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Application of selective bio-product recovery process developed for flax shive to additional biomass feedstocks: oat hulls, sunflower hulls, hemp hurds, and cereal straw

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ABSTRACT Development has been already underway to recover multiple high-value products from flax shive, an agricultural by-product of flax straw processing to remove fibre. Sequential extractions of flax shive in earlier work yielded separate streams of phenolics and hemicellulose-derived carbohydrate polymers. Preliminary experiments were undertaken to extend application and evaluation of this same two-step extraction process to four other agricultural by-products: oat hulls; hemp hurd; sunflower hulls; and cereal straw, specifically the flaked Simply Straw™ product, made from a combination of wheat and barley straw. Compared to flax shive, oat hulls were found to be the second highest ranked feedstock on a preliminary basis. Oat hulls, quantitatively, showed roughly 50% higher yield of recovered organics than flax shive, but confirmation of product quality is still necessary. Oat hulls also showed a high yield of carbohydrate precipitates, however, in terms of product quality, this was compromised by the likely presence of significant amounts of starch that could interfere with recovery of hemicellulose carbohydrates. Follow-up work on oat hulls is warranted, but a somewhat different processing approach would be necessary to take account of starch. In ranked order the remaining feedstocks were: hemp hurd; followed by sunflower hulls; followed by the Simply Straw™ product. None of these appeared to show significant promise. Hemp hurd appears better suited to use as an absorbent product, and sunflower hull appears better suited to use as a solid-fuel energy product. Cereal straw is the most problematic feedstock. Despite abundance across Western Canada, significant future work would be required to consider such material for high-value product recovery based on the extraction processes employed. Work to date suggests that flax shive and oat hulls are the best biomass materials to use as feedstock for chemicals and high-value product recovery.

Keywords: high-value bio-products; oat hulls; sunflower hulls; hemp hurds; cereal straw; flax shive; phenolics; hemicellulose carbohydrates.

INTRODUCTION The recovery of high-value constituents has been identified as an important practical approach to address the high costs and uncertainties associated with biomass processing, particularly for ligno-cellulose (Zhang 2011). However, the nature of products and the extent of associated yield and purity depend both on the feedstock involved and the recovery process. The suitability of any combination of process and feedstock is thus not guaranteed.

Analysis by Parsons et al. (2011a) suggested that within the Province of Manitoba, a number of prospective feedstocks exist that might be suitable for high-value product recovery. Foremost among these has been flax shive, which was identified as the top priority feedstock for such applications. Flax shive represents the woody xylem tissue left over after decortication of flax straw to remove fibre from the stem periphery. Flax shive is available in large quantities at low cost, and with relatively consistent particle size and composition. A sequential, two-step biomass extraction process for recovery of high-value products from flax shive has been recently reported, with all extractions undertaken at ambient conditions to reduce energy inputs and costs.

The first-step extraction, reported by Parsons et al. (2011b), employs sodium ethoxide in anhydrous ethanol, a catalyst similar to that used for industrial biodiesel, to recover a stream of phenolic organics. At small-scale, the mass yield of organic product using 1.0 M sodium ethoxide in anhydrous ethanol was 54.5 ± 14.5 mg/g on a dry basis (db) (n = 6). Yield appeared to be linearly related to the catalyst concentration, but not affected by particle size, at least in the tested range of about 0.5 mm to 3.0 mm. Analyses using ^{1}H NMR consistently showed the extracts to be phenolic in nature, and to contain no carbohydrate constituents. The presence of phenolics suggested that these extracts were likely derived from lignin.

The second-step extraction, reported by Parsons et al. (2013), employs aqueous sodium hydroxide (NaOH) to recover a stream of hemicellulose-derived polysaccharides, notably glucoronoxylan. At small-scale the mass yield of carbohydrate precipitates using aqueous 1.0 M NaOH was 99.4 ± 5.1 mg/g (db) (n = 16), and was unaffected by pretreatment using sodium ethoxide. Analysis of polysaccharide backbone monomers using high pressure liquid chromatography (HPLC) showed extracts, with or without pretreatment, to have consistently high molar concentration ratios of xylose-to-glucose, i.e., 25.5 ± 3.4 (n = 12), and with no mannose present. These results suggested a high concentration of glucuronoxylan polymer, likely greater than 90% by mass, and with no glucomannan polymer present.

