Quality and Variability of Commercial-Scale Short Rotation Willow Biomass Harvested Using a Single-Pass Cut-and-Chip Forage Harvester

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Abstract To date, most of the data on the characteristics of many short rotation woody crops has come from biomass that was hand harvested from small-scale yield trials. Concerns have been raised that there is insufficient information regarding the variability in biomass characteristics when material is harvested at commercial scales, which can impact the efficiency of biorefineries and other end users. The objectives of this study are to (1) characterize the biomass (i.e., ash, moisture, energy and elemental content, and particle size distribution) generated from commercial-scale shrub willow harvests at two sites, (2) evaluate compliance the published International Organization for Standardization (ISO) standards, and (3) contrast with "pristine" biomass from yield trials. Commercially generated chips were generally compliant with ISO standards for B1 chips. The mean ash content was 2.1 % (SD 0.59) dry basis and ranged from 0.8 to 3.5 % for samples collected from 224 truckloads of chips. There was a site effect for ash: 100 % compliance at one site and 82 % compliance at the second; loads exceeded the 3 % standard by less than 0.5 percentage points. The ash content of the Fish Creek cultivar was almost 1 % less than other cultivars and it is significantly lower ($P<0.0001$). The mean moisture content was 44 % (SD 2.2) and ranged from 37 to 51 %. The harvested biomass was similar to pristine biomass with the exception of ash content, and the variability was similar across all characteristics measured. The low variability of willow biomass characteristics suggests that material with a consistent set of characteristics can be generated from willow crops with a cut-and-chip harvesting system.

Keywords Short rotation woody crops · Shrub willow · Feedstock quality · Feedstock variability · Ash content · Moisture content

Introduction

Biomass for bioenergy and/or bioproducts can be sourced from forests, agricultural crops, various residue streams, and dedicated woody or herbaceous bioenergy crops [1, 2]. Despite this wide spectrum of promising feedstocks, none can singularly meet the projected demand or are clearly superior to alternatives with regards to costs, quality, or acceptance [3–5]. The variety of supply and demand results in a complex array of choices that will affect the economics and development of emerging biomass supply systems as they are scaled up. Woody biomass has the potential to be an important source of biomass in the northeastern USA where forests occupy 67 % of the land area [6], agricultural production has been in a 20-year decline, and crop residues are limited because of the dominance of dairy in the agricultural sector, which results in the majority of corn crops being harvested for silage. An assessment of biomass supply in New York State indicated that about 50 % of the potential sustainable biomass could come from forests and another 20–25 % from short rotation woody crops (SRWC) like shrub willow [7–9].

When multiple sources of woody biomass are used or blended together, feedstock can be available year-round so end users are not dependent on a single source of material. Blending and mixing promote a consistent feedstock supply, reduce the risk of dramatic price fluctuations, and eliminate the need for complicated and expensive long-term storage of the material [10]. Shrub willow biomass crops may be grown

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on a range of agricultural land using a coppice management system that allows multiple harvests, usually every 3 or 4 years, from a single planting of improved shrub willow cultivars [11]. Despite the benefits associated with shrub willow biomass crops systems, their expansion and deployment have been constrained by higher production costs and lower market acceptance associated with perceptions of chip quality and wood characteristics [5, 12–14].

Several key attributes of SRWC and other biomass feedstocks (i.e., ash content, moisture content, particle size distribution, and energy content) are a persistent concern because of their effect on costs, process efficiency, or materials handling [15–19]. Although the properties of many biomass feedstocks are typically quite variable, they are generally only reported as a mean or range of means [4, 20, 21]. For example, the reported mean ash content for a variety of bioenergy crops ranges from below 0.5 to just over 6 % [1]. The ash content of willow specifically is reported as 1.3–2.7 [9, 14]. While the mean values might be sufficiently low for modern conversion technology, the ash content for individual samples from large-scale harvests in some biomass feedstocks can spike to 15, 20, or more than 40 % in some cases [4]. Conversion systems are optimized for a limited range of variability in feedstock characteristics and do not respond well to large changes in feedstock characteristics, variability, and inconsistency which can cause serious problems for handling systems and refining or conversion processes [17, 22].

Although there is room for improved flexibility of conversion and handling technologies [17], the current level of variability in large-scale biomass harvests over time and growing seasons has been identified as an important barrier to the large-scale commercialization [4]. Various strategies have been proposed to decrease the variability and improve the properties of biomass feedstocks such as a uniform format feedstock supply system [4, 10]. For example, blending feedstocks is an emerging approach that could be effective in attenuating low-quality biomass feedstocks to make them more acceptable, and formulated feedstocks may even improve desirable properties such as energy content [23]. Understanding the variability associated with feedstocks, and the sources of variability are one key to expanding the production, use, and acceptance a wider range of biomass feedstocks.

Numerous entities have developed standards for wood chips, the most prevalent being various European-led standards (e.g., CEN/TC 335—Europe; ONORM M17135—Austria; SS 187120 and 187121—Sweden) that have recently formed the basis for the development of international standards [24]; the International Organization for Standardization (ISO) is a worldwide federation of national standards bodies of 28 participating countries. ISO has published standards for solid biofuels (ISO 17725) that includes specifications for pellets, briquettes, wood chips, and firewood [24]. Wood chips (ISO 17725-4) are classified into four different grades (A1, A2, B1, and B2) depending on where they are sourced and some key characteristics like moisture and ash. The standard specifically states that wood chips from short rotation coppice would be in the B1 category based on the origin and source criteria. The standard requires that the maximum wood chip moisture content be reported and that ash content is less than 3 % for B1 material. There are also threshold values for a number of elements (i.e., N, S, Cl, As, Cd, Cr, Cu, Pb, Hg, Ni, or Zn) that need to be met. Particle size distribution of the chips is divided into three categories based on whether ≥60 % of the mass of the chips is in 3.5–16 mm (P16S), 3.5–31.5 mm (P31S), or 3.5–45 mm (P45S) size class.

