Original Research Article

Papermaking using Willow *(Salix dasiclados)* 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 forpaper production. The hansheet 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 more bulky and dense then the used hardwood fibers.

Tensile and tear index show comparable properties for the handsheet and pilot paper machine study, except for the 50%, 705 and 90% DIP containing paper sheets.

Porosity values for the hansheet 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, threads 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 dasiclados*) that might replacing 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

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

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

Table 1: Gross chemical composition of wood for paper production

Component	Willow [%] [11, 12, 13]	Hardwood [%] [9,10]	Softwood [%] [9, 10]
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

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

2. MATERIALS AND METHODS EXPERIMENTAL DETAILS

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

2.1 Experimental Regime

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

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

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

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

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

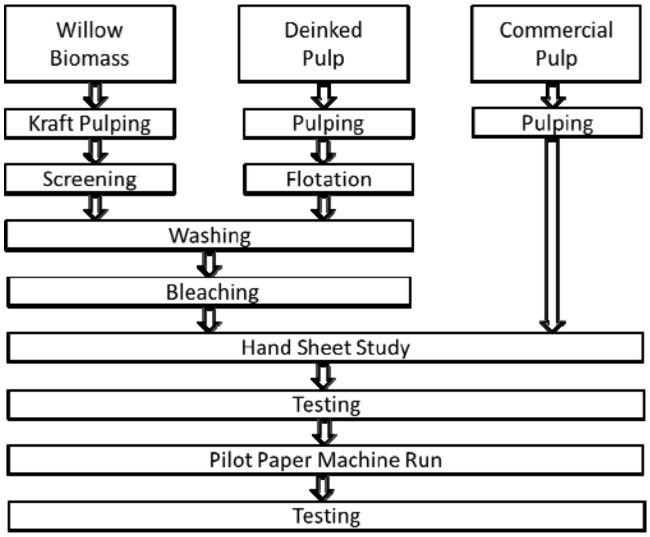

Fig. a. Process study sequence

2.2 TAPPI Methods

Pulp refining was done according to T 200 sp-06 “Laboratory beating of pulp (Valley beater method) [14], Handsheets for physical testing were prepared accordance with T 205 sp-06 15], As was tested with T 211 0m-02, “Ash in wood pulp, paper and paperboard: combustion at 525°C” [16],

Physical testing of handsheets was performed in accordance to T 220 sp-06, “Physical testing of pulp handsheets” [17], the freeness of pulp was measured as Canadian Standard Freeness (CSF) according to T 227 om-09 “Freeness of pulp (Canadian standard method)” [18]. “Forming handsheets for physical tests of pulp”. Kappa number of the recycled pulp was measured in accordance with T 236 om-06, “Kappa number of pulp” [19].

Screening of pulp was performed in accordance to T 274 sp-08, “Laboratory screening of pulp (Master Screen-type instrument) [20], the instrument used was a Valley type Screen with a 350 µm screen plate and a Voith Valley screen with 150µm screen plate. Conditioning of the paper samples was done according to T 402 sp-08, “Standard conditioning and testing atmospheres for paper, board, pulp handsheets, and related products” [21]. Tensile strength was measured in accordance with T404 cm-92, “Tensile breaking strength and elongation of paper and paperboard” [22]. Basis weight was measured with T 410 om-08. “Grammage of Paper and Paperboard (weight per unit area)” [23].The paper thickness was measured by T 411 om-10 “Thickness (caliper) of paper, paperboard, and combined board” [24]. Moisture content of pulp was determined by T412 om-06 “Moisture in pulp, paper and paperboard” [25]. The tear strength was done by following the T 414 om-12, “Internal tearing resistance of paper (Elmendorf-type method)” [26]. Opacity of paper handsheets was performed according to T 425 om-06, “Opacity of paper (15/d geometry, illuminant A/2°, 89% reflectance backing and paper backing) [27]. Porosity of the paper samples was tested according to T 460 om-06, “Air resistance of paper (Gurly method)” [28]. Brightness of pulp was measured according to T 452 om-08, “Brightness of pulp, paper and paperboard (directional reflectance at 457 nm)” [29]. Tensile strength was performed following T494 om-06, “Tensile properties of paper and paperboard (using constant rate of elongation apparatus)” [30]