Flax shive, however, is not the only potential feedstock that could be used for high-value product recovery. Additional major biomass feedstocks identified by Parsons et al. (2011a) include: oat hulls, sunflower hulls, hemp hurd, and cereal straws (primarily wheat and barley). The objective of this work was to undertake a preliminary evaluation of product recovery for the other available biomass materials using the same two-step extraction process as employed for flax shive under the same processing conditions.

METHODS AND MATERIALS

Feedstock Sources The four feedstock materials and their respective sources were as follows: (i) oat hulls were provided from Viterra Inc.'s oat processing facility in Portage la Prairie, Manitoba, with separate samples of unground and ground oat hulls being provided; (ii) sunflower hull samples were provided by Keystone Grain Ltd. of Winkler, Manitoba; (iii) hemp hurd samples were provided from Emerson Hemp Distribution Company of Emerson, Manitoba; and (iv) Simply Straw™ product samples were provided by Biovalco Innovative Products Inc. of Winnipeg, Manitoba. The Simply Straw™ product is a flaked biomass material composed primarily of wheat and barley straw.

Feedstock Screening Feedstock materials were pre-screened to ensure consistent particle sizes. To be consistent with earlier tests using flax shive (Parsons et al. 2013), all materials were tested using samples between 1.18 mm to 2.36 mm (i.e., passing #8 mesh screen and retained by

#16 mesh screen). For ground oat hulls, a second particle size fraction was also tested, between 600 μm and 850 μm (i.e., passing #20 mesh screen and retained on #30 mesh screen).

Feedstock Moisture Content Moisture contents for sample materials were determined using ASAE standard S358.2 (ASAE 2004). This involving three random samples retained in a controlled temperature oven at 103°C ± 2°C for a period of twenty-four hours.

Sequential Extraction Steps Extraction procedures were as outlined in Parsons et al. (2013). Samples for all materials were extracted sequentially, first using 1.0 M sodium ethoxide in anhydrous ethanol, followed by aqueous 1.0 M NaOH. For the two ground oat hull fractions, samples were also extracted solely using aqueous 1.0 M NaOH without pretreatment.

The recovered mass of organics from sodium ethoxide extraction was determined as outlined in Parsons et al. (2011b). The recovered mass of carbohydrate precipitates from 1.0 M NaOH extraction was determined as outlined in Parsons et al. (2013). No further composition-related analysis was undertaken for either the recovered organics or carbohydrate precipitates.

Filtrate Absorbency/Transmission Colour is key measure of purity for recovered hemicellulose carbohydrates (Watson and Williams 1959). Although directly measuring the colour of precipitates in this case was not practical, it was possible to evaluate the absorbency/transmission of carbohydrate filtrate solutions after removal of precipitates. In this case a lower absorbency (higher transmission) would correspond to a more desirable characteristic.

Measurements were undertaken using a Philips Model 8675 VIS Spectrophotometer with ethanol solution (80%) used as the zeroing-blank liquid. Given uncertainty of constituents present in the filtrates, a single wavelength was used, 570 nm. This represented essentially a generic "yellow" colour. In this case measurements were reported as percent transmission given relatively low absorbency levels. In order to provide a baseline for comparison, all replicate filtrate samples from flax shive extraction combinations undertaken in Parsons et al. (2013) were also measured for percent transmission in the same way.

Comparative Criteria-Based Feedstock Evaluation Given diversity of available materials and multiple product streams, a standard business-based criteria-ranking process was used as a simple preliminary approach to compare the different feedstock materials to flax shive. Four criteria were employed to score each of the biomass materials in terms of suitability. These were: (i) quantitative yield of organic extracts as compared to flax shive; (ii) apparent organic extract product quality compared to flax shive; (iii) quantitative yield of carbohydrate precipitates compared to flax shive; and (iv) apparent carbohydrate precipitate product quality compared to flax shive. Scoring for each criterion was as follows: score +2 if demonstrably better than flax shive; score +1 if better than flax shive; score 0 if similar to flax shive; score -1 if not as good as flax shive; and score -2 if demonstrably worse than flax shive.