In a thorough discussion of the variability of biomass feedstocks, Kenney et al. [4] raise two intriguing issues regarding feedstock quality: (1) due to their inherent variability, biomass feedstocks must be characterized beyond a mean value in order for end users to design effective systems and processes for the material; (2) more information is needed to characterize materials obtained from "field-run" operations as compared to "pristine" material obtained from small-scale yield trials. The objectives of this study are to meet that call by characterizing the properties of freshly harvested willow chips, obtained from over 50-ha of harvests conducted in the winter of 2012–2013 in central New York State. The second objective was to compare the fresh willow biomass chips produced to the current ISO standards for grade B1 wood chips and, third, to contrast them with pristine chips obtained from hand-harvested yield trials. Properties evaluated include ash content, moisture content, energy content, selected elements (N, P, K, Ca, Cu, Mg, Na, S, and Zn), and particle size distribution.

Methods

Harvest Sites

Two sites were selected for mechanical harvest in late 2012 and early 2013: (1) two fields of coppiced, first-rotation, 4-year-old aboveground (5-year-old belowground), willow biomass crop totaling about 40-ha located near Auburn, NY (42° 55′ 22″ N, 76° 40′ 21″ W) with standing biomass ranging between 20 and 65 Mg_{wet} ha^{-1} and (2) two uncoppiced, first-rotation, 5-year-old willow biomass crops totaling 14 ha located near Groveland, NY (42° 42′ 09″ N, 77° 44′ 49″ W) with standing biomass ranging between 30 and 95 Mg_{wet} ha^{-1}. Both sites consisted of homogeneous plantings of multiple willow cultivars [25]. These cultivars represent a wide range of productivities, heights, diameters, stem forms (bowed to straight), and stem densities [26, 27]. All stems on two stools were measured at 30-cm height using calipers in 10 random

plots per cultivar prior to harvest for three selected cultivars (SV1, SX67, and Fish Creek).

Willow Biomass Crop Harvest

Harvest dates at Auburn and Groveland, NY followed leaf senescence and occurred between November 21, 2012 and February 4, 2013. Harvesting generally occurred on clear or overcast days; there was little or no rain on days that the harvesting occurred. It snowed on the final day of harvest (eight truck loads included in the subsequent analysis). The harvester platform used was a New Holland FR9080 harvester, equipped with a New Holland 130FB coppice header. The length of cut selected by the operator was the largest setting (33 mm) in order to maximize fuel economy and harvesting rate [26]. Additional site details of the harvesting operations are available in Eisenbies et al. [25].

The machinery was precisely tracked through the stands in a time-motion study so that the cultivar composition of each load was known; samples from individual loads of single cultivars were identified, and mixed loads of material consisted of chips from more than one cultivars. Samples (2–4 kg$_{wet}$) of fresh chips were collected from each truck load upon delivery to short-term storage. The sample was taken from within the chip mass of each truck load; Briggs et al. [28] showed that further stratification of the sample from a truck bed was not necessary. Each sample was weighed at the time of collection to the nearest 0.1 g at the edge of the field on a scale (Mettler-Toledo PG 5002-S) in order to obtain the wet weight before the samples could dry in the sun or become wetter due to their proximity to other samples. The samples were subsequently returned to the lab within 12 to 24 h and dried at 60 °C until they reached a constant weight. The moisture content was determined gravimetrically [29].

Lab Analysis

Based on sampling guidelines for wood chips [28, 30–32], nine 10 to 15 cm^3 subsamples (three scoops from three layers) were carefully taken from each of the 224 sample bags and combined, each combined sample representing one truck load. These were subsequently ground in a Wiley mill through a 40-mesh screen for subsequent lab analyses yielding approximately 50 ml of the sample. The ash content was analyzed for all 224 samples following the dry-ashing method by placing a crucible with 2-g samples in a cold muffle furnace and raising the temperature slowly up to 550 °C [33]. The net calorific content on a dry basis (higher heating value) was analyzed on 55 random samples (stratified across sites and cultivars) using Parr 6200 Oxygen Bomb Calorimeter (Moline, IL) [34]. The net calorific content as received (lower heating value) was calculated using moisture content and the formula described in Section D2 of Annex D of the ISO

standard [32]. A subset of 46 random samples (stratified across sites and cultivars) was sent to a commercial lab (Agricultural Analytical Services Lab at Pennsylvania State University) for chemical analysis. Ca, Cu, Fe, K, Mg, Mn, Na, P, S, and Zn were analyzed by dry-ashing, acid digestion using an Environmental Express AutoBlock II sample digester (Charleston, SC), followed by digestion solution analysis on Varian 730-ES inductively coupled plasma–optical emission spectrometry (ICP-OES, Santa Clara, CA) [35]. The total nitrogen was analyzed using the Elementar Vario Max CN Analyzer (Mount Laurel, NJ) following dry combustion [36].

Particle Size

The particle size of 122 samples ranging from 0.88 to 1.7 kg$_{dry}$ was determined for six size classes using a modified Williams classifier equipped with five sieve sizes with circular holes (6.35, 12.7, 19.1, 25.4, 31.8 mm) [37] and a correction factor that was developed for willow biomass crops. There are well-established methods for determining the particle size on standard wood chips in the paper industry [37, 38]. However, the cylindrical or semi-cylindrical chips generated from willow are different in shape than standard wood chips and many established methods are prone to error. The cylindrical shape of the willow chips results in a high occurrence of "spearing" (narrow chips passing through a screen based on their width as opposed to their length). Savoie et al. [38, 39] recommend that mechanical screening of this type of material be coupled with more precise calibration and correction methods. The issue was addressed mechanically by adding a 12-mm-thick plywood plates with hole patterns that matched each screen. The sieving time for each sample was 15 s. This modification reduced pass-through by increasing the angle pieces had reached in order to clear the screen; however, pass-through was still unacceptably high. A correction factor was subsequently developed using 20 samples of two of the most dissimilar cultivars: SX67 (wider), and Fish Creek (narrower). After sieving with the additional plywood plates installed, the chips were hand sorted on each screen to determine the mass percentage belonging to each of the larger screen sizes using a size gauge for each hole diameter used in the five screens. A correction factor is reported and is subsequently applied for each screen size based on these results.

