Common Reed (*Phragmites australis*), Eradicate or Utilize? Part II: Potential Use as an Industrial Fiber Source after Hot Water Extraction

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The potential usefulness of an invasive common reed as biomass feed to a biorefinery was investigated. This investigation focused on the cellulosic fiber and a comparison of with and without a hot-water extraction (HWE) pretreatment process step. Handsheets were made before and after bleaching and compared to handsheets made from other pulped grass family (Poaceae) fibers. Machine-made simulated copy-grade paper was compared with and without HWE at varied percentages of reed replacement for hardwood fiber in the furnish. The HWE appeared to cause a dramatic increase in the tensile and burst strength while the tear strength reduced slightly. The effects of HWE on woody biomass strength properties were compared.

Keywords: Phragmites australis; Invasive species; Renewable fuel; Biochemicals; Hot water extraction; Value prior to pulping; Soda pulping; Industrial fiber source; Elemental chlorine free bleaching; Machine-made paper

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INTRODUCTION

Common reed, (*Phragmites australis*) (Cav.), Trin. ex Steudel is native to North America. However, within the last few decades an aggressive European genotype (Saltonstall 2002) has produced dense monoculture or near monoculture stands in the wet regions of southern Canada and the United States (Chambers *et al.* 1999; Meyerson *et al.* 2000; Tulbure *et al.* 2007). Common reed is a member of the grass (Poaceae) family, and its crop yield fares well compared to other grasses that the Environmental Protection Agency (EPA) approved as renewable fuels under the Renewable Fuels Standard (RFS) program. This reed species would have a distinct advantage as a designated cellulosic energy crop because reed grows well even in marginal soils, and therefore it would not compete with prime agricultural land feed crops. The concept here is why not utilize this prolific plant biomass rather than attempt eradication, which has proven very difficult and nearly impossible once established (Holm *et al*. 1977; Tewksbury *et al*. 2002).

Part I of this investigation (Burry *et al.* 2014) compared common reed with other renewable energy crops but with a focus on the potential benefit when processing the complete aboveground crop into multiple bioproducts, rather than total crop conversion into biofuel. Such processing is consistent with the concept of a biorefinery (Kamm and Kamm 2004; Fernando *et al.* 2006; Amidon *et al.* 2008). Various forms of biorefineries have existed for decades with the most notable being the kraft pulp and paper mill (Yoon and van Heiningen 2008; Kautto *et al*. 2010; Jun *et al*. 2012; Johnson and Hart 2016). The separation and refining of extractives and hemicelluloses into biochemicals is referred to as "Value Prior to Pulping" or "VPP" when integrated into a pulp mill (Thorp and Raymond 2004; Liu *et al*. 2012). The pulping process focuses on lignin separation. Part I of this investigation included extractives, hemicelluloses, and lignin separation and compared results with other grasses. The remaining biomaterial is primarily cellulosic fiber. This is Part II of the investigation, to characterize pulped common reed fiber, fiber bleachability, and fiber papermaking quality.

Many researchers have subjected various grasses to a hot water extraction (HWE) process (Shatalov and Pereira 2002; Hu *et al.* 2010; Madakadze *et al.* 2010; Villaverde *et al.* 2010; Williams and Biswas 2010; Abril *et al.* 2012; Burry *et al.* 2014). Other researchers have processed various grasses through a soda pulping process (Khristova *et al.* 2006; Kumar *et al.* 2013; Burry *et al.* 2014) or a kraft pulping process (Goel *et al.* 1998; Thykesson *et al.* 1998; Byrd 2000; Shatalov and Pereira 2002; Madakadze *et al.* 2010; Williams and Biswas 2010). Additionally, some of these researchers bleached their pulped grass fibers (Goel *et al.* 1998; Byrd 2000; Shatalov and Pereira 2006; Khristova *et al.* 2006; Williams and Biswas 2010; Burry *et al.* 2014). The test sheets of paper were hand formed (handsheets) from unbleached and bleached pulped grasses to determine several paper properties. None of these mentioned studies, except Burry *et al.* 2014, made handsheets from pulped grasses that were first subjected to a HWE treatment consistent with a biorefinery operation. In addition, this investigation includes paper properties on machine-made paper that was first subjected to HWE followed by soda pulping and one stage of bleaching. Machine-made paper properties were compared to similarly processed reed fibers without HWE.

EXPERIMENTAL

Materials

Two lots of the common reed, *Phragmites australis,* were harvested from test beds at the Minoa Wastewater Treatment Facility (Minoa, NY, USA). The first lot (S11) was harvested in January and represented the crop growing during the previous growing season. The second lot (S12) was harvested in the fall and represented the current growing season. Both lots were air-dried prior to one pass through a 5 hp chipper/shredder (MTD, Valley City, OH, USA). The group S11 was screened at 12-mesh with fines discarded, which represented 12.8% by mass of the whole. The remaining material included stalk segments up to three inches in length. Whole S12 harvested material was used for experimentation with stalk segments up to five inches in length and large tassel fines.

Hot water extraction (S12 P. australis lot material only)

Hot water extractions (HWE) were performed in 2 kg feed batches (based on oven-dried biomass) of S12 reed material tightly wrapped in cotton cloth, fitted into a pair of screened baskets and placed into a one cubic foot digester. Water was added to achieve an 8:1 ratio of water to oven-dried (o.d.) biomass feed.

Batch extractions were conducted to three different final temperatures of 150 °C, 160 °C, and 170 °C. Further description of the HWE performed may be found in Part I of this study (Burry *et al.* 2014).

Soda chemical pulping

Both S11 (without HWE treatment) and S12 (after HWE treatment) lots of reed material were subjected to soda cooking in one cubic foot batch digesters. The S11 cooks used 16 h presoaks with water as feed. For the S12 soda cooks, the feed was a continuation of the HWE.

The make-up water was calculated to achieve an 8:1 ratio of soda cooking liquor to the feed material (based on pre-extracted o.d. feed). See Part I of this study for further information on the soda chemical pulping that was performed (Burry *et al.* 2014).