RESULTS AND DISCUSSION

Moisture Content Moisture content and resulting dry matter results for each of the biomass materials are presented in Table 1. Sunflower hulls were found to have the lowest moisture content of all the materials tested. Ground oat hulls had the highest moisture content, in particular higher than unground oat hulls. Dry matter content data were used to adjust product yields to a dry basis.

Table 1. Moisture content and resulting dry matter content of biomass materials, with each mean and standard deviation value based on three replicate experiments, presented in order from highest to lowest moisture content.

Biomass Material	Moisture Content (mg/g wb)	Mean Dry Matter (mg/g)
Oat hulls – ground	111.8 ± 0.7	888.2
Oat hulls – unground	95.5 ± 2.7	900.4
Hemp hurd	89.3 ± 5.4	910.7
Simply Straw™ product	74.9 ± 1.1	925.1
Sunflower hulls	65.7 ± 1.2	934.2

Recovered Organics (First-Step Extraction) Yields of organic constituents extracted using sodium ethoxide in anhydrous ethanol are presented in Table 2 for the various biomass materials. From these results only oat hulls had a quantitative yield that was obviously different from flax shive, in this case being appreciably higher. At the same time there was little apparent effect due to particle size, or whether the oat hulls were ground or unground. The average yield of organics from the three samples of oat hulls was 83.3 ± 4.7 mg/g (db) (n = 3), more than 50% higher than for flax shives. Given that oat hulls are commonly used for animal feed, their typical chemical composition is understood (Heuze et al., 2013), as discussed later. Quantitatively, the other biomass materials, including sunflower hulls, hemp hurd and Simply Straw™ product, all had yields that were within the error band associated with the extraction of organics from flax shive.

Qualitatively, the organics derived from sunflower hulls were more liquid-like and “flowable” at room temperature than those from flax shive. Such characteristics could be explained by the presence of fatty-acid esters, i.e., lipid fatty-acid materials being converted to biodiesel-type esters in the presence of alkoxide catalyst. Sunflower hulls in the past have been specifically analyzed for lipid materials (Cancalon, 1961), and found to contain significant lipids, including waxes, i.e., around 50 mg/g (db). Sunflower hulls, like oat hulls, are used for animal feed, and as such their typical chemical composition is also understood (Heuze et al., 2012). The typical content of lipids (ether extractable) has been recently indicated to be around 52 mg/g (db), which is consistent. Further, given the seed-oil nature of sunflower, with high lipid content, it is also possible for residues of lipid containing seed-meal to be retained with the hulls. Given observed characteristics, product derived from sunflower hulls was deemed not as good as flax shive extracts.

The organics derived from Simply Straw™ product produced a very odd, and potentially offensive odour. This odour was distinctly different from the more “phenolic” aroma associated with extracts from flax shive and the other biomass materials. This extract from Simply Straw™ product obviously contained very different, albeit undetermined constituents, and was deemed to be demonstrably worse than flax shive extracts.

The organics derived from oat hulls and hemp hurd appeared similar to flax shive extracts. As such, from a qualitative perspective, oat hull and hemp hurd extracts were both deemed similar to flax shive. The actual chemical nature of oat hull extracts will need to be confirmed, but oat hulls typically contain little in terms of lipid materials (i.e., ether extractable around 22 mg/g db). Further, the oat seed itself contains only in the range of 4% to 10% lipid (Young et al., 1977), such that any carry over of seed-meal into the hulls would not entail appreciable lipids either. These observations suggest that the extracted organics could be reasonably derived from the lignin component, as the suspected case with flax shive extracts (Parsons et al. 2011b). The similar product quality, combined with the much higher yield, suggested that further investigation of oat hulls was warranted in terms of organics extraction.