Statistics

Each load of harvested willow biomass (4.5–5.4 Mg$_{dry}$; 10–12 Mg$_{wet}$) represents an area of 2,000 m^2 (mean for Groveland) to 3,000 m^2 (mean for Auburn) of willow biomass crops; thus, they were treated as experimental replicates. Loads were identified as either one cultivar or mixed as determined by the time-motion data [25]. The FREQ procedure (SAS 9.2) was used to evaluate the compliance rate for particle size,

moisture (M), ash, energy (Q), Cu, N, S, and Zn as described in the table "specification of graded wood chips" found in the ISO 17225-4 standards for graded wood chips [24]. A Wilson confidence interval [40] of the pass/fail binomial response was determined for freshly harvested loads compared against the ISO standard at the alpha=0.05 level. Differences between the physical and chemical properties of chips were evaluated as a completely randomized design with a two-way treatment structure (model, Y=site cultivar site * cultivar) in the GLM procedure (SAS 9.2); mean comparisons were made using Fisher's protected least significant difference [41]; Ca, K, Mg, Na, and P were included in addition to other characteristics in these analyses.

Results and Discussion

ISO Standard Compliance by Load

The chips harvested from willow biomass crops meet the source requirements for the ISO B1 standards for graded wood chips because they are considered a short rotation coppice crop [24]. Wood chips generated from the commercial-scaled harvests in Auburn and Groveland generally conformed to the standard with the exception of ash content (Table 1). Eighteen percent of 108 individual loads sourced from Auburn exceeded the 3 % ash standard by a mean of 0.23 % and a 0.49 % maximum. Two percent of the 116 loads from Groveland also failed the B1 ash standard, but this was not significantly different from 100 % compliance based on the 95 % confidence interval. One sample from Auburn also failed the B1 zinc standard (100 mg kg_{dry}^{-1}) by 20 mg kg_{dry}^{-1}, but the result was not significantly different from 100 % compliance across all the samples.

There was a significant relationship between cultivar and biomass quality. Over 80 % of the loads from Fish Creek were below the ISO B1 standard for ash content (≤3 %) at both sites and had a 2–3 % lower moisture content than other cultivars

(P=0.0003) (Table 2). The relationship between cultivar and ash content could be associated with the stem sizes. Smaller stems typically have a larger proportion of bark, and bark has a higher ash content [27, 42]. However, while Fish Creek stem diameters were among the largest at Auburn, they were only mid-sized at Groveland (Fig. 1). In contrast, the diameters of the SV1 cultivar were among the smallest at both sites, but SV1 ash content was the second highest at Auburn and second lowest at Groveland. Thus, while stem diameter distributions play a role in ash content, there also appears to be a disposition of some cultivars for lower ash content and this may also be influenced by site conditions. Differences in yields among these cultivars have been reported [26, 43] and have been a main driver in selecting plant material for commercial expansion. However, Kenney et al. [4] has suggested that a focus on biomass yield alone maybe overemphasized and that greater attention should be placed on quality in combination with yield.

Biomass Properties

For these harvests, there were significant site effects with every property tested with the exception of both the net calorific values on both dry basis and as received (Table 3). The weighted sums of squares suggest that the cultivar effects were more important than site when they were statistically significant, with the exception of nitrogen. In most cases, the coefficients of variation for any single characteristic were in the range of 5 to 30 %. Other than the deviations noted in Table 1, the properties were compliant with ISO standards.

To compare the characteristics of the wood chips from large-scale harvests with "pristine chips" from hand-harvested productivity trials, we used data from first- and second-rotation willow yield trials that were hand harvested in 2008 [26] and in 2011. These sites had similar cultivar groups as the Auburn and Groveland sites, but the hand-harvested sites were located on better drained soils and more attention was paid to stand establishment (weed control and fertilization). Although there is no satisfactory way to

Table 1 Compliance of loads of willow biomass chips with ISO 17225-4 standards for grade B1 wood chips for ash, moisture, and chemical characteristics

	Moisture content ISO standard	Calorific content	Ash content	Cu	N	S	Zn
	Maximum %	Minimum as received MJ kg^{-1}	3 %	10 mg kg_{dry}^{-1}	1 %	1,000 mg kg_{dry}^{-1}	100 mg kg_{dry}^{-1}
			Percent compliance (95 % confidence interval)				
Overall	51	9.8	91 (86–93)	100	100	100	98[a] (89–100)
Auburn	51	9.9	82 (74–88)	100	100	100	96 (80–99)
Groveland	48	9.8	98[a] (94–100)	100	100	100	100

[a] Not significantly different from 100 % compliance

Table 2 Ash content and compliance with ISO 17225-4 standard for B1 wood chips of willow biomass chips from different shrub willow diversity groups

Cultivar species grouping	Number	Mean (SD) Percent ash	ISO B1 ash content standard at 3.0 % Percent compliance (95 % CI)
Auburn			
Mixed loads	69	2.3 AB (0.67)	80 (69–88)
SX61, SX64, SX67 (*S. miyabeana*)	25	2.5 A (0.48)	80 (61–91)
SV1 (*S. x dasyclados*)	6	2.4 AB (0.26)	100
Fish Creek (*S. purpurea*)	8	1.4 C (0.19)	100
Groveland			
Mixed loads	68	2.1 B (0.52)	99 (92–100)
SX61, SX64, SX67 (*S. miyabeana*)	24	2.3 AB (0.38)	96 (80–99)
SV1 (*S. x dasyclados*)	18	1.5 C (0.19)	100
Fish Creek (*S. purpurea*)	6	1.3 C (0.24)	100

Letters indicate significant differences within columns at the alpha=0.05 level

combine these dissimilar experiments statistically, the material was similar with regards to moisture and energy content (Table 4). The ash content of the mechanically harvested biomass was about 0.4 percentage points greater than the hand-harvested material. The concentrations of Ca, Cu, K, Mg, P, S, and Zn were all higher in the mechanically harvested biomass but still below the ISO thresholds for these elements. The N concentration was similar in both sets of samples, and the Na concentration was lower in the mechanically harvested samples compared to the pristine samples. The coefficient of variation was similar between the mechanically and pristine harvested samples for ash, moisture, energy content, Ca, and P. The coefficient of variation (CV) was lower for mechanically harvested biomass for Cu, K, Mg, N, Na, S, and higher for Zn. Additionally, the main site effect was always

significant, but the importance of cultivar over site seen in these large-scale harvests did not occur for the pristine samples.