Bleaching

Table 1 summarizes three elemental chlorine free (ECF) bleaching conditions. All soda cooked screened accepts were subjected to one stage of alkaline peroxide (P) bleaching in 150 g (o.d.) batches sealed in poly bags and submerged into a large steam heated water bath. Bleaching conditions included 3% sodium hydroxide (Fisher Scientific Company LLC, Pittsburgh, PA, USA) and 3% hydrogen peroxide (VWR, Radnor, PA, USA) based on o.d. screened accepts pulp feed. Bleaching was performed at 15% consistency. The bags were heated for 1 h at an average temperature of 85 °C. All of the peroxide bleached pulps were washed well before further analysis and processing.

Table 1. ECF Bleaching Conditions on *P. australis*

	S11	S12
Peroxide (*P*) with Alkali		
Pulp consistency (%)	15	15
Temperature (°C)	85	85
Time (min)	60	60
Hydrogen peroxide (% o.d. soda pulp)	3.0	3.0
Sodium hydroxide (% o.d. soda pulp)	3.0	3.0
Chlorine Dioxide, ClO_2 (*D*)		
Pulp consistency (%)	10	10
Temperature (°C)	70	70
Time (min)	60	60
Chlorine dioxide (% o.d. peroxide pulp)	*	*
Alkali Extraction with Oxygen (E_o)		
Pulp consistency (%)	7.5	7.5
Temperature (°C)	70	70
Time (min)	60	60
Oxygen (psi)	50	50
Sodium hydroxide (% o.d. ClO_2 pulp)	2.5	2.5

* % ClO_2 calculated as 0.1 X peroxide pulp Kappa number

Portions of S11 160 °C and S12 160 °C peroxide-bleached pulps were subjected to two more stages of bleaching using a Quantum Mark IV reactor in 200 o.d. g to 300 o.d. g batches. A chlorine dioxide (D) second stage was performed. The percent chlorine dioxide applied to o.d. pulp (P stage product) was based on 0.1 times the P-stage Kappa number (T 236). The D-stage bleaching was conducted at 10% consistency for 1 h at 70 °C. After thorough washing, a small portion of the D-stage bleached pulp was retained for analysis while the remaining pulp was processed through a third stage of bleaching.

The third stage of bleaching was an alkali extraction enriched with oxygen (E_o) performed at 7.5% consistency with 2.5% sodium hydroxide (based on o.d. pulp feed) and with 50 psig oxygen pressure supplied by a compressed gas cylinder. The washed final stage bleached pulps were analyzed.

Machine-made paper

Sufficient quantities of both *P. australis* lots were batch processed to provide enough fiber for comparative runs on a 12-in wide pilot paper machine (Eastern Machine Builders Corp., Queens Village, NY, USA). The paper machine was initially started (lined-out) with a control furnish of 60% bleached *Eucalyptus* hardwood (HW) kraft and 40% northern bleached softwood (SW) kraft refined through a conical Jordan refiner (Allis-Chalmers Co., Milwaukee, WI, USA) until a Canadian Standard Freeness (CSF) of 400 mL was obtained. Ground calcium carbonate (GCC, at 20% to o.d. fiber) and cationic starch (at 1% to o.d. fiber for the S11 run and at 3% to o.d. fiber for S12 run) was added to the machine chest. The objective was to produce a copy-grade paper at 75 grammage and 100 µm thickness. Cooked and bleached *P. australis* fiber already at 375 CSF (without refining) was incrementally added to replace the hardwood fiber in two blended furnishes of 30% HW:30% Reed:40% SW and 60% Reed:40% HW before running 100% Reed. The replacement *Phragmites* reed fiber also contained 20% GCC and starch (at 1% to o.d. fiber for the S11 run and at 3% to o.d. fiber for S12 run).

Methods

Soda pulping

The S11 and S12 pulp yields were calculated and reported in Part I of this investigation (Burry 2014). The Kappa number was reported in Part I of this investigation but is also included here for continuity according to the TAPPI T236 om-06 (2006) standard. The handsheets were prepared following the TAPPI T205 sp-06 (2006) standard, except that 1.5 g sheets were produced (75 grammage, g/m^2) from unbleached and unrefined pulps.

Bleaching

The Kappa number (T 236) was determined on pulps after each bleaching stage. Handsheets were prepared on the unrefined bleached pulps and paper properties measured (for each bleaching stage) as discussed above for the unbleached and unrefined soda pulps.

Paper testing

Handsheets were conditioned in a controlled environment as per TAPPI T402 sp-08 (2008) standard followed by testing the most relevant characteristics in pulping and bleaching, namely z-span (TAPPI T231 cm-07 (2007), using a Pulpmac ZST-15 instrument (Pulmac Systems, Williston, VT, USA). The brightness and opacity were measured using a Technidyne Colortouch II instrument (Technidyne, New Albany, IN, USA) according to ISO 2469 (2007) and ISO 2471 (2008), respectively. Grammage (g/m^2) as per the TAPPI T220 sp-10 (2010) standard was performed to correct each z-span value for variations in the sheet grammage.

Representative portions of each machine-made paper furnish were conditioned following the TAPPI T402 sp-08 (2008) standard. However, two furnishes were no longer available when deciding to report this investigation, the control furnish (60% HW:40% SW) for the S11 run and the 30% HW:30% Reed:40% SW furnish for the S12 run. The control furnish for the S12 run (also 60% HW:40% SW) may be considered a reasonable control run for S11. Paper testing of machine-made papers included those tests methods and instruments listed in Table 2. The tensile and tear strength properties were measured in both machine direction (m.d.) and cross-machine direction (c.d.).