Table 2. Extraction yield of solvent-soluble organics from biomass for first-step extraction using 1.0 M sodium ethoxide in anhydrous ethanol, based only on single experiment per biomass material, and presented in order from highest to lowest yield value.

Biomass Material	Organic Product Yield (mg/g db)
Oat hulls - ground (600 to 850 µm)	87
Oat hulls - ground (1.18 to 2.36 mm)	85
Oat hulls - unground (1.18 to 2.36 mm)	78
Sunflower hulls (1.18 to 2.36 mm)	66
Hemp hurd (1.18 to 2.36 mm)	48
Simply Straw™ product (1.18 to 2.36 mm)	46
Flax shive (Parsons et al. 2011b)	54.5 ± 14.5 (n = 6)

Recovered Carbohydrate Precipitates (Second-Step Extraction) Yields of carbohydrate precipitates extracted using aqueous 1.0 M NaOH are presented in Table 3 for the various biomass materials. Yields from all were either higher or lower, quantitatively, than from flax shive. None was within the error band for flax shive. Oat hulls and the Simply Straw™ product had higher yields than flax shive, while hemp hurd and sunflower hulls were lower.

The yield from oat hulls was affected both by particle size and by pretreatment method. The use of sodium ethoxide caused a significant reduction in precipitate yield by 30% to 50%. This was very different from the behaviour observed for flax shive, where in contrast the organics extraction did not impact carbohydrate precipitate yield, either quantitatively or qualitatively (Parsons et al. 2013). Particle size of ground oat hulls also affected carbohydrate precipitate yield. The larger-sized fraction had a significantly higher yield in this case, i.e., 80% to 140% higher, whether using aqueous NaOH alone or after pretreatment. This result was unexpected, in that smaller particle-size generally would be expected to result in either higher or at least equivalent yield, not lower yield, given higher aggregate surface area and resulting enhanced mass-transfer.

Qualitatively, flax shive-derived precipitates obtained by Parsons et al. (2013) had a puffy appearance, never settling compactly. Such observations were consistent with the presence of charged glucuronoxylan polymer. Observations of extracted products from hemp hurd and sunflower hulls were similar to flax shive. Both hemp and sunflower, like flax, are dicotyledons, and in all cases glucuronoxylan is a major hemicellulose matrix polymer (Ebringerova and Heinze, 2000). As such, qualitatively, both were deemed as similar to flax shive.

Polysaccharide precipitates from oat hulls and the Simply Straw™ product, on the other hand, appeared to be obviously different from flax shive. Precipitates from Simply Straw™ product were much darker, including obvious dark fragments that were not observed for any other feedstock. As such, it was deemed as demonstrably worse, qualitatively, than flax shive.

Precipitates from oat hulls appeared to contain some puffy materials, similar to flax shive, but also significant amounts of white, stringy solids that settled quickly and compactly. In addition, extraction solutions using aqueous NaOH alone quickly became gelatinous. These observations strongly suggested the presence of significant starch in the extracts (Watson and Williams, 1959). Importantly, these likely starch solids appeared to be present in all extracts whether or not pretreatment was involved.

It was not initially expected that oat hulls would contain significant starch residues, but it was entirely logical. Oats, as a crop, is exploited primarily for its starch content. In dehulling, the inner seed is the priority to obtain as pure as possible. As such, the wastage of some residual seed into the hull fraction would be acceptable, rather than the other way around. This translates to the presence of some residual starch content in oat hulls. Indeed, Heuze et al. (2013) confirmed that oat hulls typically contain seed fragments, and have a typical starch content of around 99 mg/g (db). Specifically regarding the recovery of hemicellulose from oat hulls, Anderson and Krznarich (1935) noted that starch can be present, and also confirmed that it can be co-extracted by NaOH.