It is preferable for wet biomass be dried before combustion or sacrifice efficiency [1]. Moisture content and the expense of drying are often brought up as a limitation of willow and other woody crops which often have moisture contents anywhere from 35 to less than 60 % for individual samples. However, in the case of conversion technologies that require wet chips, moisture content is not an issue since adding water is part of the process. In some locations, wetter chips may even be more desirable so that additional water does not have to be acquired and added in the processing in the biorefinery. The mean moisture content of the biomass from the Auburn and Groveland harvests was 44 % and ranged between 37 and

Fig. 1 Proportional stem diameter distributions for three shrub willow cultivars at the end of the 2012 growing season prior to harvesting. The plants at Auburn were 4 years old on a 5-year-old root system, and the plants at Groveland were 5 years old on a 5-year-old root system
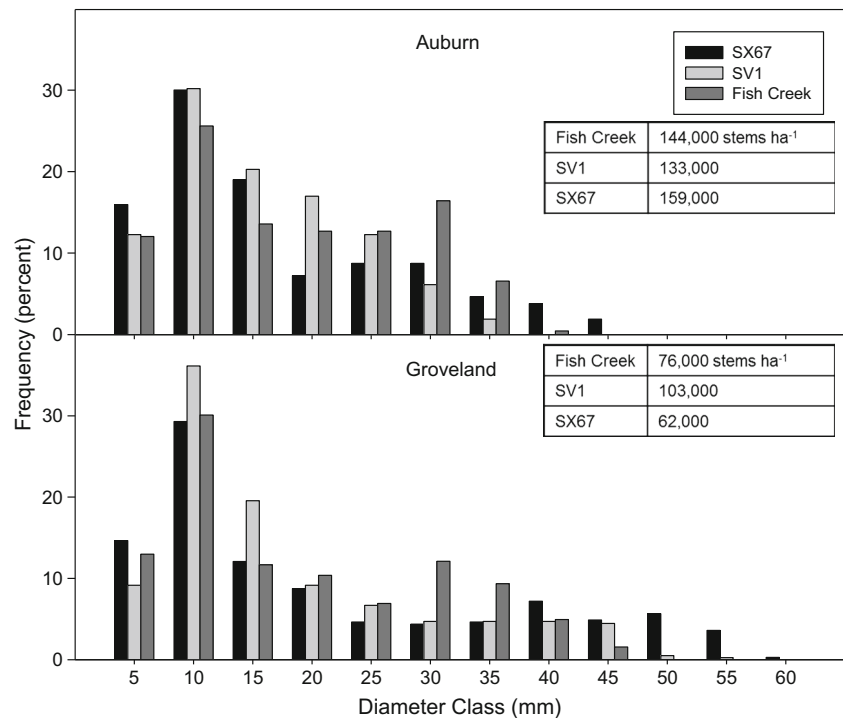
Table 3 Characteristics of willow biomass chips harvested at a commercial scale with single-pass cut-and-chip system

		Property												
	Ash	Moisture	Calorific		Ca	Cu	K	Mg	N	Na	P	S	Zn
			Gross	As received									
Units	%	%	MJ kg$_{dry}^{-1}$	MJ kg$_{dry}^{-1}$	mg kg$_{dry}^{-1}$	mg kg$_{dry}^{-1}$	mg kg$_{dry}^{-1}$	mg kg$_{dry}^{-1}$	%	mg kg$_{dry}^{-1}$	mg kg$_{dry}^{-1}$	mg kg$_{dry}^{-1}$	mg kg$_{dry}^{-1}$
Sample size (n)	224	205	55	54	46	46	46	46	46	46	46	46	46
Auburn mean (CV%)	2.3 (28)	44 (5)	18.6 (1)	10.4 (5)	6,828 (34)	6.8 (11)	1,919 (14)	473 (13)	0.4 (13)	9.5 (56)	828 (10)	208 (12)	77 (20)
Groveland mean (CV%)	2.0 (26)	45 (6)			5,485 (37)	5.7 (10)	1,454 (9)	350 (9)	0.33 (15)	6.4 (26)	612 (10)	177 (13)	55 (23)
Overall percentiles													
Maximum	3.5	51	19.1	12.1	13,000	8.6	2,300	600	0.49	24	940	280	120
90th	3.0	47	19.0	11.3	9,000	7.4	2,200	520	0.44	11	900	230	89
75th	2.6	46	18.8	10.5	7,400	7.0	2,000	470	0.40	8.5	850	210	84
50th	2.1	45	18.6	10.3	6,300	6.1	1,700	400	0.35	7.0	690	190	64
25th	1.6	44	18.4	10.1	4,700	5.5	1,400	340	0.31	5.0	620	170	52
10th	1.4	41	18.4	9.9	3,300	5.2	1,400	320	0.29	4.5	550	160	45
Minimum	0.8	37	18.3	9.8	2,500	5.0	1,300	300	0.26	4.0	520	130	34
Test of the model Y=site cultivar site*cultivar													
Global P value	<0.0001	<0.0001	0.0052	0.7948	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	0.0061	<0.0001	0.0023	<0.0001
R^2	29	15	34	1	55	58	60	63	55	39	75	42	61
Percentage of the sum of weighted squares (type III)[a]													
Site	17	27	ns	ns	11	70	94	98	65	27	93	70	67
Cultivar	73	59	92	ns	87	ns	ns	ns	32	51	ns	ns	ns

Sample size, mean, coefficient of variation (CV%), and overall percentiles are reported. R^2 indicates the degree to which the full model (Y site cultivar site × cultivar) explains the variability of the data. The percentage of the weighted squares indicates the relative importance of the two main effects describing the data in the full model; the interaction term was not significant for any of the properties

ns not significant

[a] Site*clone term was not significant for any property

Table 4 Characteristics of willow biomass chips mechanically harvested with single-pass cut-and-chip systems at a commercial scale and hand-harvested chips obtained from smaller yield trials