Table 2. Machine Made Paper Testing Methods

Paper Property	Test Standard	Instrument
Grammage (g/m^2)	TAPPI T410 om-08 (2008)	Balance B303 (Mettler Toledo, Columbus, OH, USA)
Thickness (mm)	TAPPI T411 om-10 (2010)	Micrometer SE 050 (L&W, Kista, Sweden)
Apparent density (g/cm^3)	TAPPI T220 sp-10 (2010)	-
Roughness (sccm)	TAPPI T538 om-08 (2008)	Sheffield (TMI, New Castle, DE, USA)
Porosity (sccm)	TAPPI T460 om-11 (2011)	Densitometer 121 (L&W, Kista, Sweden)
Brightness (%)	ISO 2469 (2007)	Colortouch II (Technidyne, New Albany, IN, USA)
Opacity (%)	ISO 2471 (2008)	Colortouch II (Technidyne, New Albany, IN, USA)
Burst index (kN/g)	TAPPI T403 om-10 (2010)	Burst SE 002 (L&W, Kista, Sweden)
Tensile index (N·m/g)	TAPPI T494 om-06 (2006)	Tensile Sintech 1/S (MTS, Eden Prairie, MN, USA)
Tear index (mN·m^2/g)	TAPPI T414 om-04 (2004)	Tear 83-11-01 (TMI, New Castle, DE, USA)
Stiffness	TAPPI T566 om-08 (2008)	Stiffness 150-E (Taber Ind., N. Tonawanda, NY, USA)
Ash content (%)	TAPPI T211 om-07 (2007)	Furnace 808 (Lindberg, Riverside, MI, USA)

RESULTS AND DISCUSSION

Pulping Comparative Data

The soda cooked *Phragmites australis* (common reed as C. Reed) results from this current investigation and Kumar *et al.* (2013) are shown in Table 3 (Kumar *et al.* 2013). Kumar's study was on *Phragmites karka*, which could be classified as a separate common reed species growing in regions of Africa and Asia. The reported pulp parameters include: screened pulp yield (*Y*ie, %) and Kappa number (Kap) as well as paper properties that represent the prepared handsheets' brightness (Bri, %), opacity (Opa, %), apparent density (Den, g/cc), tensile strength index (Ten Ind, N·m/g), tear index (Tea Ind, mN·m^2/g), and burst index (Bur Ind, kN/g). A definitive reason for a 22% higher screen yield and 80% lower Kappa in Kumar's study compared to S11 soda pulp was not clear at this point. There were numerous variables, several of which were discussed in Part I of this investigation (Burry et *al.* 2014). The purpose of this investigation was to demonstrate the potential for using common reed as a biorefinery feedstock. No attempt was made to optimize the process variables.

Other pulped grasses are shown in Table 3, which included giant reed (G. Reed, *Arundo donax*), sugar cane (S. Cane, *Saccharum officinarum*, bagasse only), switchgrass (Switch, *Panicum virgatum*), and Napier or elephant grass (Napier, *Pennisetum purpureum*) and continued the comparative grass study begun in Part I (Burry *et al*. 2014).

Table 3. Unbleached and Bleached Pulp Properties of Various Grasses

Grass	Proc.	Yield (%)*	Kap.	Bri. (%)	Opa. (%)	Den. (g/cc)	Ten. Ind.	Tear Ind.	Burst Ind.	Reference
C. Reed	Soda	37.4	33.2	27.0	98.3	0.56	-	-	-	S11, Current Inv.
	Soda	30.2	42.1	21.4	99.9	0.48	-	-	-	S12, Current Inv.
	Soda‡	45.7	18.5	-	-	-	40.2	4.2	2.6	Kumar et *al.* 2013**
	ECF	31.2	4.0	58.2	91.0	0.68	-	-	-	S11, Current Inv.
	ECF	25.0	4.7	62.2	94.0	0.54	-	-	-	S12, Current Inv.
G. Reed	Kraft	40.8	19.8	35.3	-	-	-	7.3	4.0	Byrd 2000
	Kraft	42.1	26.0	22.8	-	0.49	17.4	10.5	0.5	Shatalov and Pereira 2006
	Kraft	36.7	22.0	23.3	-	-	31.0	8.9	1.8	Williams and Biswas 2010
	TCFǂ	37.1†	3.0	85.1	72.8	0.67		10.2	3.9	Byrd 2000
	TCF	38.8	12.8 ♦	79.6	-	-	12.4	8.8	1.1	Shatalov and Pereira 2006
	ECF	-	-	86.5	-	-	25.0	8.0	1.6	Williams and Biswas 2010
S. Cane$^{\Delta}$	Soda	53.2	13.9	33.6	-	0.58	38.2	6.3	2.2	Khristova *et al.* 2006
	TCF‡	-	3.9	71.6	-	0.78	67.9	6.0	4.3	Khristova *et al.* 2006
Switch	Kraft	46.8	15.5	27.6	98.2	0.46	76.0	5.6	4.1	Madakadze *et al.* 2010
	Kraft	48.5	13.5	29.5	-	0.50	-	5.5	3.1	Goel *et al.* 1998
	ECF	-	-	88.0	-	0.63	-	5.0	3.8	Goel *et al.* 1998
	TCF	-	-	87.2	-	0.67	-	4.7	3.6	Goel *et al.* 1998
Napier	Kraft	49.4	9.2	26.2	96.8	0.68	93.3	4.4	5.9	Madakadze *et al.* 2010
	Kraft	55.4	36.3	-	-	-	37.5	6.6	-	Thykesson *et al.* 1998

*Screened pulp yields and cumulative bleached yields are based on o.d. grass feed; pulps beaten to SR 30‡; PFI 200ǂ; **Study on *Phragmites karka*; $^{\Delta}$Sugar cane bagasse as feed to pulping process; ♦Kappa calculated from reported total lignin on o.d. pulp as 1.66% / 0.13 Kappa factor (see T 236); †Two stage TCF bleaching

Of the pulped grasses (unbleached), giant reed has similar pulp yields to S11, whereas the switch and Napier grasses exhibited 10% to 15% higher pulp yield. The high yield of sugar cane bagasse at 53.2% was expected because the pulped bagasse had already been subjected to HWE. Most of the cane stalk mass was removed during sugar processing with typical yields of 70% soluble bioproducts and 30% bagasse solids. Thus, the overall (cumulative) yield for soda pulped bagasse was 16.0% compared to S12 with 87.2% HWE solids yield and a reported overall yield at 30.2% in Table 3.

Thykesson *et al*. (1998) obtained the highest pulping process screened yield (for Napier grass) with corresponding high Kappa numbers similar to those in the current investigation. Madakadze *et al.* (2010) reported (for Napier grass) a much lower Kappa apparently at the expense of an approximately 5% yield. Neither reed grass responded very well to pulping relative to other grasses shown in Table 3. This initial investigation of common reed provided low yields with high Kappa numbers, which left much room for improvement in future studies.