Qualitatively, carbohydrates from oat hulls were deemed to be not as good as flax shive for the extraction process as undertaken, specifically given the presence of starch contaminating the hemicellulose. At the same time, the presence of starch in oat hull extracts and its co-extractability using NaOH, have additional important implications. In order to obtain hemicellulose-derived polysaccharides in a relatively pure form, it would be necessary to separately remove residual starch first. As such, a different process would be required, more tailored specifically to oat hulls.

Table 3. Carbohydrate precipitate yield from biomass feedstocks on dry basis (db) for second-step extraction using aqueous 1.0 M NaOH, based only on single experiment per biomass material, and presented in order from highest to lowest yield value.

Biomass Material	Precipitate Yield (mg/g db) using 1.0 M NaOH	
	No Pretreatment	After Sodium Ethoxide
Oat hulls - ground (1.18 to 2.36 mm)	249	177
Oat hulls - ground (600 to 850 µm)	140	75
Simply Straw™ product (1.18 to 2.36 mm)		134
Oat hulls - unground (1.18 to 2.36 mm)		133
Hemp hurd (1.18 to 2.36 mm)		83
Sunflower hulls (1.18 to 2.36 mm)		55
Flax shive (Parsons et al. 2013)	99.4 ± 5.1 (n = 16)	

Carbohydrate Filtrate Transmission (Absorbency) Percent transmission results at the selected wavelength of 570 nm for all of the flax shive extractions tested by Parsons et al. (2013) are presented in Table 4. The order of treatment conditions, from highest to lowest percent transmission, was virtually the reverse of the order for both precipitate mass yield and molar ratio of xylose-to-glucose for digested precipitate samples. All percent transmission values, nevertheless, were high, ranging from 98.4 to 99.7%T. These values suggested a relatively low content of contaminating colourants.

Percent transmission results for carbohydrate precipitate filtrates from all the other biomass materials are presented in Table 5. The results for oat hull extract filtrates were similar to flax shive, ranging from a low value of 98.8%T to a high value of 99.7%T. As such, the level of contaminating colourants would be expected to be in a similar range as for flax shive. The other three materials all showed lower percent transmission levels, suggesting higher levels of contaminating colourants. Based on this, carbohydrates from both hemp hurd and sunflower hulls were deemed, qualitatively, to be not as good as flax shive. Consistent with earlier observations of dark fragments, the percent transmission value for the Simply Straw™ product filtrate was the lowest overall. Given these results, carbohydrates from the Simply Straw™ product, as representative of cereal straw, were deemed, qualitatively, to be demonstrably worse than flax shive.

Table 4. Filtrate percent transmission results at selected wavelength of 570 nm for all extraction conditions tested in Parsons et al. (2013), after carbohydrate precipitate removal from flax shive (1.18 to 2.36 mm particle size in all cases), with mean and standard deviation based on four replicate experiments, and presented in order of highest to lowest transmission.

Flax Shive Extraction Conditions	Percent Transmission at 570 nm
Saturated aqueous $Ba(OH)_2$ after pretreatment with 1.0 M sodium ethoxide in anhydrous ethanol	99.7 ± 0.4 %T
Saturated aqueous $Ba(OH)_2$ only	99.7 ± 0.2 %T
Saturated aqueous $Ba(OH)_2$ with 1.0 M NaOH added	99.3 ± 0.3 %T
1.0 M aqueous NaOH after pretreatment with 1.0 M sodium ethoxide in anhydrous ethanol	99.0 ± 0.7 %T
1.0 M aqueous NaOH after pretreatment with azeotropic ethanol	98.7 ± 0.3 %T
2.0 M aqueous NaOH elevated concentration	98.4 ± 0.4 %T
1.0 M aqueous NaOH only	98.4 ± 0.1 %T

Table 5. Filtrate percent transmission results of filtrate samples after carbohydrate precipitate removal for biomass material samples at selected wavelength of 570 nm, based on only single measurement per set of conditions, and presented in order from highest to lowest transmission.