Property	Mechanically harvested biomass				Pristine harvested biomass			
	N	Mean	CV	Primary component[a]	N	Mean	CV	Primary component[a]
Ash (%)	224	2.1	28	Cultivar	97	1.7	28	Cultivar
Moisture (%)	205	44	5	Cultivar	97	46	6	Cultivar
Net calorific value dry (Q)	55	18.6	1	Cultivar	97	18.8	1	Site
Net calorific value as received (Q_{ar})	54	10.4	5	ns	97	10.1	5	ns
Ca	46	6,157	37	Cultivar	97	3,355	36	Cultivar
Cu	46	6.2	14	Site	97	2.9	27	Site
K	46	1,687	19	Site	97	753	23	Site
Mg	46	412	19	Site	97	139	29	Cultivar
N	46	0.36	17	Site	97	0.34	25	Site
Na	46	7.9	53	Cultivar	97	47	33	Site
P	46	720	18	Site	97	510	20	Cultivar
S	46	193	15	Site	97	130	22	Site
Zn	46	66	27	Site	97	35	22	ns

[a] As determined by the percentage of the sum of weighted squares (type III)

51 % (Table 3). The moisture content of willow is often reported as on the high end for woody biomass (55–60 %) [16]. Examples from the literature report that mean moisture contents for willow can be as high as 55 or 56 % [42, 44], but it is not uncommon to observe values in the vicinity of 45 % [26, 45], which was recorded for these harvests and the pristine biomass used for comparison in this study. Others have reported values between 45 and 55 % [9, 46]. The distributions of moisture content were similar for both sites, but the mean moisture content of the hand-harvested sites was 2 percentage points higher with a single outlier at 57 % while the distribution from the commercial harvest had a noticeable tail on the dryer end (Fig. 2). Overall, the coefficients of variation were low (5–6 %) for both mechanically harvested and pristine chips.

Although the statistical model for moisture was significant ($P<0.0001$), the amount of variability described was poor

($R^2=0.15$) and improved when finer particle sizes were included as covariates ($R^2\approx0.3$). The loss of fine particles during mechanical harvesting, particularly as the chips are blown from the harvester to the collection vehicle and then dumped into trucks, may be a factor that contributes to the slightly lower moisture content of mechanically harvested material. Given that the plants had senesced, the moisture content would only be sufficient to maintain the cell structure, and coupled with the low variability (CV% between 5 and 6 %), there may be little else to explain the observed differences outside of random variation. However, the variability that was described by the model was more a function of cultivar than site (Table 4). Fish Creek had moisture contents that were significantly 2–5 percentage points less than other cultivars.

The energy content of freshly harvested biomass is influenced by the moisture content of the material. On a dry basis, the net calorific value of the material was 18.6 MJ kg^{-1} for

Fig. 2 Distribution of moisture content of 205 samples of willow biomass chips that were mechanically harvested using a single-pass cut-and-chip harvester compared to 97 hand-harvested samples collected from yield trial plots
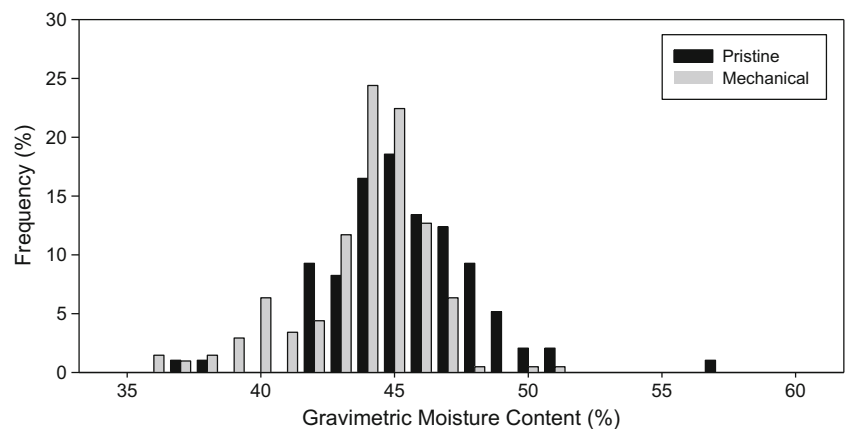
mechanically harvested material and 18.8 MJ kg^{-1} for pristine material. However, because the moisture content of the pristine material was slightly higher than the mechanically harvested biomass, the net calorific content as received of the pristine material was slightly lower (10.1 MJ kg^{-1}) than the mechanically harvested material (10.4 MJ kg^{-1}).

The ash content of wood is commonly lower and less variable than non-woody feedstocks, but willow biomass crops tend to have higher ash content than other hardwoods, especially clean or debarked chips, due to the higher bark/wood ratios [4, 20, 21]. However, willow chips commercially harvested using single-pass cut-and-chip machinery, such as the system utilized in this study, do not touch the ground during harvesting. The New Holland forage harvester specifically has on-board systems that automatically control header height above the soil surface to prevent the saw blades from cutting into the ground and picking up soil. Forestry residues are often dragged or skidded to a landing which increases chances for soil contamination.

The distribution of ash content of pristine samples had a lower mean, less variation, and a positive skew compared to the commercial-scaled harvest (Fig. 3). The ash content for willow in the commercial harvest ranged between 0.8 and 3.5 % with a mean of 2.3 for Auburn and 2.0 for Groveland (Table 3); coefficients of variation were approximately 26–28 %. The ash content range at the hand-harvested sites was 0.8 to 3.0 % and an overall mean of 1.7 %, which would make them 100 % compliant with the B1 ash standard. As with the mechanically harvested sites, the Fish Creek and SV1 cultivars had significantly lower ash content at the hand-harvested sites compared to the SX cultivars (SX61, SX64, and SX67) or the mixed loads. Although the coefficients of variation were similar between the mechanical and pristine harvests from Serapiglia et al. [26], Tharakan et al. [42] reported coefficients of variation of only 10 % for ash content separately in the wood and bark of willow. By peeling the bark from willow stems and analyzing wood and bark separately, some of the variation associated with how these materials mix in larger

samples and are collected when a subsample is taken was removed. In addition, Tharakan et al. [40] used samples from a 10-cm section of the willow stems at the centroid so variation in ash content associated with this smaller diameter portions of the plant, such as the branch tips, was eliminated in that study. Recent analysis suggests that the centroid underestimates the bark to wood ratio in willow (Volk, unpublished data), which may be another factor contributing to the differences in these studies.

