119 Material preparation started with harvesting of 57.3 kg Willow stems (Figure 1a), with a  
120 diameter between 25 mm and 50 mm (1.0 in to 2.0 in) and 1.5 m to 1.8 m (5.0 ft. to 6 ft.) in  
121 length. The stems were manually debarked (Figure 1b) and processed with a commercial  
122 Carthage wood chipper (Figure 1c), capable of chipping a wood log of with up to 200 m in  
123 diameter. Presorting of the chips followed, using a vibrating shaker screen having a square  
124 mesh opening of 31.75 mm (1.25 in) for the top screen and a 3.2 mm (0.125 in) for the  
125 bottom screen (Figure 1d). The willow chips remaining on the top screen and the willow  
126 material falling through the bottom screen are rejected. The willow fraction of 28.1 kg  
127 remaining on top of the bottom screen was further processed in a shaker screen (Figure 1e)  
128 using sieves with a hole diameter of 28.58mm (9/8in), 22.23 mm (7/8 in), 15.88 mm (5/8 in)  
129 and 9.53 mm (3/8 in). The 9.0 kg chips remaining (Figure 1d), on the two central perforated  
130 screens, 22.23 mm (7/8 in), and 15.88 mm (5/8 in) were used for further processing.  
131



132  
133 Fig. 1. Willow processing a) willow stems, b) debarking, c) chipping, d) pre-screen, e)  
134 final screening f) willow chips [31]  
135

### 136 2.3.2. Deinked Pulp Preparation

137



138 For the project DIP is produced from collected ONP and OMG (Figure 3a). First all foreign  
 139 objects like staples, pins, and plastics are removed and glued backings are cut off. Second,  
 140 a mixture of 70% ONP and 30% OMG is prepared. The moisture analyses of the paper  
 141 mixture revealed 5% moisture content. Oven Dry (OD) of paper of the amount of 5562.6 g of  
 142 the paper mixture is added into a hydro-pulper (Figure 3b) simultaneously with 83 l of water.  
 143 After complete disintegration of the sheets for 15 minutes in the Hydro pulper, 590ml of  
 144 sodium hydroxide was added. This resulted into a pH of the pulp suspension of 9.5. Then  
 145 the temperature is raised by to about 50°C. 500 ml of Surfactant is added at 0.2% based on  
 146 OD fibers. Pulping continued for an additional 10 minutes. At the end of the pulping time, the  
 147 pulp suspension in the hydro pulper is inspected visually to ensure even disintegration and  
 148 the consistency was measured with 6.6%.  
 149 The disintegrated fibers were transferred into dump tank, diluted with water and the ink  
 150 particles floating on the surface, are skimmed off with a sieve till no floating ink particles are  
 151 present (Figure 3c). The remaining pulp was pumped into a Crofta dissolved air laboratory  
 152 flotation cell (Figure 3d) which removes ink particles with micro bubbles from the pulp  
 153 suspension. The ink foam is removed on the top of the pulp suspension is automatically  
 154 skimmed off. The deinked fibers are discharged into a screen box where some dewatering of  
 155 the fibers occurs and stored in 5-gallon plastic buckets (Figure 3e). Final dewatering to about  
 156 27.3% consistency is done using a Buchner funnel (Figure 3f). This resulted in a total OD  
 157 DIP fiber content of 4391 g, revealing an 80.2% fiber yield of the described deinking process.  
 158 The dewatered pulp is crumbled per hand and into a pre-tarred and labeled bag and stored  
 159 in the cold room until preparing the paper making pulp suspension.  
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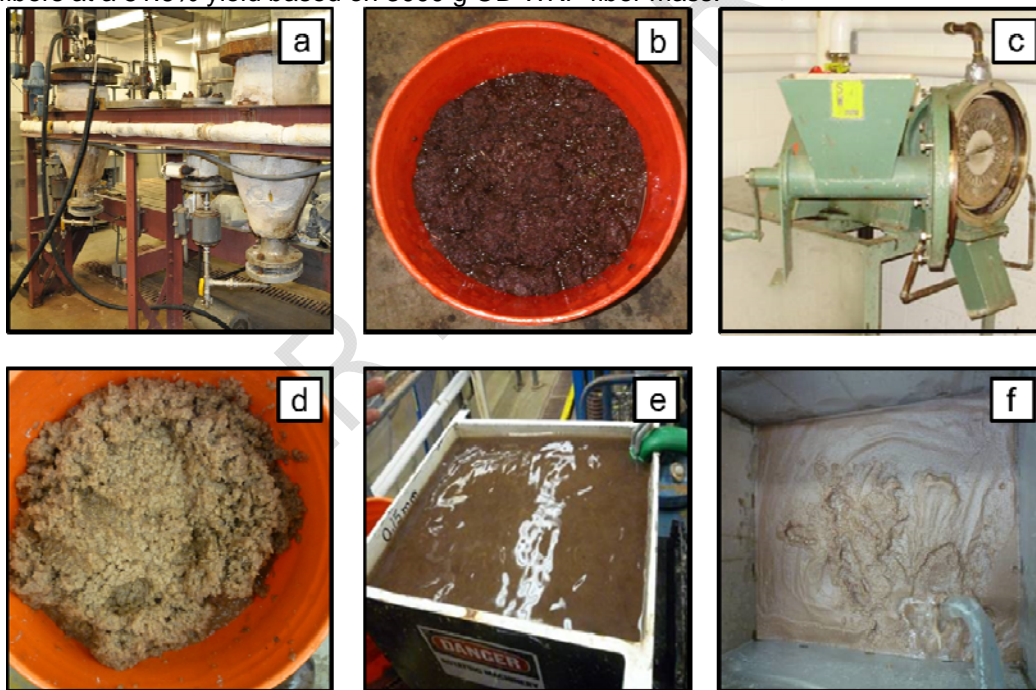
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**Fig. 3. DIP processing a) ONP & OMG, b) hydro pulping, c) ink removal, d) flotation, e) pulp storage f) dewatering [32]**

#### **2.3.4. Willow Kraft Pulping**

For the willow Kraft cooking followed the procedures outlined in [34, 35]. Instead of a MK Digester two 3 cuft digester was used (Figure 4a). From the prepared willow wood, described in Section 2.3.1 3000 g are loaded evenly in the two holding vessels of Digesters.