Biomass Material	Percent Transmission (570 nm) for Filtrate	
	No Pretreatment	After Sodium Ethoxide
Oat hulls - unground (1.18 to 2.36 mm)		99.7 %T
Oat hulls - ground (600 to 850 µm)	99.2 %T	99.5 %T
Oat hulls - ground (1.18 to 2.36 mm)	98.8 %T	99.2 %T
Hemp hurd (1.18 to 2.36 mm)		97.9 %T
Sunflower hulls (1.18 to 2.36 mm)		97.5 %T

Simply Straw™ product (1.18 to 2.36 mm)	96.2 %T

Overall Feedstock Evaluation Compared to Flax Shive An overall evaluation of the biomass materials compared to flax shive is presented in Table 6, based on the four identified criteria. Overall, oat hulls were shown to be likely the second best feedstock choice next to flax shive for extraction of high-value products. This was, however, subject to the identified constraint that a different processing approach would be required given the presence of significant starch. The yield for organic product recovery, in particular, was found to be significantly higher for oat hulls than for flax shive.

Of all of the biomass materials evaluated so far, flax shive and oat hulls appear to represent the most promising candidates for the production of chemicals, including high-value products. It is important to note that in the past oat hulls have had a much stronger association with high-value chemical products, in this case primarily as a feedstock for furfural production (Dunlop, 1948). Further investigation into oat hulls as a feedstock is warranted.

The remaining products in order were hemp hurd, followed by sunflower hulls and the Simply Straw™ product, the last two ranked the same. Hemp hurd did not show any strong promise for high-value product extraction. The results emphasized key differences between hemp hurd and flax shive. Although both are derived from dicotyledonous plants via decortication processes, their prospective applications appear very different. Earlier work by Parsons and Cenkowski (2011) showed hemp hurd to exhibit a relatively high liquid-holding capacity across a range of liquids, much higher than flax shive, and thus well suited as an absorbent, particularly for non-aqueous solvents. The current work showed hemp hurd to be much less promising as a feedstock material for high-value product extraction.

Similarly, sunflower hulls showed little potential promise for high-value product extraction. Sunflower hulls were hampered in particular by organic extracts with lower apparent quality, and poor extraction yield of hemicellulose carbohydrates. At the same time, as noted by Heuze et al (2012), sunflower hulls have a high gross energy content of more than 20 MJ per kg. Their use as solid-fuel for energy likely would make more sense.

The Simply Straw™ product was the most problematic overall. Cereal straws are abundance across Western Canada, including within Manitoba. However, product quality was very poor both for recovered organics and carbohydrate precipitates. Significant further development work would be required in order to consider such materials for high-value product extraction, at least based on the extraction methods considered.

Table 6. Results of evaluation of biomass materials compared to flax shive for value-added product pre-extraction, based on preliminary tests.

Evaluation Criterion	Oat Hulls	Hemp Hurd	Sunflower Hulls	Cereal Straw
1. Organics yield	+1	0	0	0
2. Organics quality	0	0	-1	-2
3. Carbohydrates yield	+1	-1	-1	+1
4. Carbohydrates quality	-1	-1	-1	-2

Comparative total score	+1	-2	-3	-3
Rank relative to flax shive	2nd	3rd	4th	5th

CONCLUSIONS The results of this preliminary evaluation identified oat hulls as the likely next best candidate feedstock for high-value product extraction compared to flax shive. Oat hulls produced a higher yield of organic extracts compared to flax shive, but the composition and quality of product will need to be verified. Oat hulls produced very high yields of carbohydrate precipitates, but this was compromised by the likely presence of undesirable starch that is co-extracted by aqueous NaOH. Given the presence of starch, a different processing approach likely would need to be developed to fully exploit oat hulls. Flax shive and oat hulls thus represent the likely best biomass materials, of those tested so far, for recovery of chemicals, including high-value products.

Hemp hurd and sunflower hulls showed little potential for high-value product extraction, and are likely better suited to other uses, namely absorbent products and solid fuel, respectively. The most problematic feedstock was the Simply Straw™ product, representative of cereal straws. Cereal straws are highly plentiful, but significant product quality concerns were identified. Much more development work would be required in order to consider cereal straws for high-value chemical product recovery, at least based on the extraction methods considered.

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