As with moisture content, the statistical models poorly described variation in ash content ($R^2=0.29$), but the variation was also primarily captured by the cultivar main effect. As mentioned previously, differences in ash content between cultivars could be driven by stem size, but there was no overwhelming evidence that it was a driving factor in these harvests. Bark has 4–12 times the ash content of wood in willow, and depending on stem sizes, the bark content is approximately 10–25 % of the biomass [27, 42].

Since this study did not track the chain of custody of specific loads past short-term storage, the characteristics of these chips are analogous to those that would be delivered directly to an end user from the edge of the field. However, there are numerous ways that further handling and storage of harvested chips could result in contamination [32]. Possible sources of contamination in transport could be road debris that settles on uncovered loads, soil that is mixed with the chips if they are stored on the ground for short-term storage or soil that is incorporated with the chips when they are reloaded from short-term storage.

About 100 Mg_{wet} of chips from the Groveland harvests (cultivars SV1 and S365) were delivered to a research facility after 4 to 10 weeks of short-term storage on an unpaved loading deck as part of a study examining the changes in biomass characteristics during storage. The mean ash content of the samples of these delivered chips was 2.7 %, which was 0.9 % higher ($P=0.0081$) than the freshly harvested chips (Eisenbies, unpublished data). Only 77 % of the samples from the delivered chips met the B1 standard compared to a 100 %

Fig. 3 Distribution of ash content (dry basis) of 224 samples of willow biomass chips that were mechanically harvested with a single-pass cut-and-chip harvester compared to 97 hand-harvested samples collected from yield trial plots
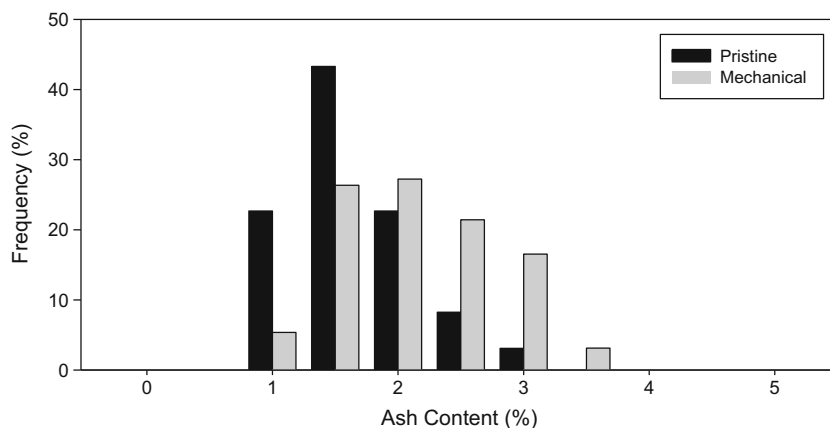
Fig. 4 Uncorrected and corrected particle size distributions of willow biomass chips from large-scale harvests using a single-pass cut-and-chip system. *Gray bars* (mean SD) indicate the percentage, or corrected percentage, of the total mass retained by the screen. *Lines* indicate the percentage of the total mass passing the through the overlying screens in the Williams classifier and caught by the tray
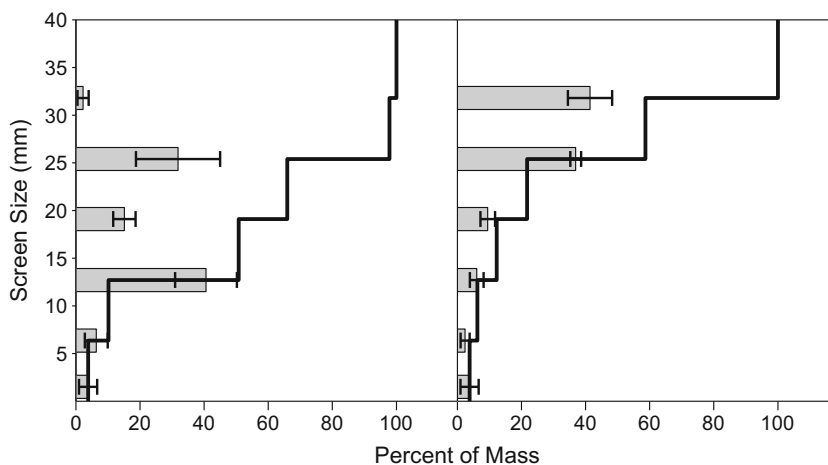
pass rate for the freshly harvested chips. The coefficient of variation for the ash of delivered and freshly harvested chips was similar at about 29 %. Dirt clods were observed in the delivered loads of willow biomass, suggesting that the contractors did not take enough care when reloading the material from the loading pad where the chips were stored. The mean moisture content of the delivered chips was also 5 percentage points higher than the freshly harvested chips ($P=0.0521$). However, the maximum moisture content only increased 2 percentage points compared to the freshly harvested chips. There was no difference in energy content between the delivered and freshly harvested chips ($P=0.9445$).

In the fall of 2013, other willow fields were harvested by a commercial operator in northern NY; the biomass was temporarily stored on the ground at the edge of the field and then delivered to a nearby wood-fired power plant. Samples of delivered willow chips were collected from 15 truck loads of willow. This material had a higher mean ash content (3.0 %) than freshly harvested chips but a similar maximum (4.5 %) and CV (22 %). The mean moisture content of these samples was similar to the freshly harvested chips (43 %) with a range of 35 to 55 % and a coefficient of variation of 12 %. Some foliage remained on these willow plants at the time of harvest and may have contributed to these differences.