170 The vessel is placed in the MK Digester and covered with a perforated cover, which allows  
171 the circulation of the process chemicals in the digester. The chemical addition is based on a  
172 liquor activity of 86% based on 16% active alkali and 25% sulfidity with a 4:1 ratio of water to  
173 OD wood as described by Doelle and Schomann [33, 34].  
174 After the Digester is filled and closed, the cooking process is started. The chips' chemical  
175 solution is heated from 25°C during a 25 min. preheating phase to the cooking temperature  
176 of 155°C, followed by a 90 min. cooking phase at 155°C. The pH of the cooking liquer was  
177 12.25 at the end of the Kraft cook. After the cooking phase, both **digesters** are depressurized  
178 and the black liquor is discharged. The resulting WPKK chips were transferred a hydro  
179 pulper and enough water was added to obtain sufficient vortex mixing. The hydro pulper was  
180 operated for 10 minutes till the WKP chips were sufficient disintegrated. The content of the  
181 hydro pulper was transferred into a 150-mesh (105 mm) screen box for dewatering and then  
182 stored in a **5-gallon** pail (Figure 4b). Next, the WKP is processed in a Sprout-Bauer  
183 laboratory refiner with a plate gap set at 0.025 mm (Figure 4c). Before processing the WKP  
184 chips in the refiner, they were washed to remove unwanted impurities. After the refining,  
185 the refined WKP fibers are washed and cleaned properly in 150-mesh (0.105 mm) screen box  
186 and stored in a **5-gallon** pail (Figure 4d). In the next process step the WKP fibers pulp  
187 fraction is then screened with a Valley type screen having a screen plate width of 150  
188 microns to remove larger impurities such as splinters that did not get disintegrated during  
189 refining (Figure 4e). After screening the usable WKP fiber fraction (Figure 4d) is dewatered  
190 using a Büchner funnel method. The pulping process resulted in 1549.7 g of usable OD  
191 fibers at a 51.6% yield based on 3000 g OD WKP fiber mass.



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196

**Fig. 4. WKP processing a) 3 cuft digester, b) WKP pulping, c) Sprout-Bauer Refiner, d) refined WKP, e) WKP screening, e) WKP fibers [35]**

### 197 **2.3.5. Peroxide Bleaching**

198 Hydrogen peroxide ( $H_2O_2$ ) laboratory bag bleaching process is selected for bleaching the  
199 1549.7 g of OD WKP fibers and 4391.0 g of DIP fibers. The bag bleaching is done in a water  
200 bath with a temperature of 80 °C. The bleaching process takes place in a highly alkaline  
201 environment allowing the  $H_2O_2$  to react with certain functional groups of lignin such as the

202 carbonyl groups. The P bleaching sequence is performed under a consistency of 10% for 60  
203 minutes. Prior to bleaching, the required amount of pulp, chemicals and water is filled in a  
204 plastic bag. The plastic bag is sealed with a bag laminator. The content of the bag is then  
205 kneaded by hand to homogenize for one minute. The plastic bag is then placed in the water  
206 bath and covered with a weight to ensure complete submersion. Every 30 minutes the bag is  
207 removed and kneaded by hand for one minute. After the specified bleaching time, the plastic  
208 bag is removed from the water, opened with scissors, and the contents are emptied into a  
209 Büchner funnel attached to a filter flask connected to a vacuum system. Part of the filtrate is  
210 sampled for pH measurement. The remaining filtrate is emptied back into the Büchner funnel  
211 to recover fines. The dewatered pulp is weighed and the dryness is tested. Approximately 25  
212 g OD of the bleached pulp is sampled and placed in a beaker with deionized water for  
213 testing of kappa number, brightness and viscosity. After bleaching 25 ml  
214 Natriumhydrogensulfit ( $\text{NaHSO}_3$ ) is added to eliminate further oxidation and an associated  
215 loss of brightness. The remaining bleached pulp is disintegrated and dewatered with a  
216 Büchner funnel attached to a filter flask connected to a vacuum system. The resulting fibrous  
217 filter pad is placed on a drying paper and dried for 24 hours under a hood, after which the  
218 solids content is evaluated.

219 The chemical addition is 0.25 %  $\text{H}_2\text{O}_2$ , 0.1 %  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ , 3% NaOH, and water to achieve  
220 final bleaching consistency.

221 Hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) laboratory bag bleaching did not reveal a large effect on  
222 bleaching the DIP pulp.

223 The bleached DIP pulp had an initial Kappa number of 21.88. After bleaching the Kappa  
224 number decreased to 21.44. The ISO Brightness was increased by 1.24 points from 46.77 to  
225 47.87.

226 Bleaching of the WKP resulted in a Kappa number decreased from 42.00 to 36.24. The ISO  
227 Brightness was increased by 10.35 points from 30.25 to 40.60.

228

#### 229 **2.4 Handsheet Study**

230 To better design the 12" pilot paper machine run a handsheet study was performed with the  
231 produced WKP, DIP and CP pulp. Sixteen different furnishes were used to make  
232 handsheets, with all handsheets containing the same amount of filler (15%) and starch (1%).  
233 The **hand-sheets** consisted of a 40%SW/60%HW CP handsheet, a 40%SW/60% CP SW  
234 and WKP handsheet and fourteen handsheets that contain a 5% incremental increase of  
235 DIP from 5% to 100% whereas percentage of DIP with the remaining furnish being a 40/60  
236 ratio of CP SW to WKP.

237 Beating of the WKP, DIP and CP pulp was done in accordance to TAPPI T 200 with a  
238 consistency of  $1.57 \pm 0.04\%$  and a temperature of  $23 \pm 2^\circ\text{C}$ . The pulp was loaded into the  
239 Valley Beater, the Valley Beater was operated with no load for 3 min. After that, the initial  
240 sample was taken and the beating was initiated applying a weight of 5500g to the grinding  
241 plate lever. The pulp was **refined** to CSF value of 395. After the pulp is refined, handsheets  
242 are made to the composition mentioned above and tested according to TAPPI standards.