2.3 Materials

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

2.3 Fiber Material Preparation

2.3.1. Willow Material preparation

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

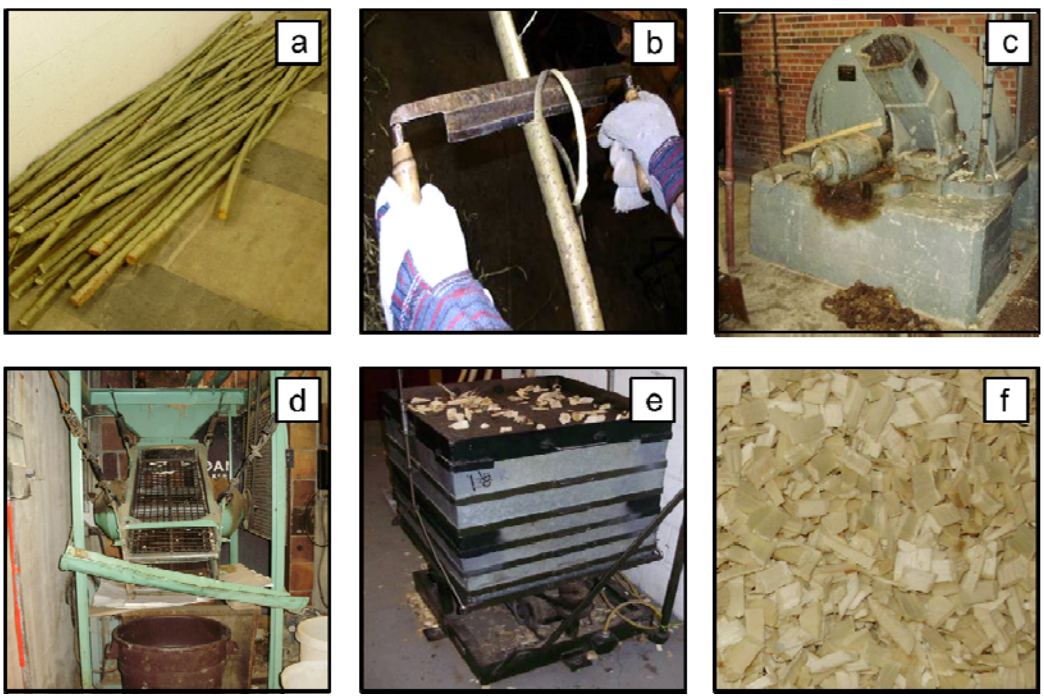

Fig. 1. Willow processing a) willow stems, b) debarking, c) chipping, d) pre-screen, e) final screening f) willow chips [31]

2.3.2. Deinked Pulp Preparation

For the project DIP is produced from collected ONP and OMG (Figure 3a). First all foreign objects like staples, pins, and plastics are removed and glued backings are cut off. Second, a mixture of 70% ONP and 30% OMG is prepared. The moisture analyses of the paper mixture revealed 5% moisture content. Oven Dry (OD) of paper of the amount of 5562.6 g of the paper mixture is added into a hydro-pulper (Figure 3b) simultaneously with 83 l of water. After complete disintegration of the sheets for 15 minutes in the Hydro pulper, 590ml of sodium hydroxide was added. This resulted into a pH of the pulp suspension of 9.5. Then the temperature is raised by to about 50^{0}C.   500 ml of Surfactant is added at 0.2% based on OD fibers. Pulping continued for an additional 10 minutes. At the end of the pulping time, the pulp suspension in the hydro pulper is inspected visually to ensure even disintegration and the consistency was measured with 6.6%.
The disintegrated fibers were transferred into dump tank, diluted with water and the ink particles floating on the surface, are skimmed off with a sieve till no floating ink particles are present (Figure 3c). The remaining pulp was pumped into a Crofta dissolved air laboratory flotation cell (Figure 3d) which removes ink particles with micro bubbles from the pulp suspension. The ink foam is removed on the top of the pulp suspension is automatically skimmed off. The deinked fibers are discharged into a screen box where some dewatering of the fibers occurs and storred in 5 gal plastic buckets (Figure 3e). Final dewatering to about 27.3% consistency is done using a Buchner funnel (Figure 3f). This resulted in a total OD DIP fiber content of 4391 g, revealing an 80.2% fiber yield of the described deinking process. The dewatered pulp is crumbled per hand and into a pre-tarred and labeled bag and stored in the cold room until preparing the paper making pulp suspension.

Fig. 3. DIP processing a) ONP & OMG, b) hydro pulping, c) ink removal, d) flotation, e) pulp storrage 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.