In both of these situations, it appears that the temporary storage and additional handling of the material increased its ash content. Clearly, the handling of chips is an important factor in maintaining quality, particularly in consideration of their surface area, but the consistency of willow chips within the supply chain is still apparent and the quality could be maintained, or possibly improved [47–49], by defining and following the best practices for handling and storage. In order to understand the impact of temporary storage and reloading future on chip quality, future studies will need to establish a chain of custody protocol for the material and track loads of chips through the various stages.

Particle Size

This study used two simple methods to improve the estimates for the particle size distribution, as opposed to the automated methods reviewed by Savoie et al. [38, 50]. The modified screens used in this study reduced spearing, but a large percentage of mass in the 19.1- to 31.8-mm size classes was still shaken down on multiple screens due to spearing (Fig. 4, Table 5). Correction factors were calculated and used in this study to adjust the particle size distribution, but it is not clear how applicable these correction factors will be for other

Table 5 Mass correction factors (to the nearest half-percent) for retained chips on screens using modified Williams classifier

Screen size (mm)	Actual chip size (mm)					
	>31.8	25.4–31.8	19.1–25.4	12.7–19.1	6.35–12.7	<6.35
	Mass of retained sample (%)					
31.8	100.0					
25.4	63.0	37.0				
19.1	52.5	42.5	5.0			
12.7	27.0	45.0	19.0	9.0		
6.35	1.5	5.5	15.0	40.0	38.0	
Tray	0	0	0	0	0	100

studies. In addition, the correction factors were less precise for the material captured by the 6.35- and 12.7-mm screens. There were no significant differences in the correction factors for the Fish Creek and SX cultivars (SX61, SX64, and SX67) at the alpha=0.05 level with the exception of the chips in the 6.35–12.7 and 12.7–19.1 mm size classes ($P<0.04$). However, the overall proportion of mass in these size classes was only 7 %. Since the chips from the two cultivars tested represent visually different chips, a single correction factor for each size class was assigned uniformly to all samples in this study.

Qualitatively, the feedstock material produced during the Groveland and Auburn harvest operations were of an acceptable quality for end users including both wood-fired power plants and a biorefinery partner. The chips had a consistent size with virtually no stringers compared to harvesting machines tested previously. It is capable of producing chips of different sizes on the fly by adjusting the feedroll speed or changing cutter drums. Cutter knives are sharpened on board using an automated process that is operator initiated and takes 15 min. The settings to produce the largest chip size were chosen to maximize fuel economy, minimize fines, and allow for increased ground speeds. Cutter drums that produce smaller chip sizes are available, but potentially at the cost of reduced harvester throughput and an increase in the amount of fines.

In the published ISO standards for graded wood chips, a table titled "Particle size of graded chips" defines five criteria (main fraction, fine fraction, course fraction, max length, and max cross section) for three size grades of wood chips [24]. Chips produced during these harvesting trials were nearly 100 % compliant with the ISO P45S standard (Table 6). A small percentage of failures (4 %) was observed for the fines criterion (<10 %), but all samples had less than 15 % fines, which is the cutoff for the P16S class. The minimum screen size used in this trial was 6.35 mm, so it is likely that all samples would have been in compliance had it been possible to include a 3.15-mm screen when the particle size distribution analysis was conducted. Only 21 % of the loads met the ISO P31S standard, and all of these samples were exclusively from Groveland, and the majority of those loads were collected from the Fish Creek and SV1 cultivars. If the P31S was the standard that was required by end users, change to feed roll speeds or the configuration of the knives on the cutter drum would be required.

From the suite of samples analyzed, neither site nor cultivar factors influenced the distribution of chips sizes. There were significant differences of the percent fines (material <6.35 mm) among sites ($P<0.0001$) and cultivars ($P=0.0483$). Loads produced in freezing weather (average temperature below freezing for 10 h preceding the commencement of an individual load) had 4 % more chips over 25.4 mm ($P<0.0119$), but it is difficult to ascribe the cause to temperature or simply the characteristics of the cultivars harvested

that day. There were no significant relationships between standing biomass (Mg ha^{-1}; $P=0.4874$) or material capacity (Mg hr^{-1}; $P=0.1970$) and percentage of fines using a minimum 6.35-mm sieve. There was a positive correlation between the consecutive order of the loads across the weeks of harvesting and the percent fines (<6.35 mm; $P<0.0001$); thus, as the harvests went along, the fines increased. The Groveland site was harvested after the Auburn; blade maintenance might be a concern, but colder temperatures also produce more fines as the wood tends to shatter; however, in these particular harvests, winds were higher on the coldest days and there were a lot of airborne fines that did not adhere to larger chips and may not have been delivered into the collection vehicles.

Our development of correction factors improved our determination of particle size distributions, but the process to develop these standards in every project are potentially difficult and time consuming. Had the correction factor not been applied, over 90 % of the samples would have passed the main fraction, fines, and course fraction criteria ISO P31S, and half the samples from Groveland for ISO P16S as well; however, based on the fourth and fifth criteria that set the maximum length and cross-sectional area, most would have failed those two grades. The fourth and fifth criteria appear to serve as adequate safeguards (although not a substitute) against imperfect sieving methodology, assuming the supplier is making an honest effort to correctly grade the chips. If the objective is to meet a more restrictive size grade, such as P16S, cultivar-specific correction factors and more rigorous lab methods may be required when applying these standards to willow biomass harvested in the manner used in this trial.

Implications

Willow biomass has a low degree of variability compared to herbaceous feedstocks which may lend it to being used as one component of biomass supply made up of multiple feedstocks. Shrub willow and other SRWC are already a small component of the woody biomass sector; linking the supply chain for these SRWC with other forest based biomass would create opportunities for mixing or blending and help decrease logistics costs [10, 51, 52] and potentially further reduce variation in feedstock quality. Various pretreatments are also available to wood chips after they are harvested. Cleaning loads of ultra fine material (<0.2 mm) could lower ash content and improve the feedstock quality [53]. Processes such as hot water extraction can serve as a pretreatment and can lower ash content by about 50 %, increase energy density, and improve storability of the material [54]. The hot water-extracted chip byproduct has been shown to have advantages for pulping, wood pellets, and reconstituted wood products. The extraction solution contains easily fermentable sugars and other compounds that have established markets [55].