243

#### 244 **2.5 Papermaking Stock Preparation**

245 Based on the handsheets results a 12" Pilot Paper Machine (PPM) run was designed for  
246 three different furnish mixtures. The first furnish mixture is for the base sheet containing a  
247 40%SW/60% HW CP mixture referred to as Base Sheet (BS). The second furnish mixture  
248 contains 40% Peace River SW CP. The 60% HW eucalyptus CP was replaced with WKP,  
249 referred to as Willow Sheet (WS). The third furnish mixture was the DIP furnish that is being  
250 added to the second furnish to achieve six base sheets that contain a DIP content of 10%,  
251 20%, 30%, 50%, 70% and 90%, referred to as Willow DIP (WD) sheet. Either pulp batch is  
252 prepared in a hydro pulper followed by refining.

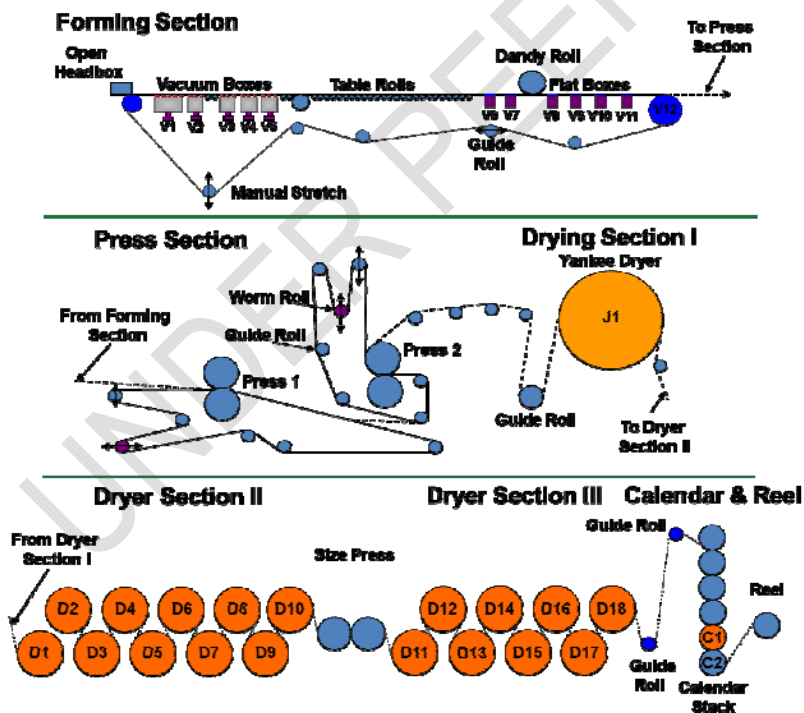
253 All furnish mixtures had a 15% filler and 1% starch content based on OD fibers. The filler  
254 was added at a 30% consistency, the starch at 3% consistency.



255 For the first and second mixture 952.5 g of SW, 1428.8 g OD of Eucalyptus HW for the first  
 256 batch and 1428.8 g of WKP for the second batch. 1410.6 g pounds of filler, and 37.6 g  
 257 pounds of starch was used as additives. The filler was assumed to have a 30% retention  
 258 rate based on previous machine runs. This would leave 421.8 g of filler in the produced  
 259 paper sheet. The third batch consisted of only DIP. The DIP furnish was made of 2857.6 g  
 260 OD DIP pulp. 1682.8 g of filler and 45.4 g of starch as additives was added. To stay  
 261 consistent with laboratory testing, each furnish batch fiber component was hydro-pulped and  
 262 refined separately. Hydro-pulping was done at 5% consistency until the flocks were  
 263 dispersed. The hydro-pulped furnish was then added to the refiner tank and diluted down to  
 264 1.9% refining consistency and refined with a Valley conical laboratory refiner to a 395 CSF  
 265 target as determined by the handsheet study. The first furnish mixture consisting of  
 266 40%SW/60% HW CP (Base Sheet) is added to machine chest 1, and the second furnish  
 267 mixture consisting of 40%SW/60% HW WKP is added to machine chest 2. The DIP  
 268 furnishes only needed to be hydro pulped to remove large flocks, since the freeness level  
 269 was already at 400 CSF. The DIP was then placed in a portable tank where it could be  
 270 added batch wise to the machine chest later on, for producing the third paper grade.  
 271

## 272 2.6 Fourdrinier Pilot Paper Machine Run

273 A 12 inches wide FPPM shown in Figure 5 was used to produce paper with the three  
 274 prepared furnish mixtures. The FPPM was operated at a speed of 1.4 m/min for the BS and  
 275 WS, and a speed 1.6 m/min for the WD base sheet. Vacuum levels for the fourdrinier table  
 276 were set at 0 for the 1<sup>st</sup>, 27579 Pa for the second vacuum section, 0 for the 3<sup>rd</sup> to 6<sup>th</sup>, 13789  
 277 Pa for the 7<sup>th</sup>, 27579 Pa for the 8<sup>th</sup>, 48263 Pa for the 9<sup>th</sup>, and 0 for the 10<sup>th</sup> vacuum section.  
 278 The fiber flow to the headbox at a consistency of 1% was set at 2.82 l/min initially and  
 279 increased up to 3.95 l/min to achieve the desired basis weight of the paper product.  
 280



302 Fig. 5. Fourdrinier pilot paper machine [36].

303  
 304 The 1<sup>st</sup> and 2<sup>nd</sup> press was operated at 206843 Pa and 275790 Pa respectively for all furnish  
 305 grades. The heat for the Yankee-Dryer is set to 50°C (122°F). The heat for dryer sections is



306 kept at 148.9°C (300°F) for the 1<sup>st</sup> and 2<sup>nd</sup>, 143°C (290°F) for the 3<sup>rd</sup> and 4<sup>th</sup>, 148.9°C  
 307 (300°F) for the 5<sup>th</sup> to the 8<sup>th</sup>, and 50°C (122°F) for the 9<sup>th</sup> dryer section. The calendar section  
 308 is operated without pressure for all three furnishes.  
 309 The 1<sup>st</sup> and 2<sup>nd</sup> press was operated at 206843 Pa and 275790 Pa respectively for all furnish  
 310 grades. The heat of the Yankee-Dryer (J1) in dryer section 1 is operated at 50°C (122°F).  
 311 The heat for the dryers in dryer section 2 is kept at 148.9°C (300°F) for the 1<sup>st</sup> to the 4<sup>th</sup> dryer  
 312 (D1-D4), 143°C (290°F) for the 5<sup>th</sup> to 10<sup>th</sup> (D5-D10) dryer. The heat in dryer section 3 was  
 313 kept at 148.9°C (300°F) for the 11<sup>th</sup> to the 16<sup>th</sup> (D11-D16) dryer, and 50°C (122°F) for the  
 314 17<sup>th</sup> and 18<sup>th</sup> (D17-D18) dryer. The calendar section for all furnishes is operated without  
 315 pressure for all calendar rolls and without heat for calendar rolls C1 and C2.  
 316  
 317