Pulped grass brightness ranged from 21.4% to 35.3%. The brightness for the other studies on giant reed shown in Table 3 were very similar to S12's results. The S11 brightness was similar to other pulped grasses. All pulped grass opacity values were greater than 95%. The pulped grass handsheet apparent density ranged from 0.46 g/cc to 0.68 g/cc. No handsheet strength testing was performed in the current investigation, but results from other authors were included for comparison to machine-made grass paper discussed later.

Bleaching Comparative Data

Table 1 shows the conditions for three stages of elemental chlorine free (ECF) bleaching on both lots of reed soda pulp. All soda cooked batches of reed pulp were processed through the first stage of alkaline peroxide bleaching. This bag bleaching stage resulted in a heterogeneous product. Other than an initial thorough massage of each bag's content, mixing was minimal. There were obvious portions of well-bleached material and portions of material that appeared untouched by peroxide. The bulk of both S11 and S12 peroxide bleached pulps was used for producing pilot machine paper. The remainder of each pulp was used for subsequent bleaching stages.

The results of multiple bleaching stages on each grass (except Napier) are shown in Table 3. Bleaching has even more variables than pulping because a bleaching sequence typically includes three or more stages each with varied parameters. The most notable differences in the investigation were whether the sequence was totally chlorine free (TCF) or elemental chlorine free (ECF).

Several pulp and handsheet properties are shown in Figs. 1 through 5 representing each process stage; soda cook (S), peroxide bleached (P), chlorine dioxide bleached (D), and alkaline extraction (E). Figure 1 represents the cumulative or overall fiber yield beginning with soda cooked accepts (S) and continuing through the ECF bleaching sequence for both lots of *Phragmites* described in Table 1. Note that S11 yields were consistently approximately 6% higher for each stage. While the S12 soda Kappa number began substantially higher (Fig. 2), both pulps obtained similar Kappa numbers in the final bleaching stage.

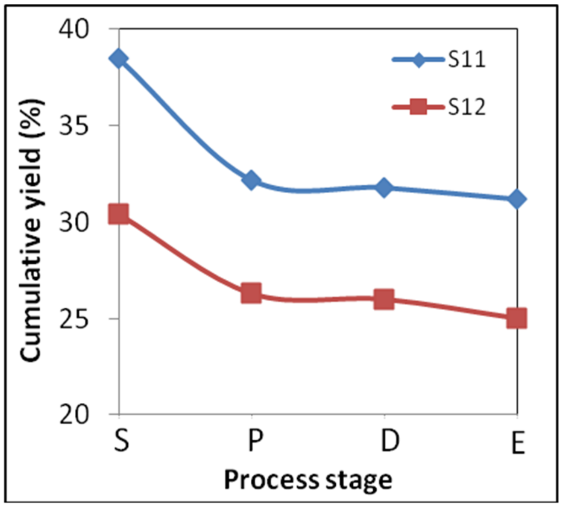

Fig. 1. *Phragmites* cumulative yield

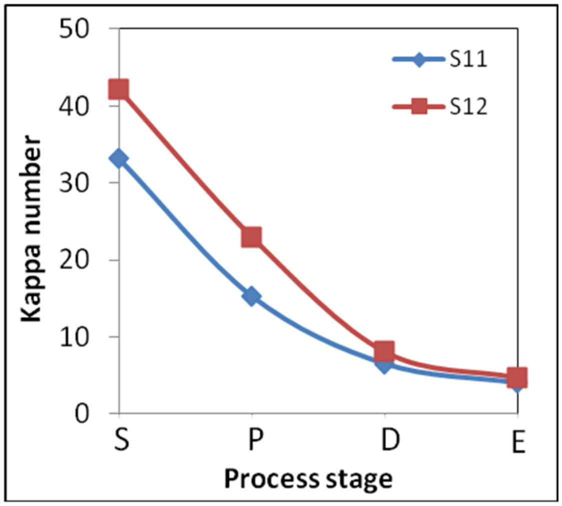

Fig. 2. *Phragmites* Kappa number (T 236)

Figure 3 indicates relative fiber strength as zero-span breaking strength for the various pulps. The S11 shows a more typical trend in which the breaking strength generally decreased as some cellulose degraded during subsequent bleaching stages. The S12 soda pulp z-span value appeared to be substantially low and may have been considered an anomaly. One might have expected S12 soda pulp breaking strength to be noticeably higher than S11 based on pulp viscosity data (Burry *et al*. 2014), where S12 viscosity was 18% higher than S11.