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

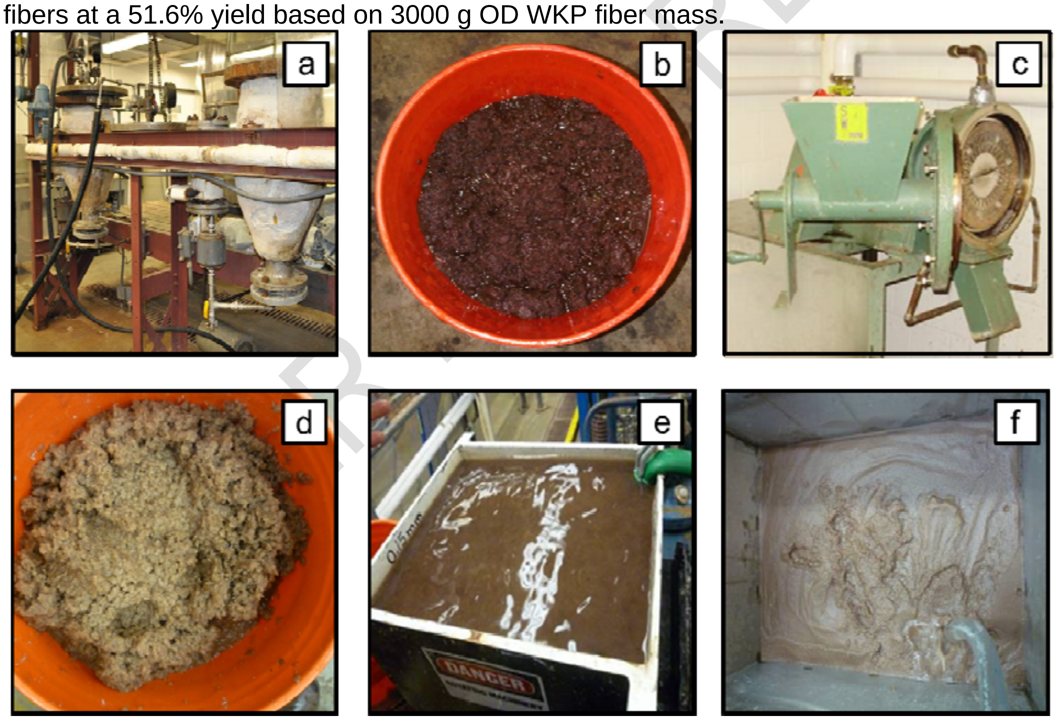

Fig. 4. WKP processing a) 3 cuft digester, b) WKP pulping, c) Sprout-Bauer Refiner, d) refined WKP, e) WKP screening, e) WKP fibers [35]

2.3.5. Peroxide Bleaching

Hydrogen peroxide (H_2O_2) laboratory bag bleaching process is selected for bleaching the 1549.7 g of OD WKP fibers and 4391.0 g of DIP fibers. The bag bleaching is done in a water bath with a temperature of 80 °C. The bleaching process takes place in a highly alkaline environment allowing the H_2O_2 to react with certain functional groups of lignin such as the carbonyl groups. The P bleaching sequence is performed under a consistency of 10% for 60 minutes. Prior to bleaching, the required amount of pulp, chemicals and water is filled in a plastic bag. The plastic bag is sealed with a bag laminator. The content of the bag is then kneaded by hand to homogenize for one minute. The plastic bag is then placed in the water bath and covered with a weight to ensure complete submersion. Every 30 minutes the bag is removed and kneaded by hand for one minute. After the specified bleaching time, the plastic bag is removed from the water, opened with scissors, and the contents are emptied into a Büchner funnel attached to a filter flask connected to a vacuum system. Part of the filtrate is sampled for pH measurement. The remaining filtrate is emptied back into the Büchner funnel to recover fines. The dewatered pulp is weighed and the dryness is tested. Approximately 25 g OD of the bleached pulp is sampled and placed in a beaker with deionized water for testing of kappa number, brightness and viscosity. After bleaching 25 ml Natriumhydrogensulfit ($NaHSO_3$) is added to eliminate further oxidation and an associated loss of brightness. The remaining bleached pulp is disintegrated and dewatered with a Büchner funnel attached to a filter flask connected to a vacuum system. The resulting fibrous filter pad is placed on a drying paper and dried for 24 hours under a hood, after which the solids content is evaluated.