### 318 3. RESULTS AND DISCUSSION

#### 319 3.1 Handsheet Study

320 The handsheets produced for the sixteen different furnish mixtures as described in Section  
 321 2.4 contain the same filler amount and were used to make handsheets, with all handsheets  
 322 containing the same amount of filler and starch. After all **hand-sheets** are made, they were  
 323 tested for basis weight, caliper, stiffness, tear, tensile, porosity, opacity and brightness  
 324 according to TAPPI testing standards. All tested properties of the prepared handsheets,  
 325 including baseline values obtained from a commercial pale copy paper sheet made out of  
 326 100% recycled paper, are show in Table 3 and graphically displayed in Figure 6 to 9.  
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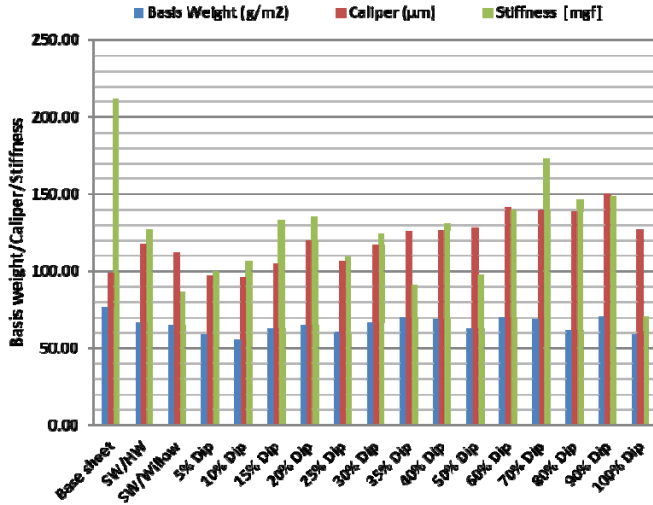
329 **Table 2: Numerical handsheet properties**

	Basis Weight [g/m <sup>2</sup> ]	Caliper [µm]	Stiffness [mgf]	Porosity [sccm]	ISO Brightness [%]	ISO Opacity [%]	Tensile Index MD [kNm/g]	Tensile Index CD [kNm/g]	Ash [%]	Tear index [mNm <sup>2</sup> /g]
Base sheet	76.50	98.60	212.23	1537.40	60.75	99.77	2.21	0.92	8.10	7.42
SW/HW	66.63	117.75	126.54	2113.50	71.18	89.05	2.60	0.96	3.47	10.21
SW/Willow	65.14	112.25	86.58	1206.67	44.93	90.08	2.39	0.74	3.63	10.04
5% Dip	59.22	97.13	99.90	1189.83	43.48	90.25	2.60	0.81	3.95	7.95
10% Dip	55.81	96.38	106.56	1648.83	43.13	90.18	2.77	0.81	4.07	8.91
15% Dip	62.80	105.38	133.20	1093.00	45.66	95.13	2.76	0.96	5.02	7.75
20% Dip	65.31	119.38	135.42	1079.00	45.47	94.99	2.71	1.02	3.35	9.41
25% Dip	60.71	106.75	108.78	678.33	44.62	95.38	2.52	0.84	4.34	8.19
30% Dip	66.80	116.88	124.32	642.83	45.25	96.80	2.55	0.69	5.70	8.62
35% Dip	70.51	125.50	91.02	611.50	44.31	97.46	2.56	0.98	5.28	8.16
40% Dip	68.67	126.13	130.98	525.33	45.85	97.37	2.30	0.81	4.89	8.19
50% Dip	62.67	128.50	97.68	686.50	44.08	98.25	2.43	0.92	5.46	10.73
60% Dip	70.26	141.50	139.86	605.67	44.16	98.36	2.49	1.01	5.43	7.98
70% Dip	68.70	140.13	173.16	748.67	42.33	98.99	2.49	0.93	6.27	11.65
80% Dip	62.10	139.13	146.52	571.00	46.14	99.07	2.43	0.87	7.00	9.27
90% Dip	70.98	150.44	148.74	734.17	42.90	99.68	2.27	0.90	9.79	9.03
100% Dip	59.14	126.96	71.04	606.33	43.64	99.51	2.51	0.91	6.13	9.73

348 The values provided in Table 2 and Figure 6 to 9 for the base sheet are the baseline values  
 349 obtained from an industry provided sheet made out of 100% recycled paper and serve as a  
 350 guide and comparison for the handsheet study.

351 Figure 6 shows the basis weight, caliper, and stiffness all together, because they are related  
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**Fig. 6. Handsheet properties: Basis weight, caliper and stiffness.**

For the basis weight the SW/HW sheet and SW/Willow sheet had comparable properties of 66.63 g/m<sup>2</sup> and 65.14 g/m<sup>2</sup> respectively. For handsheets manufactured with DIP the basis weight had a range of 55.81 g/m<sup>2</sup> (10% DIP) to 70.98 g/m<sup>2</sup> (90% DIP).

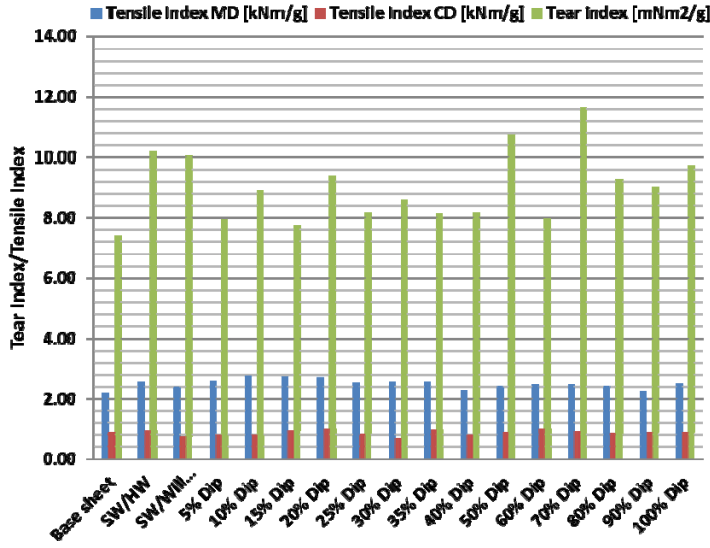
Caliper of the SW/HW sheet and SW/Willow handsheet was had comparable properties of 61.75 μm and 112.25 μm respectively. The DIP containing handsheets had lower values for the 5% to 25% content and higher values for the 30% to 90% DIP content.