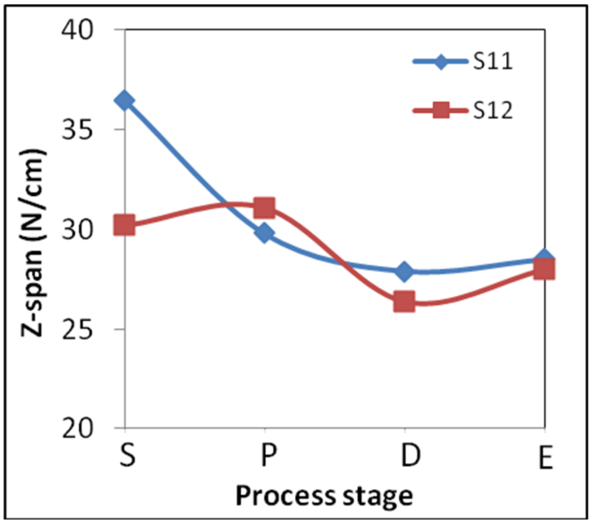

Fig. 3. *Phragmites* Zero-span breaking strength, T 231

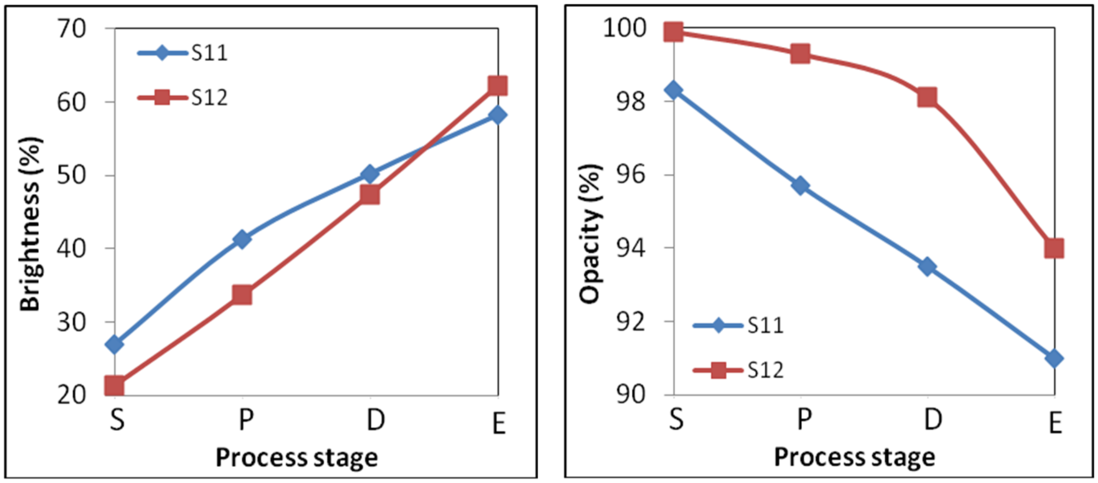

Fig. 4. *Phragmites* brightness, ISO 2469

Fig. 5. *Phragmites* opacity, ISO 2471

The brightness and opacity optical properties were measured representing the four process stages with results shown in Figs. 4 and 5, respectively. The S12 brightness was generally lower than S11. Investigations comparing with and without various pulping pre-treatments (VPP) on woody biomass indicated a VPP of 4% to 10% advantage (increase) in brightness (Helmerius *et al*. 2010; Kautto *et al*. 2010; Martin-Sampedro *et al*. 2014) to a 13% decrease as a consequence of VPP (Liu *et al*. 2012).

Handsheet opacity was consistently higher for the S12 lot presumably because of less interfiber bonding with the loss of additional hemicellulose (Yoon *et al*. 2008; Helmerius *et al*. 2010; Kautto *et al*. 2010).

Machine Made Paper Properties Comparative Data

Some fundamental properties of machine-made papers are shown in Table 4. All paper properties were compared to Boise Aspen 100 copy-grade (Boise Paper Holdings, LLC) made with 100% recovered post-consumer fiber. Pilot machine made paper target grammage of 75 g/m^2 (± 5%) was exceeded in two furnishes (S12 with 0% *Phragmites* and S12 with 100% *Phragmites*) and fell below target for the S12 with 60% *Phragmites* furnish. Fortunately, many strength properties compensated for variation in grammage by reporting an index value (strength property divided by grammage). The thickness fluctuated above target (0.1 mm) for all furnishes; especially for the S12 papers. Because none of the pilot machine papers were calendered, exceeding thickness target was not surprising. However, as with variation in grammage, data comparison became more difficult especially when reporting paper stiffness. The measured ash content indicated that calcium carbonate retention was closer to 60% (on average) rather than expectation at 90% (target filler loading of 20%). Filler retention was extremely low for S12 with 100% *Phragmites*.

Table 4. Fundamental Machine Made Paper Properties

	Grammage (g/m^2)	Thickness (mm)	Ash content (%)
Aspen 100	78.0	0.108	19.3
S11 with 30% *Phragmites*	78.4	0.122	10.6
S11 with 60% *Phragmites*	73.5	0.125	11.8
S11 with 100% *Phragmites*	78.2	0.125	14.4
S12 with 0% *Phragmites*	80.4	0.138	10.0
S12 with 60% *Phragmites*	67.3	0.116	14.3
S12 with 100% *Phragmites*	81.0	0.146	7.7

Apparent density, calculated from data in Table 3, was plotted as a function of the percent replacement of hardwood with *Phragmites* and is shown in Fig. 6. Clearly, the commercial calendered copy paper was more consolidated than uncalendered pilot machine papers.

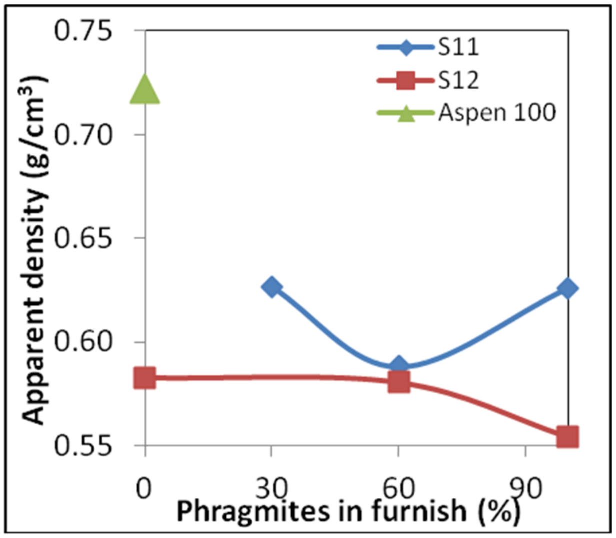

Fig. 6. Apparent density calculated from fundamental data in Table 3

Comparative optical brightness data is presented in Fig. 7. The S12 with zero percent *Phragmites* made with commercially bleached kraft market pulp was ten points lower in brightness than Aspen 100. No optical brighteners were added to the pilot machine papers. The addition of processed reed fibers substantially decreased brightness; as expected because only one moderately successful bleaching stage was performed. The S12 papers with *Phragmites* were approximately ten points lower in brightness than S11 papers.

The S11 and S12 papers with 100% *Phragmites* exhibited almost identical brightness to their counterpart handsheets after peroxide bleaching shown in Fig. 4. One would have expected the machine papers to be brighter with added filler. Measured opacity is shown in Fig. 8. Increased reed replacement fiber in the furnish continued to increase sheet opacity. See also discussions relative to Figs. 4 and 5.