The chemical addition is 0.25 % H_2O_2, 0.1 % $MgSO_4*7H_2O$, 3% NaOH, and water to achieve final bleaching consistency.

Hydrogen peroxide (H_2O_2) laboratory bag bleaching did not reveal a large effect on bleaching the DIP pulp.

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

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

2.4 Handsheet Study

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

Beating of the WKP, DIP and CP pulp was done in accordance to TAPPI T 200 with a consistency of 1.57±0.04% and a temperature of 23±2°C. The pulp was loaded into the Valley Beater, the Valley Beater was operated with no load for 3 min. After that, the initial sample was taken and the beating was initiated applying a weight of 5500g to the grinding plate lever. The pulp was refine to CSF value of 395. After the pulp is refined, handsheets are made to the composition mentioned above and tested according to TAPPI standards.

2.5 Papermaking Stock Preparation

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

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

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

2.6 Fourdrinier Pilot Paper Machine Run

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

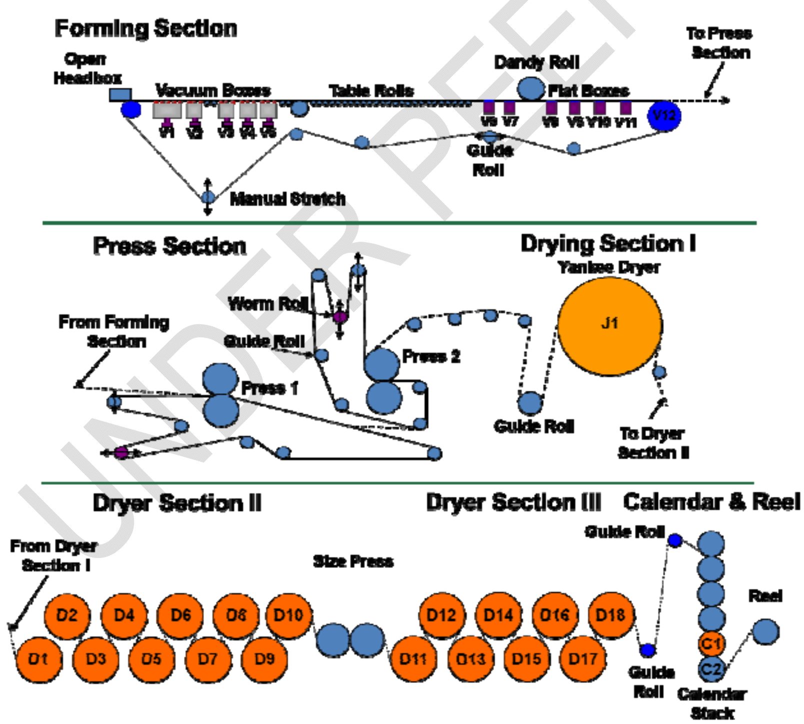

Fig. 5. Fourdrinier pilot paper machine [36].

The 1st and 2nd press was operated at 206843 Pa and 275790 Pa respectively for all furnish grades. The heat for the Yankee-Dryer is set to 50°C (122°F). The heat for dryer sections is kept at 148.9°C (300°F) for the 1st and 2nd, 143°C (290°F) for the 3rd and 4th, 148.9°C (300°F) for the 5th to the 8th, and 50°C (122°F) for the 9th dryer section. The calendar section is operated without pressure for all three furnishes.

The 1st and 2nd press was operated at 206843 Pa and 275790 Pa respectively for all furnish grades. The heat of the Yankee-Dryer (J1) in dryer section 1 is operated at 50°C (122°F). The heat for the dryers in dryer section 2 is kept at 148.9°C (300°F) for the 1st to the 4th dryer (D1-D4), 143°C (290°F) for the 5th to 10th (D5-D10) dryer. The heat in dryer section 3 was kept at 148.9°C (300°F) for the 11th to the 16th (D11-D16) dryer, and 50°C (122°F) for the 17th and 18th (D17-D18) dryer. The calendar section for all furnishes is operated without pressure for all calendar rolls and without heat for calendar rolls C1 and C2.

3. RESULTS AND DISCUSSION

3.1 Handsheet Study

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

Table 2: Numerical handsheet properties

	Basis Weight [g/m^2]	Caliper [µm]	Stiffness [mgf]	Porosity [sccm]	ISO Brightness [%]	ISO Opacity [%]	Tensile Index MD [kNm/g]	Tensile Index CD [kNm/g]	Ash [%]	Tear index [mNm^2/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

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

Figure 6 shows the basis weight, caliper, and stiffness all together, because they are related properties.