The resulting stiffness was significantly higher for the SW/HW sheet (126.54 mgf) and lower for the SW /Willow handsheet (86.58 mgf). In general stiffness increased with increasing DIP content up to 90% DIP.

The basis weight fluctuations and caliper and stiffness increase with increasing DIP content might be caused by the higher fine content, filling the voids during sheet making and increasing therefore the density of the handsheet. The lower basis weight, caliper and stiffness number of the 100% DIP handsheet are related to the higher fine content of the multiple times processed DIP recycled fibers. In addition, virgin HW and SW fibers are not available as support matrix for the handsheet forming.

Figure 7 shows the tensile and tear index. As can be seen from the graph the tensile index for MD and CD is constant throughout the testing, while the tear index fluctuates greatly with DIP content from 7.75 mNm<sup>2</sup>/g for 15% DIP to 11.65 mNm<sup>2</sup>/g for 70% DIP. However, tensile and tear index of the **hand-sheets** are higher than the comparable industrial base sheet. The tensile index in CD showed a value below 0.92 kNm/g of the commercial Base Sheet for the SW/Willow and the 10%, 15%, 25%, 30%, 40%, and 80% DIP pulp containing handsheets.

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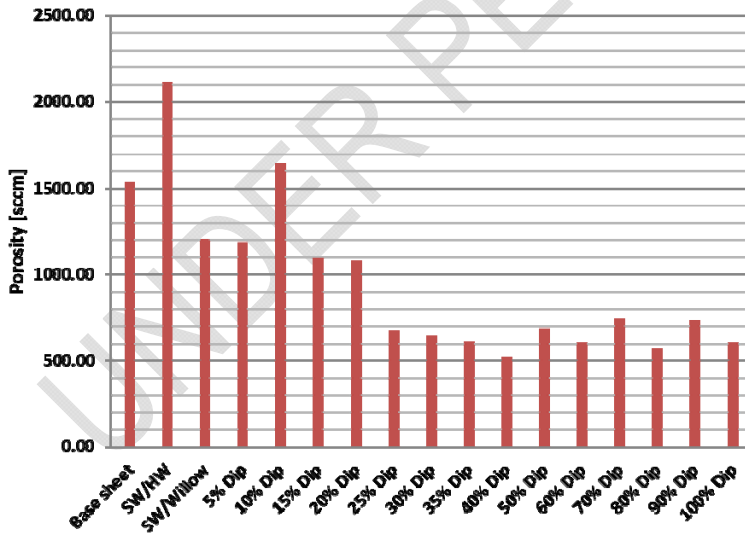
430 **Fig. 7: Handsheet properties: Tensile Index and Tear Index**

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Figure 8 shows the porosity values. The commercial Base Sheet had 1537.40 sccm compared to the SW/HW sheet with 2113.5 sccm. SW/Willow and 5% DIP sheet had similar values of 1206.67 and 1189.83 sccm. The 10% DIP sheet had with a value of 1648.83 a slightly higher value then the commercial Base Sheet. For the remaining handsheets manufactured with an increasing DIP amount the porosity value decreases below 1100 sccm for the 15% and 20% DIP sheet. The 100% DIP sheet had only a value of 606.43 sccm, half of the SW/Willow sheet.

SW/Willow and 5% DIP sheet had similar values of 1206.67 and 1189.83 sccm

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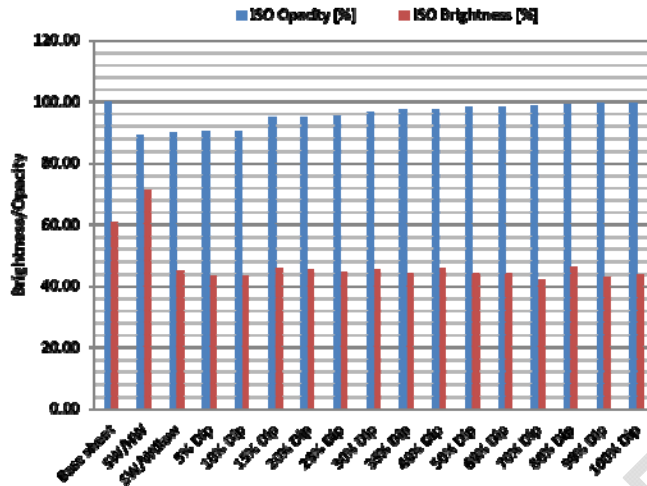


459 **Fig. 8: Handsheet properties: Porosity**

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Brightness and Opacity values are shown in Figure 9. The commercial Base Sheet had a brightness and opacity value of 60.75 and 99.77 respectively. The SW/HW sheet exceeded the brightness value due to the usage of bleached virgin HW and SW material, but was close to the opacity value of the commercial base sheet with 89.05. The HW/Willow sheet had a

465 lower brightness value of 44.93 due to low brightness value of 40.60 for the KWP. The  
 466 opacity value with 90.08 was higher than the SW/HW sheet, but still lower than the  
 467 commercial base sheet. Increasing the DIP content kept the Brightness value between 42.33  
 468 and 45.25, close to the SW/Willow sheet value. The Opacity values with increasing DIP  
 469 content above 35% achieved levels of the commercial base sheet.



487 **Fig. 9: Paper Properties: Brightness and Opacity**

489 **3.2 Fourdrinier Pilot Paper Machine Run**

490 These results from the handsheet paper study from Section 3.1 are the basis for the design  
 491 of the 12" FPPM. The study design contained a base sheet of a 60/40 CP SW and HW  
 492 mixture. For the willow sheet the 60% HW eucalyptus CP was replaced with WKP, referred  
 493 to as Willow. The third furnish mixture was the DIP furnish that is being added to the second  
 494 furnish to achieve six base sheets that contain a DIP content of 10%, 20%, 30%, 50%, 70%  
 495 and 90%. The preparation of the pulp for the study is done according to Section 2.5. Section  
 496 2.6 explains the operational procedure for the 12" FPPM.