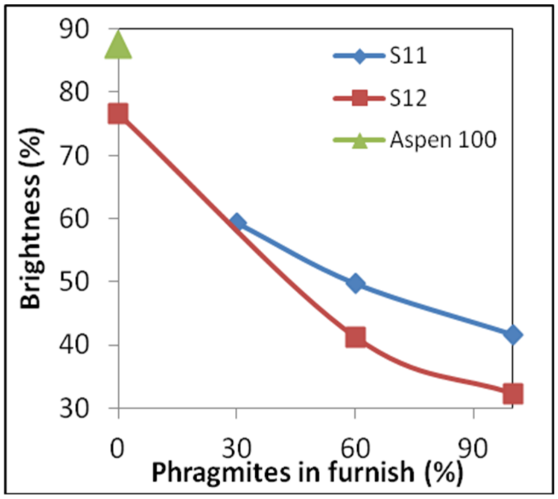

Fig. 7. Brightness, ISO 2469

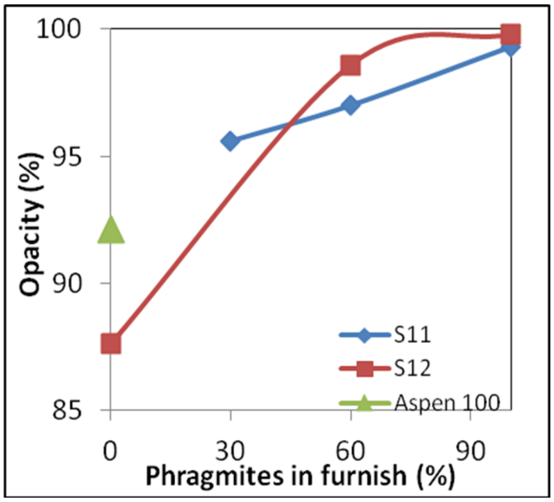

Fig. 8. Opacity, ISO 2471

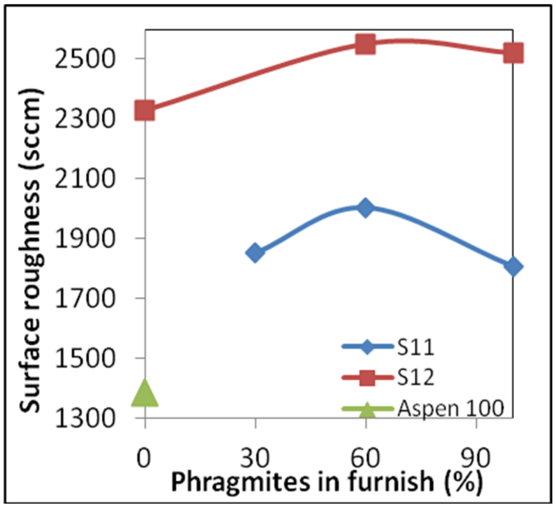

Fig. 9. Surface roughness, T 538

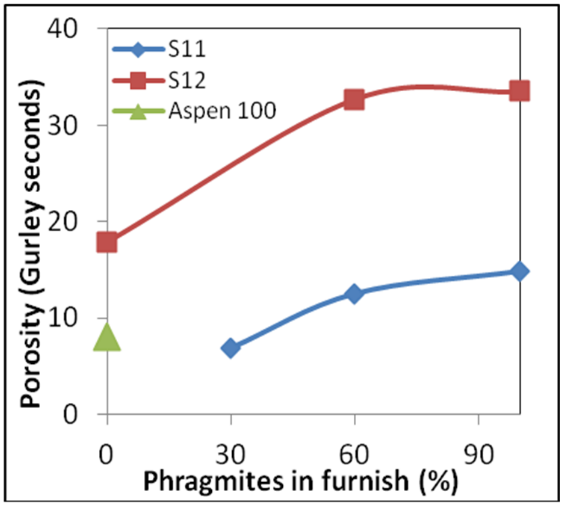

Fig. 10. Porosity, T 460

Surface roughness and sheet porosity were measured for each paper as shown in Figs. 9 and 10, respectively. Calendered commercial copy was considerably smoother than all uncalendered pilot papers while S11 papers were noticebly smoother and less porous than S12 papers. Similar reasoning discussed for apparent density (Fig. 6) may have been applied to sheet porosity where the removal of hemicelluloses resulted in less interfiber bonding and thus higher porosity.

The bending moment results are shown for pilot machine papers in both machine direction and cross direction in Figs. 11 and 12, respectively. Both the filler content and sheet thickness play a major role in the bending moment of paper and, as such, make comparisons difficult. The S11 papers had less variability in both filler content and thickness and therefore this stiffness data appeared less random than the S12 data.

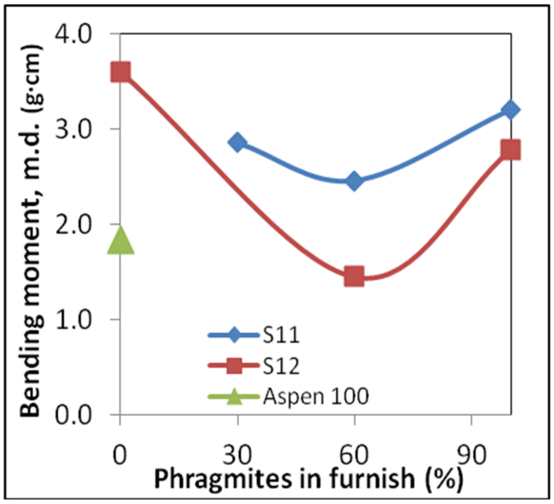

Fig. 11. M.d. Taber stiffness, T 566

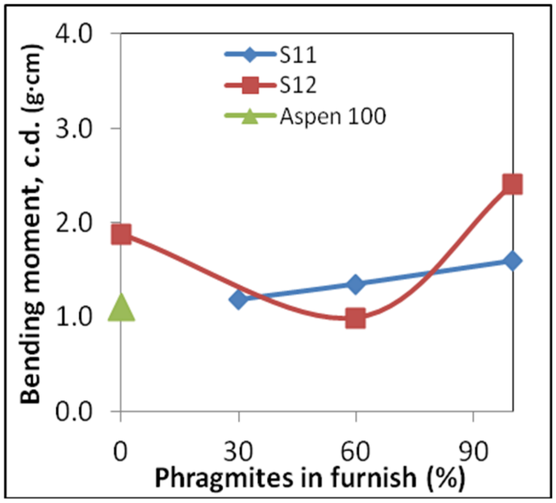

Fig. 12. C.d. Taber stiffness, T 566

The next five figures represent various paper mechanical strength properties as a function of the percent of *Phragmites* in the furnish. All strength data were expressed as an index to compensate for variation in grammage. Tensile index in the machine direction (m.d.) and cross direction (c.d.) are shown in Figs. 13 and 14, respectively.

