# The Papermaking Properties of Washington State Wheat Straw

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Abstract

# The Papermaking Properties of Washington State Wheat Straw

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Current environmental and social pressures are limiting wood harvest from U. S. public forest land. As a byproduct of the cereal grain industry, wheat straw may also be used for papermaking. However, studies of wheat straw have identified heterogeneity within this potential raw material. The chemical composition and fiber properties of the wheat straw vary with growing condition, wheat cultivar, and with the part of the plant processed. These differences in chemical composition may affect both pulping yield and the non-process elements entering the pulp mill. The taller plants have higher average fiber lengths which correlate with pulp strength properties. This local range of growing conditions, cultivars, and the abundance of straw will give papermakers the prospect of selectively collecting the straw best suited for their process and product. With these opportunities, wheat straw has the potential to be an additional industrial papermaking raw material.

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# Chapter 1
# Introduction

Worldwide paper and paperboard consumption has been predicted to increase by 50% over our current production rates by the year 2010 [Tombaugh, 1998]. While increases in pulp yield may compensate for some of the demand [Courchene, 1998], changes in our fiber supply will also be needed. Some of the possible changes include an increased use of tree plantations [Dvorak and Hodge, 1998; Robinson *et al.*, 1998; Koncel, 1998] and secondary fibers [Finchen, 1998] along with a closer look at alternative raw materials like urban wood and nonwoods [Byrd and Gratzl, 1998; Baker, 1998; Staniforth, 1979].

Nonwood fiber supplies are often divided into two main categories, fiber crops and agricultural residues. With fiber crops like kenaf, the primary agricultural product is the papermaking raw material. Therefore, a mill or raw material supplier would need to obtain long term contracts from these farmers or grow the fiber crop themselves to ensure the availability of the fiber supply. However, agricultural crops like sugar cane, corn, wheat, and rice are already well established. Selling the crop residues as papermaking raw materials could be a secondary income for the farmer while providing a stable source of fiber to the paper industry.

In Washington State, wheat grain is one of the top export commodities [Washington State OFM, 1997]. Wheat is grown on three million acres in the state [Washington Agricultural Statistics Service, 1998] and generates 2-6 tons of straw per acre [Wyoski, 1989]. While some of this straw must remain on the land to retain soil quality, a significant portion is recoverable for alternate uses.

Pulp mill production size and location are highly dependent on local situations, and those considerations are far beyond the scope of this project. However for reference, processing this amount of wheat straw would be equivalent to approximately three 500 ton per day pulp mills, each using the straw produced in about a 25 mile growing radius of each mill in irrigated regions or a 50 mile radius in dryland areas.

Wheat straw could be used in many types of paper. Wheat straw pulp has been used to make corrugating medium and been used as part of the furnish in linerboard, grocery bags, toilet paper, and communications paper [Misra, 1987; Tabb, 1974]. When adding wheat straw pulp to a blend, the resulting sheet has improved paper properties. The increase in fines content with wheat straw pulp improves the sheet formation [Watson and Garner, 1997; Rab and Szikla, 1992; Chiou, 1990; Tabb, 1974]. These same fines have been found to flocculate with fillers thereby improving filler retention and reducing the amount of dusting at higher filler contents [Rab and Szikla, 1992; Papp and Renner, 1990; Watson and Garner, 1997]. Papp and Renner found the addition of wheat straw pulp allowed higher filler contents at the same paper strength. The higher hemicellulose content of straw pulp contributes to superior hydrogen bonding between the fibers [Watson and Garner, 1997; Papp and Renner, 1990; Tabb, 1974]. Wheat straw containing sheets also have good bulk and stiffness [Watson and Garner, 1997].

Wheat straw fibers are thin walled and help create smooth, closed sheet surfaces [Watson and Garner, 1997; Rab and Szikla, 1992]. This improved sheet surface may increase printability, but the presence of the nonfibrous cells in wheat straw pulp leads to excessive linting on printing presses, an increase in cationic starch demand, and poorer drainage [Cheng *et al.*, 1994; Watson and Garner, 1997; Rab and Szikla, 1992]. Lower freeness reduced or eliminated refining but also impaired the removal of water from the straw pulp in pulping, bleaching, and papermaking operations. Addition of wheat straw has also lead to a more brittle, hygroexpansive sheet (moisture-sensitive in both the machine and cross-machine directions) and in higher quantities may decrease sheet strength [Watson and Garner, 1997; Rab and Szikla, 1992].