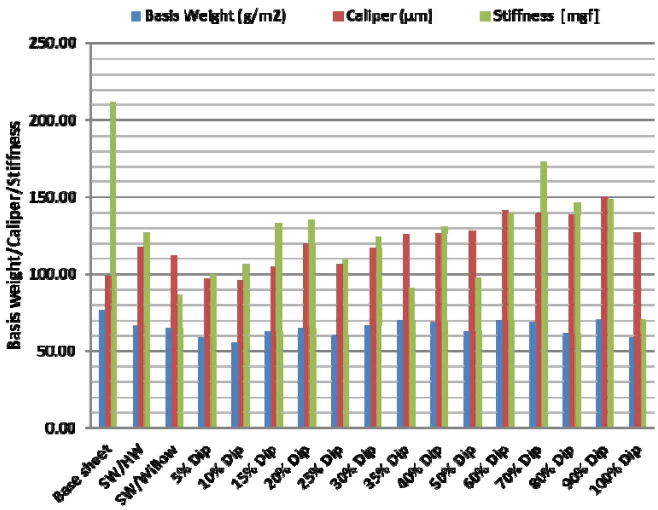

Fig. 6. Handsheet properties: Basis weight, caliper and stiffness.

For the basis weight the SW/HW sheet and SW/Willow sheet had comparable properties of 66.63 g/m² and 65.14 g/m² respectively. For handsheets manufactured wit DIP the basis weight had a range of 55.81 g/m² (10% DIP) to 70.98 g/m² (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 significant 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 cause 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 processes 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 mNm²/g for 15% DIP to 11.65 mNm²/g for 70% DIP. However, tensile and tear index of the handsheets are higher than the comparable industrial bases 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.