497 The final dryness achieved of the finished paper sheets was 95%. The Filler retention was  
 498 90% which resulted into a 30% filler content in the finished paper sheets.

499 All tested properties of the FPPM paper sheets, are show in Table 3 and graphically  
 500 displayed in Figure 10 to 15.

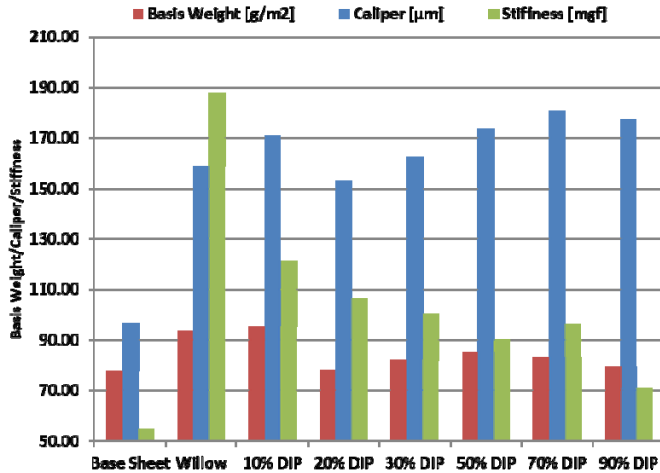
502 **Table 3: Numerical FPPM sheet properties**

	Basis Weight g/m <sup>2</sup>	Caliper [μm]	Stiffness [mgf]	Porosity [sccm]	ISO Brightnes s top [%]	ISO Brightness bottom [%]	ISO Opacity top [%]	ISO Opacity bottom [%]	Tensile index MD [kNm/g]	Tensile index CD [kNm/g]	Ash [%]	Tear index [mNm/g]
Base Sheet	78.02	96.67	54.76	451.67	90.49	90.49	100.00	100.00	0.79	0.34	31.55	8.93
Willow	93.66	159.00	187.96	676.00	64.70	64.89	99.21	99.15	1.14	0.43	31.47	8.22
10% DIP	95.58	171.00	121.36	630.00	64.77	64.76	99.23	98.75	1.00	0.41	28.37	8.29
20% DIP	78.23	153.33	106.56	593.67	62.52	62.57	99.72	99.23	1.03	0.38	26.92	10.03
30% DIP	82.22	162.67	100.64	517.33	61.13	61.12	99.38	99.43	0.85	0.36	29.93	8.43
50% DIP	85.11	173.67	90.28	459.67	58.83	58.72	99.64	99.64	0.80	0.33	31.21	7.45
70% DIP	83.44	181.00	96.20	396.67	57.61	57.59	99.85	99.94	0.68	0.32	30.84	7.05
90% DIP	79.57	177.33	71.04	340.67	56.78	56.76	100.07	99.61	0.67	0.30	33.94	6.41

516 Figure 10 shows the basis weight, caliper, and stiffness all together, because they are  
 517 related properties.



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**Fig. 10: 12” Pilot Paper Machine Paper Properties: Basis Weight, Caliper and Stiffness**

The basis weight target was 75.00 g/m<sup>2</sup> with a range of 70 g/m<sup>2</sup> to 90.00 g/m<sup>2</sup>. As shown the basis weight was within this specification for most of the run. During the run changes were made to adjust the grammage. By increasing the machine speed, the grammage would decrease and by increasing the thick stock flow the grammage would increase. Both of these changes were successful in order to keep the basis weight to specification, as can be seen in the Figure 10. The FPPM run did not achieve a caliper of 100 µm with a range of 95-105 µm. Based on the grammage and caliper data from the run, one can determine that the willow fiber is a more bulky and dense fiber than the eucalyptus fiber.

The stiffness paper property for the base sheet was 54.76 µm compared to 187.96 µm of willow sheet. Stiffness is almost completely dependent on thickness as the caliper increases so does the stiffness. The stiffness for the DIP containing paper sheets decreased from 121.35 µm for the 10% DIP containing sheet to 71.04 µm for the 90% DIP containing paper sheet.

Figure 11 shows the values achieved for the tensile index in MD and CD of the produced paper. As shown in the graph the Willow paper CD tensile index of 0.48 kNm/g has a 26.5% higher value based on the base sheet with 0.34 kNm/g. The CD tensile index for the 10% to 90% DIP containing paper sheets decreased from 0.41 kNm/g to 0.30 kNm/g.

The MD tensile index for the Willow paper of 1.14 kNm/g has a 69.3% higher value based on the base sheet with 0.79 kNm/g. The MD tensile index for the 10% to 90% DIP containing paper sheets decreased from 1.00 kNm/g to 0.67 kNm/g, whereas the 10%, 20%, 30% DIP sheets have higher MD tensile index as the base sheet.

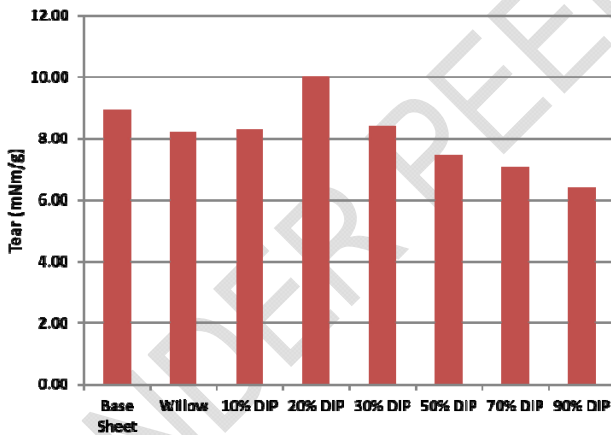
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**Fig. 11: 12" Pilot Paper Machine Paper Properties: Tensile Index**

Figure 12 shows the values achieved for the tear index. As shown in the graph, the tear index for the willow paper of 8.22 mNm/g has an 8.6% lower value based on the base sheet with 8.93 mNm/g. The tear index for the 20% DIP containing paper sheets was with 10.03 mNm/g 10.96% higher than the base sheet.