While wheat straw pulp can successfully be made into paper, the chemical composition makes the processing of straw challenging. Inorganics content is a significant difference between wood and straw composition. Most wood has an almost negligible ash content, however the ash content of wheat straw ranges from 3 to 11%. These wheat straw inorganics can be 65-70% silica dioxide [Eroglu and Deniz, 1993]. This silica blunts the blades of cutting equipment, reduces digestibility for animals, interferes with pulping process, and renders straw combustion more difficult [Staniforth, 1979]. Silica often dissolves during alkaline pulping and is found as sodium silicate or other compounds in the black liquor [Eroglu and Deniz, 1993]. The increased silica content in black liquor interferes with chemical recovery through [Watson and Garner, 1997; Staniforth, 1979]:

- scaling formed by precipitation in the multiple effect evaporators,
- a reduction in the efficiency of causticization by the formation of a colloid in smelt tanks,
- creating glass coatings on both the individual lime particles and the inside of the lime kiln, and
- an increase in smelting temperature caused by an increased smelting point.

To deal with the impact of silica, alternative pulping methods and silica removal operations have been and are continuing to be explored [Chen *et al.*, 1996].

When examining wheat straw as a papermaking raw material, the fiber properties and chemical composition of the local straw are of interest. Great variations have been reported in these values. Table 1.1 lists some of the reported average fiber lengths for wheat straw. While some references report average fiber lengths lower than hardwood [Cheng *et al.*, 1994; Rydholm, 1965], others describe the reverse [Utne and Hegbom, 1992].

Unfortunately, measurement technique is not always mentioned with the reported values; and the various fiber length measuring and reporting methods produce drastically different results. For example, the Kajaani FS-200 is better able to detect fines in a sample than the FS-100 [Bichard and Scudamore, 1988]. Using the same straw pulp sample, previous studies at the University of Washington have found that the weighted average fiber length (WAFL) values from the Kajaani FS-200 can be 50% smaller than the values from the Kajaani FS-100 [McKean, 1996]. The ability to detect and measure fines is a key part in understanding straw pulp. Straw can contain 40-60% non-fibrous cells [Cheng *et al.*, 1994; Utne and Hegbom, 1992; Rydholm, 1965]. These non-fibrous cells are short in length and speculated to be the cause of poor drainage in wheat straw

Table 1.1. Wheat Straw Average Fiber Lengths.

| | Fiber Length (mm) |
|---|---|
| Cereal Straw [Rydholm, 1965] | 0.3-0.5 (cells) 1.5 (fiber) |
| Wheat Straw, Norway [Utne and Hegbom, 1992] | 1.3 (plant) 1.1 (pulp) |
| Wheat Straw Pulp, NE Spain [Montané, *et al.*, 1996] | 1.13-1.22 |
| Wheat Straw, China [Zhang, *et al.*, 1990] | 1.73 (stem) 0.82 (node) |
| Wheat Straw Pulp, China [Cheng, *et al.*, 1994] | 0.26 (NAFL) 0.63 (WAFL) |
| Typical Wheat Straw, China [Hua and Xi, 1988] | 1.32 (NAFL) 1.49 (WAFL) |
| Eucalyptus Market Pulp* | 0.54 (NAFL) 0.69 (WAFL) |
| Canadian Aspen Pulp* | 0.62 (NAFL) 0.80 (WAFL) |

NAFL--numerical average fiber length, WAFL--weighted average fiber length
* Kajaani FS-100

pulp [Misra, 1987; Watson and Garner, 1997]. The average length of straw pulp has been reported to be 0.3 - 0.5 mm when measuring all of the straw cells. When examining just the fiber fraction of straw, the average fiber length was 1.5 mm [Rydholm, 1965]. Such reporting distinctions could, in part, explain the range shown in Table 1.1.

Measurement technique alone may not account for the large variation in average fiber length. Within-the-plant variation, genetics and the growing environment also influence fiber length. Wheat straw is a heterogeneous raw material containing internodes, leaves, and nodes (Figure 1.1) along with small quantities of debris (chaff, fines, residual grain). The relative quantity of each fraction varies (Table 1.2). These variations in composition may be due to the plants or to harvesting technique [Rab and Szikla, 1992] and have not been extensively examined. The parts of the plant have different average fiber lengths and