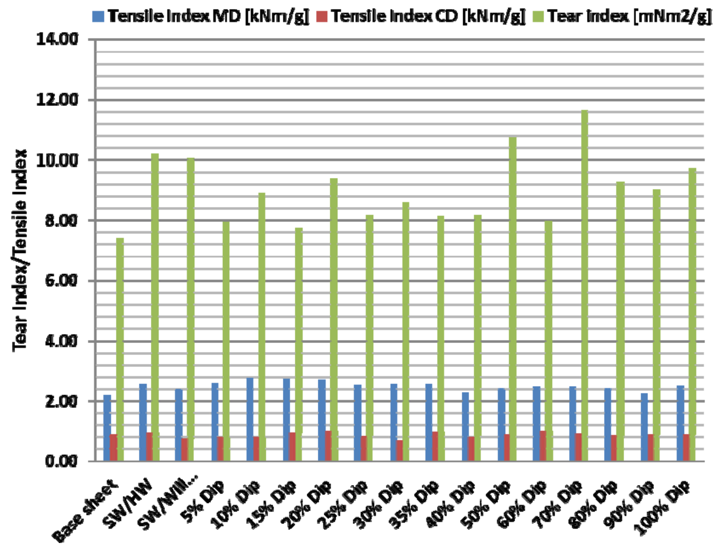

Fig. 7: Handsheet properties: Tensile Index and Tear Index

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

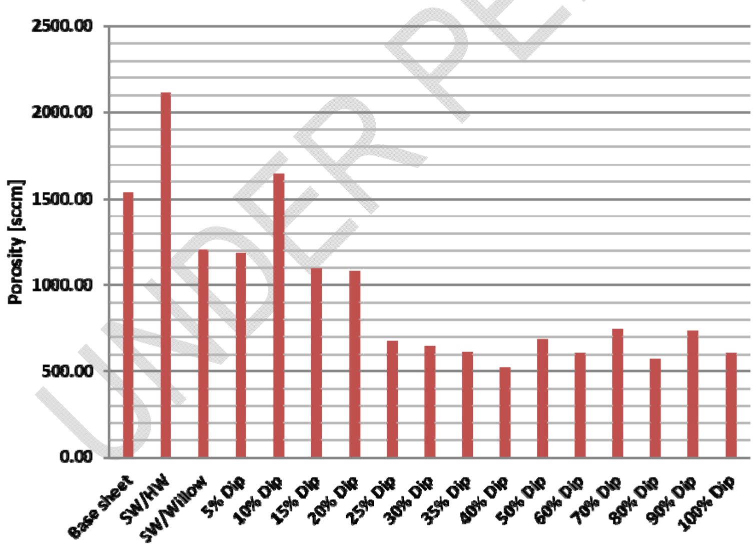

Fig. 8: Handsheet properties: Porosity

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

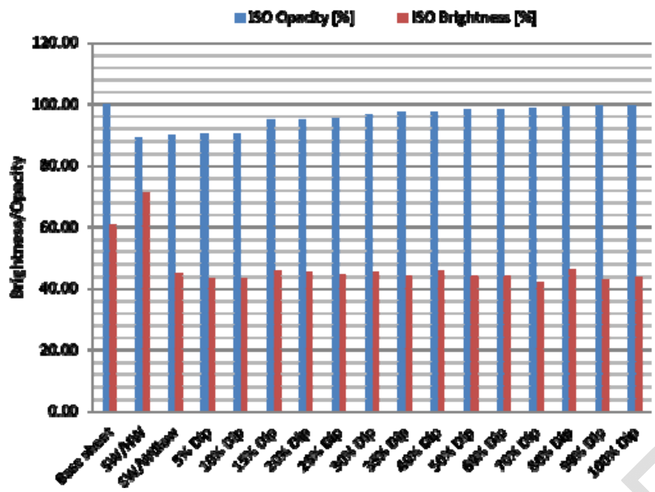

Fig. 9: Paper Properties: Brightness and Opacity

3.2 Fourdrinier Pilot Paper Machine Run

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

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

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

Table 3: Numerical FPPM sheet properties

	Basis Weight g/m^2	Caliper [µm]	Stiffness [mgf]	Porosity [sccm]	ISO Brightness 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

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

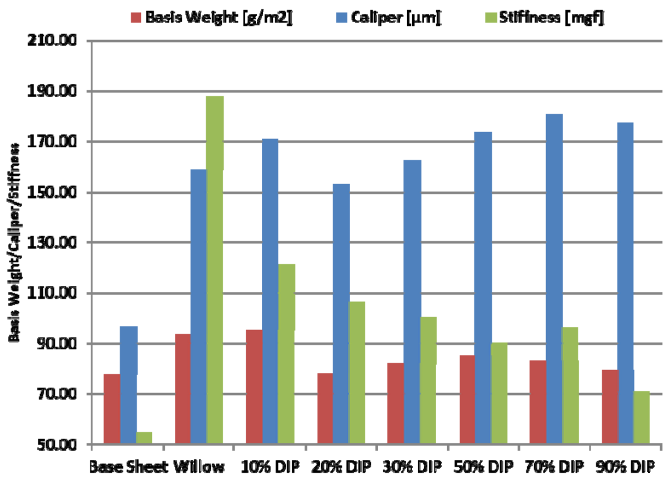

Fig. 10: 12" Pilot Paper Machine Paper Properties: Basis Weight, Caliper and Stiffness

The basis weight target was 75.00 g/m² with a range of 70 g/m² to 90.00 g/m². 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 containg 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.

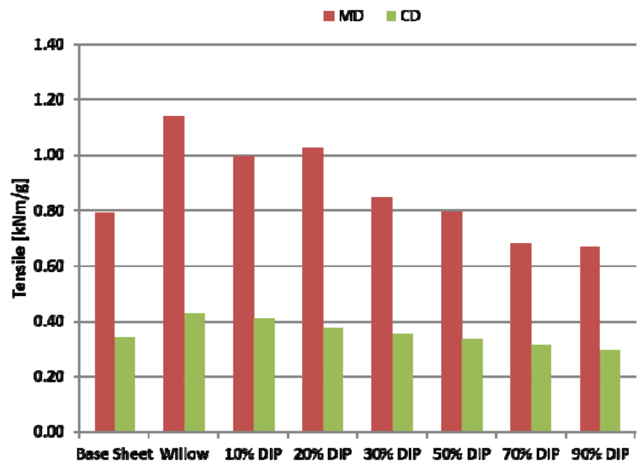

Fig. 11: 12” Pilot Paper Machine Paper Properties: Tensile Index

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 kNm/g has an 8.6% lower value based on the base sheet with 8.93 kNm/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.