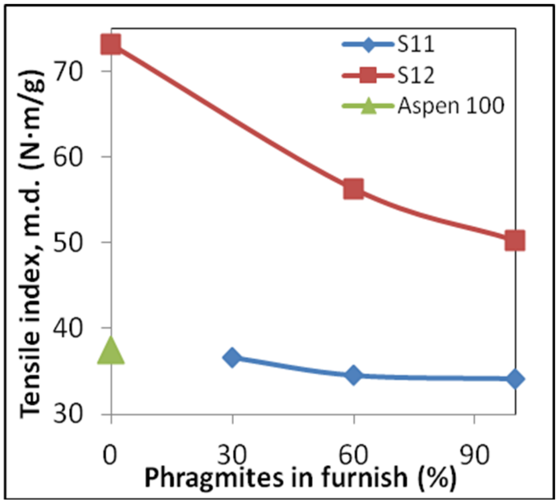

Fig. 13. M.d. tensile strength, T 494

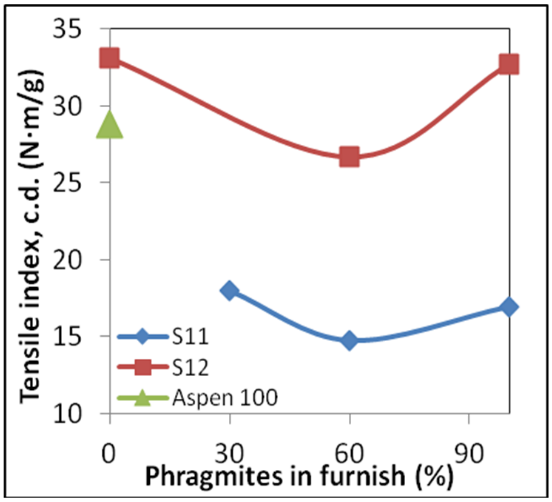

Fig. 14. C.d. tensile strength, T 494

The machine direction tensile strength for S11 was comparable to commercial Aspen 100 copy paper in all of the furnishes investigated. However, S11-made paper was substantially lower in tensile in the cross machine direction than Aspen 100 for reasons that were unclear. Most S12 machine made papers investigated exceeded Aspen 100 and far exceeded the S11 papers in tensile strength. The HWE apparently provided an increased tensile strength. The m.d. tensile index increased 47% with HWE (Fig. 13 for 100% *Phragmites*) while c.d. tensile index increased over 100% with HWE (Fig. 14 for 100% *Phragmites*). These results were not consistent with the concept that the loss of hemicellulose in HWE pulps resulted in less interfiber bonding, causing strength properties to generally decline, except for tear strength (Yoon *et al*. 2008; Helmerius *et al*. 2010; Kautto *et al*. 2010).

The comparison of various VPP treatments' effect on paper (handsheets) mechanical strength properties from research on woody biomass indicated mixed results. Jun *et al*. (2012) performed an alkali extraction on aspen (*Populus tremuloides*) prior to a large test matrix of kraft pulping and reported increased tensile index over the whole matrix. Liu *et al*. (2012) investigated sulfuric acid extraction of hemicelluloses prior to aspen chemi-thermomechanical pulping (CTMP) and reported an increased tensile index by as much as 93%. Kautto *et al*. (2010) performed HWE on softwood, primarily Scots pine (*Pinus sylvestris*), followed by kraft pulping. Bleached and refined pulps calculated to constant handsheet density indicated HWE decreased tensile index by as much as 13%. Granted, these are very different processes. Other researchers reported a decrease in tensile strength when HWE was incorporated prior to kraft pulping. Martin-Sampedro *et al*. (2014) showed a 24% decrease using steam rather than hot water in pretreating eucalyptus. Hasan *et al*. (2010) reported a 55% decrease for sugar maple (*Acer saccharum*). An investigation into HWE followed by kraft pulping using loblolly pine (*Pinus taeda*) resulted in a 24% decrease in tensile strength index (Yoon *et al*. 2008).

Tear index results are shown in Figs. 15 and 16 for m.d. and c.d., respectively. A more complete test matrix would be needed to verify the apparent optimal S11 tear strength with 60% *Phragmites* (equivalent to complete replacement of HW (hot water) in the furnish but with still 40% SW).