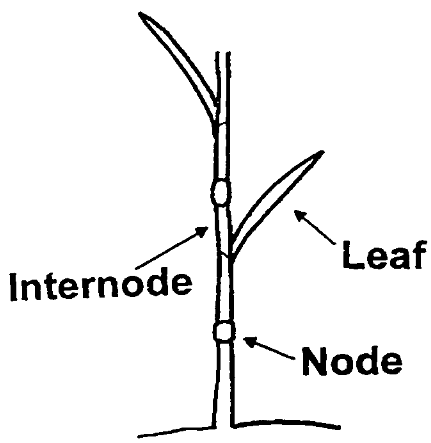

Figure 1.1. Above-Ground Wheat Tissue

Table 1.2. Composition of Wheat Straw

| | Ernst, 1960 | Mansour, 1985 | Zhang *et al.*, 1990 |
|---|---|---|---|
| Internodes, % | 68.5 | 46.7 | 52.1 |
| Nodes, % | 4.2 | 6.9 | 9.3 |
| Leaves (blade and sheath), % | 25.8 | 39.9 | 23.5 |
| Other, % | 1.5 | 6.5 | 15.1 |

chemical composition. Therefore, different amounts of nodes and leaves in the raw material could influence the average fiber length for the straw.

Growing conditions and genetics also influence fiber length. Ravn [1993] found that the pulping and papermaking characteristics of wheat straw varied with variety. When studying rice straw from Taiwan and Indonesia, Kuo and Shen [1992] found differences in fiber length and diameter. Therefore, the range in average fiber length reported for wheat straw pulp may be due to any or all of the following: measurement technique, different quantities of leaves and nodes in the raw material, growing environment, and wheat genetics.

As with fiber properties, the chemical composition of wheat straw has been examined by many researchers (Tables 1.3 and 1.4). These reported cellulose contents

Table 1.3. Chemical Composition of Wheat Straw

| | Ali *et al.* [1991] Pakistan | Aronovsky *et al.*[1948] Illinois | Mohan *et al.* [1988] India | Utne & Hegbom [1992] | Misra [1987] U.S. | Misra [1987] Denmark |
|---|---|---|---|---|---|---|
| Cellulose (alpha) (%) | 33.7 | 34.8 | | 29-35 | 39.9 | 41.6 |
| Hemicelluloses (%) | 25.0 | 27.6 | 28.9 | 26-32 | 28.2 | 31.3 |
| Lignin (%) | 16-17 | 20.1 | 23.0 | 16-20 | 16.7 | 20.5 |
| Ash (%) | 7.5-8.5 | 8.1 | 9.99 | 4-9 | 6.6 | 3.7 |
| Silica & Silicates (%) | 4.5-5.5 | | 6.3 | 3-7 | | 2.0 |
| EtOH-Benzene Extr. (%) | 5.8 | 4.5 | 4.7 | | 3.7 | 2.9 |

Table 1.4. Chemical Composition of Summer and Winter Wheat [Casey, 1980]

| | Summer Wheat Heavy Soils | Summer Wheat Light Soils | Winter Wheat Heavy Soils |
|---|---|---|---|
| Cellulose (alpha) (%) | 34.6 | 34.4 | 35.2 |
| Pentosans (%) | 26.2 | 27.0 | 27.1 |
| Lignin (%) | 16.3 | 17.0 | 15.7 |
| Ash (%) | 11.2 | 5.1 | 11.1 |
| Silica & silicates (%) | 4.4 | 0.9 | 4.9 |

range from 29-42% and the lignin, ash, and silica content also have wide ranges. The origin of these variations is inconclusive and may also be due to measurement methods, sampling, genetics, or growth conditions.

As seen with average fiber lengths, the parts of the plant have different chemical compositions. For example, the leaves contain more silica and less lignin than the node and internodal sections. If different harvesting techniques vary the amount of leaves, recovered straw collection practices would explain some of the range in composition.

In addition to within-the-plant variation, both climate and soil conditions affect the chemical composition of wheat straw. Compared to wood, annual crops such as wheat exhibit more year-to-year variation in the straw [Watson and Garner, 1997].

Tables 1.3 and 1.4 list a wide range of silica contents. While some of this variation may be due to differences in leaf content, the presence of silica is also dependent on soil conditions. Wheat straw grown in heavy soils (higher clay content) had more silica than straw grown in lighter soils (lower clay content). Without mentioning cultivars, Eroglu and Deniz [1993] found silica contents of 3.4 and 4.4% for two European straw samples grown in heavy soils while a straw grown in light soil had silica contents of only 0.9%. Casey [1980] reported similar trends (Table 1.4).

While most of the discussion about wheat straw chemical composition have compared the soil type to ash and silica content, little work has been reported on the impact of growing conditions and cultivar on the lignin and carbohydrate contents. Casey listed the chemical composition of a winter wheat and a summer wheat both grown in heavy soils (Table 1.4). Summer wheat had a higher lignin, lower pentosan and a lower alpha cellulose content than the winter wheat. Such variation in chemical composition may lead to differences in pulping and pulp properties.