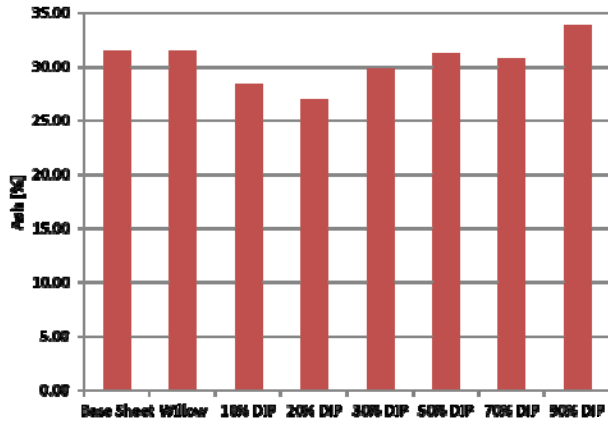
The tear index for the Willow paper containing DIP was lower than the base sheet, whereas the 10%, 20%, 30% DIP sheets have a 0.9%, 18.0%, and 2.5% higher tear index respectively as the willow sheet.



**Fig. 12: 12" Pilot Paper Machine Paper Properties: Tear**

Figure 13 shows the ash values. The base and the willow sheet are almost identical with 31.55% and 31.47%. The DIP containing paper sheets show a lower ash value between 28% to 31.21%, except for the 90% DIP containing sheet which has an 33.94% ash content.

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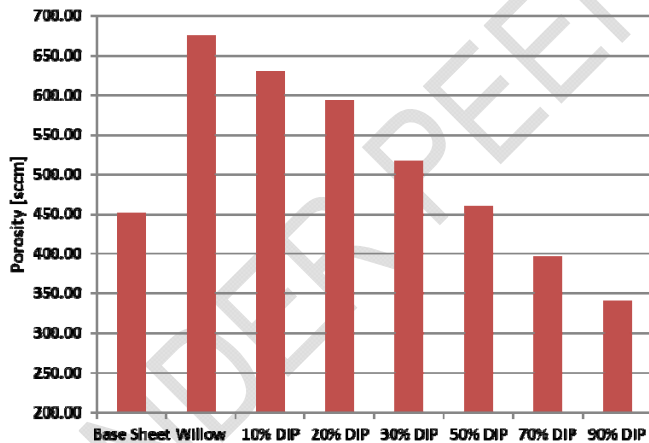


639 **Fig. 13: 12" Pilot Paper Machine Paper Properties: Ash**

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Figure 14 shows the porosity values. The commercial base sheet and 50% DIP sheet have almost identical porosity values of 451.67 and 459.67 sccm a 1.7% difference, compared to the willow sheet with 676.00 sccm which has a 33.2% higher value compared to the base sheet. As shown in the graph, the porosity value for the 10%, 20%, and 30% DIP sheet with 630.00, 593.67, and 517.33 sccm is 28.3%, 23.9%, and 12.6% respectively higher than the base sheet, but lower than the willow sheet. The 70% and 90% DIP containing sheets have a porosity of 396.67 and 340.67 sccm, a 13.86% and 32.6% lower value than the base sheet.

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663 **Fig. 14: 12" Pilot Paper Machine Paper Properties: Porosity**

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Brightness and Opacity values shown in Figure 9. The commercial Base Sheet has a brightness value of 90.49 for top and bottom and an opacity value of 100.00. The Opacity values for the willow and DIP sheet are slightly, 0.60 to 0.85% based on the base sheet, with a range of 99.15 to 99.94, except for the 90% DIP sheet that has an identical opacity as the base sheet. The brightness value of the willow and the 10% DIP containing sheet KWP pulp has a 39.7% lower brightness value of 64.70 and 64.77. Increasing the DIP content to 20% to 90% kept the brightness between 62.57 to 56.78 or 3.5% to 13.9% below the willow sheet. The lower brightness of the willow and DIP containing sheets can be explained by the lower brightness value of 40.60 for the KWP and 47.87 for the DIP pulp.

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Fig. 15: 12” Pilot Paper Machine Paper Properties: Brightness and Opacity

#### 4. CONCLUSION

The main objective of this study is to investigate if willow can replace commercial hardwood for papermaking with a handsheet and FPPM study. The handsheet study contains 16 different furnish mixtures. The FPPM study contains eight different furnish mixtures. For both studies, the base sheet contains a 40%SW/60% HW CP, followed by a 40%SW/60% HW WKP mixture. For the handsheet study, the recycled pulp contains DIP at 5% increments up to 100%, whereas for the FPPM study the DIP content is 10%, 20%, 30%, 50%, 70% and 90%.

For both the handsheet and the FPPM study revealed the result that WKP can replace HW, based on the pulp processing prior to papermaking, the chosen pulp mixtures and additives. Basis weight and caliper and stiffness as related properties showed comparable results for the handsheet study.

Based on the basis weight, caliper and stiffness data from the FPPM run, it can determine that the willow fiber is a more bulky and dense fiber than the eucalyptus fiber increasing caliper and stiffness.

Tensile and tear index showed comparable properties for the handsheet study. For the FPPM study, the tensile index in both the MD and CD direction shows higher values than for the base sheet and comparable results of the tear index, except for the 50%, 70% and 90% DIP containing paper sheets.

Porosity values for the handsheet study showed lower results than the base sheet, whereas the FPPM study showed higher results except for the 70% and 90% DIP containing handsheet.

Opacity levels revealed comparable for the handsheet and FPPM study. Brightness levels of the handsheet and FPPM study were significantly lower due to the lower brightness of the manufactured willow and DIP pulp furnish.

Despite the opportunity of replacing commercial hardwood with willow, more research needed to optimize willow into pulp fibers and preparation of the fibers for papermaking. In addition, willow production needs to be increased significantly to fulfill a future paper industry need for willow. At present time, according to the Willow Biomass Producers Handbook, about 300 to 400 Acres are utilized in 2016 for willow production [7]. Based on this, willow production is about 1200 to 2000 short tons annually, which results in approximately 600 to



730 1000 short tons of pulp at a 50% pulping yield. If all available willow would be used to  
731 produce paper pulp, a medium sized paper machine producing 500 short tons per day can  
732 be supplied for 6 to 10 days if the produced paper contains 20% willow pulp fibers. It is the  
733 hope that the potential of willow as a HW fiber source will be seen favorable by the industry  
734 in order to foster larger willow plantations.