Table 6 Summary of compliance of willow biomass chips generated at Auburn or Groveland with two ISO 17225-4 particle size standards

Cultivar	Number	Percentage of loads meeting each ISO criterion for particle size of graded wood chips									
		Criterion									
		31-mm standard (P31S)[a]					45-mm standard (P45S)				
		1-Main (3.5–31 mm)	2-Fines[b] (<6.35 mm)	3-Coarse (>45 mm)	4-Length (150 mm)	5-Cross section (4 cm²)	1-Main (3.5–45 mm)	2-Fines[b] (<6.35 mm)	3-Coarse (>120 mm)	4-Length (200 mm)	5-Cross section (6 cm²)
Mass requirement		>60 %	<10 %	<6 %			>60 %	<10 %	<6 %		
Overall	122	21 (14–29)	96 (92–99)	100	100	0	100	96 (92–99)	100	100	100
By site											
Auburn	71	1 (0–6)	100	100	100	0	100	100	100	100	100
Groveland	51	49 (35–63)	90 (82–98)	100	100	0	100	90 (82–98)	100	100	100
By cultivar											
Mixed	58	22 (12–33)	96[c] (91–100)	100	100	0	100	96[c] (91–100)	100	100	100
SX61, SX64, and S67 (S. miyabeana)	30	0	100	100	100	0	100	100	100	100	100
SV1 (S. dasyclados)	22	54 (34–75)	91[c] (79–100)	100	100	0	100	91[c] (79–100)	100	100	100
Fish Creek (S. purpurea)	12	92[c] (24–100)	100	100	100	0	100	100	100	100	100

Criterion is defined as (1) main fraction, (2) fine fraction, (3) coarse fraction, (4) maximum length, and (5) maximum cross-sectional area in the table titled "Particle size of graded chips" in the ISO standard [24]. The particle size in this case was measured to a minimum size of 6.35 mm instead of 3.15 mm due to equipment limitations

[a] This standard is probably achievable in field with minor adjustments to the harvester blade settings, but it may have an impact on harvester throughput [60]

[b] These likely meet the 3.15-mm fine fraction standard, but the minimum screen size available for this project was 6.35 mm

[c] Not significantly different from 100 % compliance

Standards define boundaries for various feedstock characteristics; however, there seems to be little discussion about acceptable failure rates given the observed variability. Information is lacking on the consequences of failed tests for ash or other characteristics when delivered to an end user. What percentage of loads must fail before chips from a supplier have to undergo greater scrutiny or are rejected altogether? Quality assurance protocols are not prescribed in the ISO documentation specifically; however, suppliers can establish quality assurance/quality control procedures based on customer needs, and accreditation processes exist for certain biomass supplies [22, 56].

Given that determining standard compliance is incumbent on the supplier, the issues raised by Kenney et al. [4] are not completely resolved when considering feedstocks that are predominantly satisfactory but may occasionally fail due to site, cultivar, or other factors that may be in large part out of the supplier's control. Suppliers will likely have to rely on developing technologies such as near-infrared spectroscopy and sampling strategies [57–59] so that biomass can be graded for key parameters such as ash, moisture, and fines as it is produced.

Strategies for mitigating or preventing loads that fail standards should be developed. Loads could be mixed, but each handling step has the potential to increase fines and incur costs. Cultivars that have been identified as having low ash, such as Fish Creek or SV1 in this study and in the pristine samples [26], could be interplanted essentially creating mixed or blended feedstocks at the point of production. However, there is little research concerning how competition between North American cultivars would affect production of mixed willow plantings [60, 61] and how harvesting operations would be impacted by the variation in stem size and form that would be present in the field. Depending on the degree of homogeneity required, the blending of cultivars could be achieved by mixing at short-term storage sites or by planting relatively small blocks of cultivars and including more than one cultivar in each load of harvested material.

This study only tracks chips from harvest in the field to delivery to short-term storage. Temporary storage and additional handling to deliver the biomass to an end user increase the risk for contamination or degradation of quality but could also provide opportunities to improve quality. Minimizing handling is one important factor because each time chips are handled, the potential for contamination exists and/or fines are increased. In supply chains that include temporary storage, soil contamination is a concern if the biomass is stored on the ground or transferred by vehicles used for other purposes. Storage, however, could be viewed as an opportunity to improve the product, particularly with regards to moisture content through various management practices [47, 62].

Conclusions

Ash and moisture were the primary quality concerns associated with the biomass generated from a commercial-scale harvest in Auburn and Groveland, NY. The amount of variation in these characteristics was small relative to data that has been reported for large-scale harvests of herbaceous crops and agriculture residues. The biomass was generally compliant with the ISO standard for B1 wood chips for ash content; however, 18 % of the loads from Auburn were over the 3 % threshold for ash content by half-percentage point or less. Results indicate that ash content is significantly affected by site and cultivar main effects. Samples obtained from the Groveland harvest were fully compliant with the ISO B1 standard for ash. The ash content could be managed in the future by selecting low-ash cultivars or identifying high-ash producing sites and interplanting low-ash cultivars to create blended loads at harvest. Certain cultivars (e.g., Fish Creek) were 100 % compliant with the ISO B1 standards for ash content regardless of the site where they were harvested.

The harvester set at its largest chip size produced material that easily met the ISO P45S particle size standard. The P31S and P16S standards are probably achievable with different settings and setups within the harvester, but it may come at a cost of the harvester throughput and harvesting costs. Qualitatively, the material generated was very consistent with regards to size, and there were no incidences of oversized material which had been a concern while testing other harvesting platforms.

This work highlights several needs for future research. Because of site factors, an experiment where hand harvesting precedes a commercial harvest should take place so that the changes in chip quality due to the mechanical harvester and collection system can be accessed. Data acquisition for time-motion work needs to improve. Specifically, methods for determining material capacity and evaluating chip quality as biomass is produced would permit precise linkages between site and stand factors on the ground to the production and quality of SRWC biomass feedstocks. Additionally, precise tracking within the chain of custody, and a rapid means to grade chips, is required to evaluate changes in quality, or sources of contamination, as biomass is transported, stored, and delivered to end users.

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