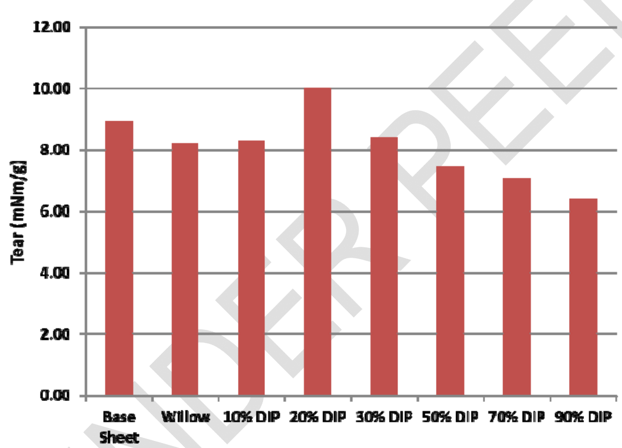

Fig. 12: 12” Pilot Paper Machine Paper Properties: Tear

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.

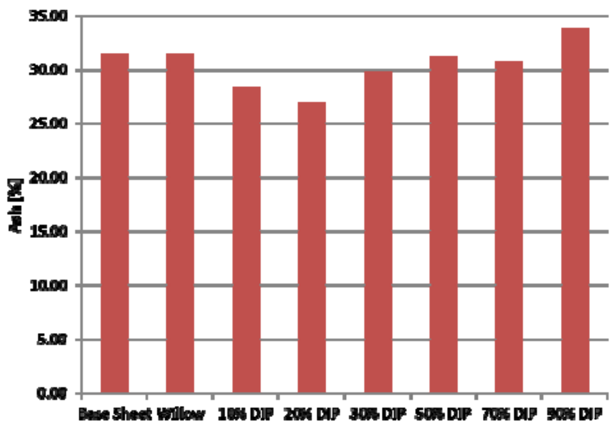

Fig. 13: 12" Pilot Paper Machine Paper Properties: Ash

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 compare d 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 then the base sheet.

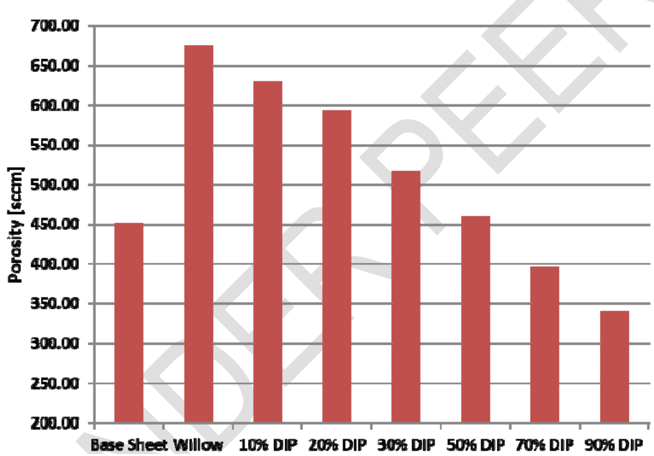

Fig. 14: 12" Pilot Paper Machine Paper Properties: Porosity

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 rage 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.

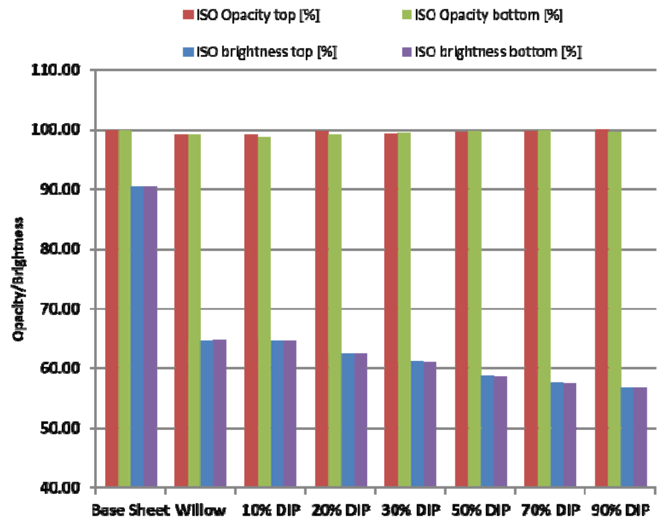

Fig. 15: 12" Pilot Paper Machine Paper Properties: Brightness and Opacity

4. CONCLUSION

This studies chare is to investigate if willow can replace commercial hardwood for papermaking with a handsheet and FPPM study. The hansheet study contains 16 different furnish mixtures. The FPPM study and 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 hansheet 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 hansheet 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 hadsheet 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%, 705 and 90% DIP containing paper sheets.
Porosity values for the hansheet 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 significant 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 Acers 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 1000 short tons of pulp at a 50% pulping yield. If all available willow would be used to

produce paper pulp, a medium sized paper machine producing 500 short tons per day can be supplied for 6 to 10 days if the produced paper contains 20% willow pulp fibers. It is the hope that the potential of willow as a HW fiber source will be seen favorable by the industry in order to foster larger willow plantations.