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## 737 5. REFERENCES

738

- 739 1. Doelle K, Amaya JJ, Application of calcium carbonate for uncoated digital  
740 printing paper from 100% eucalyptus pulp. TAPPI JOURNAL, Jan. 2012;11(1):41-49.
- 741 2. Lyon SW, Quesada-Pineda HJ, Crawford SD. "Reducing electrical consumption in the  
742 forest products industry using lean thinking," *BioRes.* 2014;9(1):1373-1386
- 743 3. Volk T, Abrahamson L, Nowak C, Smart L, Tharakan P, White E. 2006. The  
744 development of short-rotation willow in the northeastern united states for bioenergy and  
745 bioproducts, agroforestry and phytoremediation. *Biomass Bioenergy.* 2006;30(8):715-27.
- 746 4. Hubbard WF, The basket willow. USDA Bureau of Forestr. 1904;46
- 747 5. Abrahamson LP, Volk TA, Smart LB, Cameron KD. 2010. Shrub willow biomass  
748 producer's handbook. Syracuse, NY: State University of New York College of  
749 Environmental Science and Forestry. 2010.
- 750 6. SUNY ESF Willow biomass Producers Handbook. 2017. Accessed January 5 2019.  
751 Available: [https://www.esf.edu/willow/documents/CropProducersHandbook2017\\_000.pdf](https://www.esf.edu/willow/documents/CropProducersHandbook2017_000.pdf)
- 752 7. Volk T, Abrahamson L, Cameron K, Castellano P, Corbin T, Fabio E, Johnson G,  
753 Kuzovkina-Eischen Y, Labrecque M, Miller R. Yields of willow biomass crops across a  
754 range of sites in north america. *Asp.Appl.Biol.* 2011;112:67-74.
- 755 8. Hübner K. Ein potentes Gas: Ozon. *Schott Info*, 2002;102.
- 756 9. Dölle K, Honig A. Laboratory Bleaching System for Oxygen and Ozone Bleaching.,  
757 *Advances Asian Journal of Chemical Science (AJOCS)*. 2018;4(2):1-12.
- 758 10. Gullichson J, Paulapuro H. *Papermaking Science and Technology. Forest Resources*  
759 *and Sustainable Management. Fapet Oy, Helsinki.* 1998.
- 760 11. Inese Sable1,\* , Uldis Grinfelds1, Laura Vikele1, Linda Rozenberga1, Dagnija Lazdina2,  
761 Martins Zeps2 and Aris Jansons. Chemical composition and fiber properties of fast-  
762 growing species in Latvia and its potential for forest bioindustry. *Estonia University of*  
763 *Life Science, Forestry Studies, Metsanduslikud Uurimused.* 2017;66:27–32.
- 764 12. Asuncion E, Suzuki K, Watabane H, Kmaya Y. Papermaking Properties of *Salix*  
765 *Serissaefolia* Pulps Prepared Under Different Pulping Condition. *J. Pac .Sci. Tech.*  
766 2009;18(6):389-401.
- 767 13. Szczukoski S., Tworkowski J., Klasa A., Stolarski M. Productivity and chemical  
768 composition of wood tissues of short rotation willow coppice cultivated on arable land.  
769 *Rostlinná Výroba*, 48. 2002;9:413-417.
- 770 14. TAPPI T 200 sp-06. Laboratory beating of pulp (Valley beater method).
- 771 15. TAPPI T 205 sp-12. Forming handsheets for physical tests of pulp.
- 772 16. TAPPI T 211 om-02. Ash in wood, pulp, paper and paperboard: combustion at 525°C.
- 773 17. TAPPI T220 sp10. Physical testing of pulp handsheets.
- 774 18. TAPPI T227 om-09. Freeness of pulp (Canadian standard method).
- 775 19. TAPPI T 236 om-13. Kappa # of pulp 2013.
- 776 20. TAPPI T 274 sp-08. Laboratory screening of pulp (Master Screen-type instrument).
- 777 21. TAPPI T 402 sp-13. Standard conditioning and testing atmospheres for paper, board,  
778 pulp handsheets.
- 779 22. TAPPI T404 cm-92 Tensile breaking strength and elongation of paper and paperboard
- 780 23. TAPPI T 410 om-08. Grammage of Paper and Paperboard (weight per unit area).
- 781 24. TAPPI T 411 om-10. Thickness (caliper) of paper, paperboard, and combined board.
- 782 25. TAPPI T412 om-06. Moisture in pulp, paper and paperboard.

- 783 26. TAPPI T414 om-12. Internal tearing resistance of paper (Elmendorf-type method).  
784 27. TAPPI T425 om-06. Opacity of paper (15/d geometry, illuminant A/2°, 89% reflectance  
785 backing and paper backing).  
786 28. TAPPI T 460 om-06. Air resistance of paper (Gurly method).  
787 29. TAPPI T 452 om-08. Brightness of pulp, paper and paperboard (directional reflectance  
788 at 457 nm).  
789 30. TAPPI T494 om-06. Tensile properties of paper and paperboard.  
790 31. Image by Klaus Dölle, Willow processing a) willow stems, b) debarking, c) chipping, d)  
791 pre-screen, e) final screening f) willow chips, jpg-file.  
792 32. Image by Klaus Dölle, Willow, DIP processing a) ONP & OMG, b) hydro pulping, c) ink  
793 removal, d) flotation, e) pulp storage f) dewatering, jpg-file.  
794 33. Doelle K, Schomann C, Amaya J.J. Hydrothermal treatment of sugar maple (acer  
795 saccharum) followed by bleaching. Lignocellulose Journal, Lignocellulose. 2015;4(1):4-  
796 21.  
797 34. Doelle K., Schomann C., (2015), "Hydrothermal Treatment of Shrub Willow Followed by  
798 Sulfate Pulping and Bleaching", International Research Journal of Pure and Applied  
799 Chemistry (JPAC). 2015;9(4):1-8.  
800 35. Image by Klaus Dölle, WKP processing a) 3 cuft digester, b) WKP pulping, c) Sprout-  
801 Bauer Refiner, d) refined WKP, e) WKP screening, e) WKP fibers, jpg-file.  
802 36. Image by Klaus Dölle, Fourdrinier pilot paper machine, jpg-file.