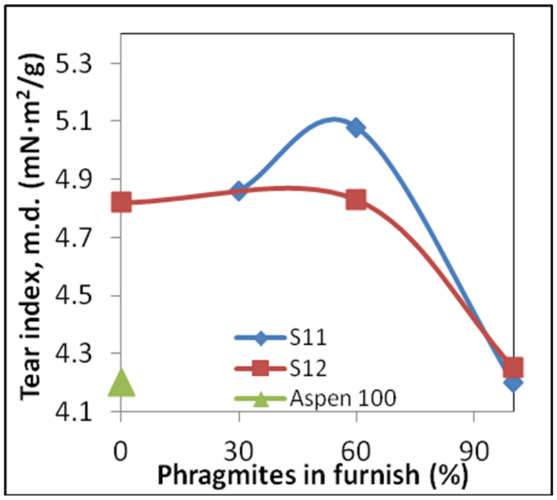

Fig. 15. M.d. tear strength, T 414

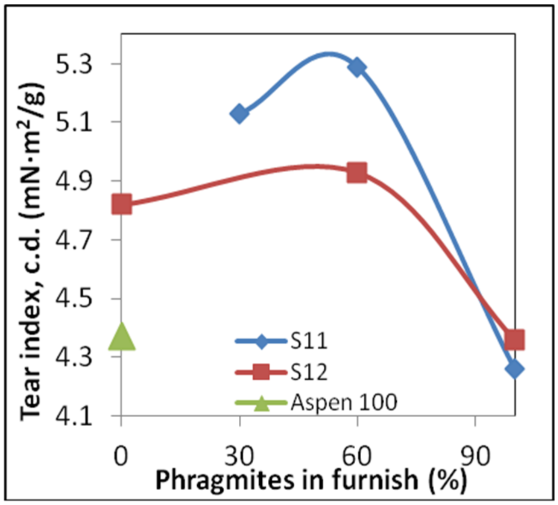

Fig. 16. C.d. tear strength, T 414

Both the S11 and S12 with 100% *Phragmites* tear strength was similar to Aspen 100. Otherwise, at lower levels of *Phragmites* in the furnish, HWE appeared to have negatively affected the tear index. Mixed results were also reported for the impact of various VPP treatments on tear strength. Several researchers reported an increase in tear index between 30% and 50% for handsheets after VPP (Yoon *et al*. 2008; Kautto *et al*. 2010; Jun *et al*. 2012; Liu *et al*. 2012). Helmerius *et al*. (2010) observed essentially no change in other strength properties but also reported a similar increase in tear strength after HWE prior to kraft pulping silver birch (*Betula pendula*). Martin-Sampedro *et al*. (2014) and Husan *et al*. (2010) both indicated decreased tear strength by 31% and 68%, respectively.

The burst strength was not of particular interest for copy grade but was performed on these machine made papers as shown in Fig. 17. Burst strength showed very similar trends as m.d. tensile (Fig. 13) where all of the S11 papers were comparable to commercial Aspen 100 copy and VPP treatment resulted in dramatic increased strength. Helmerius *et al*. (2010) reported essentially no change in burst index comparing VPP handsheets *versus* kraft only handsheets, while Jun *et al*. (2012) reported an average 15% increase in burst index with VPP.

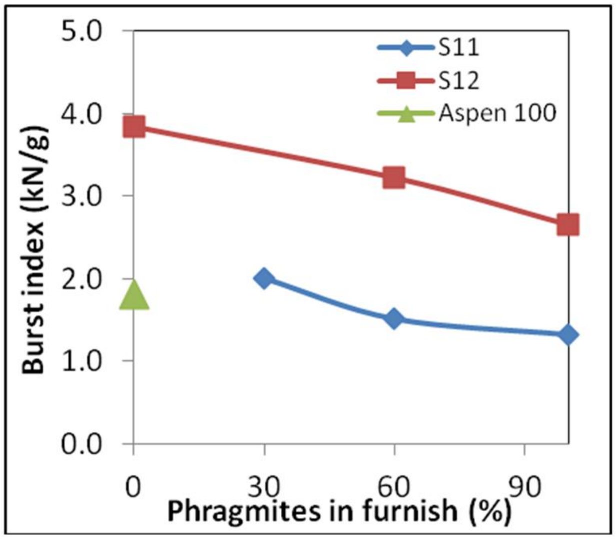

Fig. 17. Burst strength, T 403

Duarte (2010) performed HWE followed by kraft pulping on both eucalyptus (*Eucalyptus globulus*) and sugar maple. All of the handsheet mechanical strength properties decreased between 29% and 57% when HWE was included. All mechanical strength data on machine papers indicated that replacing hardwood in the control copy-grade furnish with *Phragmites* decreased strength with the exception of tear up to 60% replacement. Recall that S11 furnish (without HWE) contained 1% cationic starch while S12 (with HWE) contained 3%. The difference in starch loading was most likely not the reason for S12 increased tensile and burst strength, because a 3% addition was twice the recommended upper limit. Overloading the starch does not greatly improve strength properties.

Apparent filler loading based on ash content (Table 4) suggested there was unusually low retention, especially for S12 with 100% *Phragmites* at 7.7% ash content.

The S11 with 100% *Phragmites* furnish (at 14.4% ash content) contained almost twice the amount of filler than S12 with 100% *Phragmites* even though all furnishes were prepared with 20% filler (consistent with commercial copy paper). The effect of filler loading on S12 tensile strength (Figs. 13 and 14) was estimated based on the general rule-of-thumb where 10% filler will decrease tensile strength by 20% to 25%. The tensile index for 100% *Phragmites* S12 paper would decrease 15% (assuming 14.4% ash content equal to S11) from 50 N·m/g to 43 N·m/g in the machine direction (Fig. 13) and from 33 N·m/g to 28 N·m/g in the cross machine direction (Fig. 14). These estimated values were still 25% and 63% higher than S11's tensile index m.d. and c.d., respectively, and similar to values shown in Table 3 for both reed grasses. Tear strength values for 100% *Phragmites* machine made papers were similar to Kumar *et al*. (2013) results in Table 3. The S12 with 100% *Phragmites* machine made paper burst strength was similar to Kumar *et al*. (2013) while analogous S11 paper was remarkably lower than several other processed grasses.

Based on this initial investigation, *P. australis* should be considered a potentially valuable renewable and sustainable lignocellulosic biorefinery feedstock for platform biochemicals and industrial fiber. Annual harvesting this feedstock can be regarded as weed control (Sathitsuksanoh *et al*. 2009). However, one should note that for common reed to be sustainable, harvest time must be at the end of the growing season when nutrients are returned to the rhizomes and stored for winter (Köbbing *et al*. 2013). Dry culms (stem portion) remain standing well into winter months suggesting a winter harvest option (Sathitsuksanoh *et al*. 2009; Köbbing *et al*. 2013).

CONCLUSIONS

1. The tensile and burst strength properties seemed to have dramatically benefited from hot water extraction (HWE) in pilot machine papers.
2. Pilot machine papers with HWE tended to be less dense (higher porosity) and less smooth than papers without HWE.
3. A comparison of various grass family pulp yields and Kappa numbers revealed pulp yields in this investigation were generally low while Kappa numbers were generally high.
4. Bleached common reed pulps exhibited low yield but with low Kappa number (in the 4 to 5 range).
5. Pulp brightening was modest for HWE pulp (S12).
6. Bleached common reed pulps exhibited similar zero-span breaking strengths for with and without HWE.
7. There appear to be advantages in HWE prior to papermaking with *P. australis*. Thus, using *P. australis* as feedstock for a biorefinery has significant potential.

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