5. REFERENCES

1. Doelle K, Amaya JJ, Application of calcium carbonate for uncoated digital printing paper from 100% eucalyptus pulp. TAPPI JOURNAL, Jan. 2012;11(1):41-49.
2. Lyon SW, Quesada-Pineda HJ,Crawford SD. "Reducing electrical consumption in the forest products industry using lean thinking," *BioRes.* 2014;9(1):1373-1386
3. Volk T, Abrahamson L, Nowak C, Smart L, Tharakan P, White E. 2006. The development of short-rotation willow in the northeastern united states for bioenergy and bioproducts, agroforestry and phytoremediation. Biomass Bioenergy. 2006;30(8):715-27.
4. Hubbard WF, The basket willow. USDA Bureau of Forestr. 1904;46
5. Abrahamson LP, Volk TA, Smart LB, Cameron KD. 2010. Shrub willow biomass producer’s handbook. Syracuse, NY: State University of New York College of Environmental Science and Forestry. 2010.
6. SUNY ESF Willow biomass Producers Handbook. 2017. Accessed January 5 2019. Available: https://www.esf.edu/willow/documents/CropProducersHandbook2017_000.pdf
7. Volk T, Abrahamson L, Cameron K, Castellano P, Corbin T, Fabio E, Johnson G, Kuzovkina-Eischen Y, Labrecque M, Miller R. Yields of willow biomass crops across a range of sites in north america. Asp.Appl.Biol. 2011;112:67-74.
8. Hübner K. Ein potentes Gas: Ozon. Schott Info, 2002;102.
9. Dölle K, Honig A. Laboratory Bleaching System for Oxygen and Ozone Bleaching., Advances Asian Journal of Chemical Science (AJOCS). 2018;4(2):1-12.
10. Gullichson J, Paulapuro H. Papermaking Science and Technology. Forest Resources and Sustainable Management. Fapet Oy, Helsinki. 1998.
11. Inese Sable1,*, Uldis Grinfelds1, Laura Vikele1, Linda Rozenberga1, Dagnija Lazdina2, Martins Zeps2 and Aris Jansons. Chemical composition and fiber properties of fast-growing species in Latvia and its potential for forest bioindustry. Estonia University of Life Science, Forestry Studies, Metsanduslikud Uurimused. 2017;66:27–32.
12. Asuncion E, Suzuki K, Watabane H, Kmaya Y. Papermaking Properties of Salix Serissaefolia Pulps Prepared Under Different Pulping Condition. J. Pac .Sci. Tech. 2009;18(6):389-401.
13. Szczukoski S., Tworkowski J., Klasa A., Stolarski M. Productivity and chemical composition of wood tissues of short rotation willow coppice cultivated on arable land. Rostlinná Výroba, 48. 2002;9:413-417.
14. TAPPI T 200 sp-06.Laboratory beating of pulp (Valley beater method).
15. TAPPI T 205 sp-12. Forming handsheets for physical tests of pulp.
16. TAPPI T 211 om-02. Ash in wood, pulp, paper and paperboard: combustion at 525°C.
17. TAPPI T220 sp10. Physical testing of pulp handsheets.
18. TAPPI T227 om-09. Freeness of pulp (Canadian standard method).
19. TAPPI T 236 om-13. Kappa # of pulp 2013.
20. TAPPI T 274 sp-08,. Laboratory screening of pulp (Master Screen-type instrument).
21. TAPPI T 402 sp-13. Standard conditioning and testing atmospheres for paper, board, pulp handsheets.
22. TAPPI T404 cm-92 Tensile breaking strength and elongation of paper and paperboard
23. TAPPI T 410 om-08. Grammage of Paper and Paperboard (weight per unit area).
24. TAPPI T 411 om-10. Thickness (caliper) of paper, paperboard, and combined board.
25. TAPPI T412 om-06. Moisture in pulp, paper and paperboard.
26. TAPPI T414 om-12. Internal tearing resistance of paper (Elmendorf-type method).

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