With the wide range of reported values for wheat straw fiber properties and chemical composition, the actual properties of the local straw would be of interest to prospective users. Within Washington State, wheat is grown in diverse growing conditions and soil types. Numerous wheat cultivars have been developed to adapt to these conditions and to meet the demand for diverse markets. Different growing conditions and cultivars have the potential for variations in fiber properties and chemical composition. Currently, only a small fraction of wheat straw is used for industrial purposes, so, with the supply of straw significantly exceeding demand, the option of selective harvesting based on preferential chemical composition and fiber properties is available.

To identify variation in the papermaking potential of local straw, a preliminary study was conducted using a limited sampling of the 1994 Washington State wheat straw harvest in Eastern Washington. Fiber properties seemed to change within the wheat plant, between different wheat cultivars, and between growing locations (Chapter 2). Within-the-plant variation was found to exist not only between the different anatomical parts, but also along the stem of the plant (Chapter 3). These differences may lead to specialized harvesting and wheat plant fractionation to upgrade the papermaking potential of this raw material.

A more comprehensive examination of Washington State wheat straw found both genetic and environmental influences on the fiber properties (Chapter 4) and the chemical composition (Chapter 5) of the straw. The diversity of straw from within our state suggested benefits in selective harvesting and controlled mixing of the different straw sources.

Using the better cultivars, growing locations, and parts of the wheat plant provides opportunities to improve wheat straw pulp properties. An additional possibility for upgrading this raw material could be through the removal of some or all of the nonfibrous cells from the wheat straw pulp. Pulp fractionation has the potential for improving drainage and paper strength properties (Chapter 6).

This extensive examination of Washington State wheat straw will give local papermakers a better understanding of this available raw material. By knowing the variation and the potential impacts of selective harvesting and fractionation, more informed decisions may be made about the future of wheat straw as an industrial-scale, papermaking raw material.

# Chapter 2
# The Papermaking Potential of Pacific Northwest Wheat Straw

When first considering wheat straw as a potential papermaking raw material for the Pacific Northwest, an examination of the local straw is needed. Wide ranges of fiber properties have been reported for this raw material and the reasons for these ranges were ambiguous (Chapter 1). Some of the potential influences of fiber properties included measurement technique, within-the-plant variation, plant genetics, and growing conditions. This chapter will summarize a series of preliminary studies examining these influences on the papermaking properties of Washington-grown wheat straw.

The within-the-plant variation for Washington grown wheat straw was examined by comparing the node and internodal fiber properties of four locally-available straw samples. Twenty-two developmental cultivars (genetic isolines) were pulped to investigate the impact of wheat genetics. Lastly, the influence of growing environment was explored by comparing the fiber properties of six commercial cultivars each grown at an irrigated and a dryland location.

## Experimental

Non-replicated wheat samples were hand harvested from Washington State University cultivar comparison experiments and WSU/USDA-ARS experimental plots in Pullman, Washington. The internodes and nodes were hand separated from the rest of the stem (Figure 1.1, page 4). Acid chlorite defiberization was done by scaling up TAPPI Method 259-om88 from one gram to fifty grams and proportionately increasing the other reactants. Samples were analyzed for fiber length and coarseness using either the Kajaani FS-100 or Kajaani FS-200. To determine average fiber diameters, six hundred fiber diameters were measured using Bioscan Optimas software on an image analysis system. Length weighted average fiber lengths (WAFL), numerical average fiber lengths (NAFL), and numerical average fiber diameters (NAFD) quoted in this report are averages of all the straw cells that survived the defiberization stage. Significant differences were defined at the 0.05 level of probability.

## Results

Since the majority of the published, wheat-straw studies were done on raw materials from outside the Pacific Northwest, a few samples of PNW wheat straw were first examined. Both the internodes and nodes were defibrated. Similar to the findings of Zhang and coworkers [1990], the node fraction of the four samples had lower average fiber lengths (Figure 2.1) and more fibers per gram (Figure 2.2) than the pulp made from the internodes. Since nodes contain more short parenchyma cells than internodes [Misra, 1987], these results were not surprising. Similar fractionation of the internodes from the nodes may be beneficial in commercial operations. Such fractionation could be done in the field during the harvest, on site at the mill, or after pulping similar to removal of knots from wood pulp.

The internodal WAFL for these four samples ranged from 1.07 to 1.31 mm (Kajaani FS-100) with the two extreme values coming from within the same Madsen cultivar. This range suggested that both environmental and genotypic factors may influence the fiber properties of the straw.

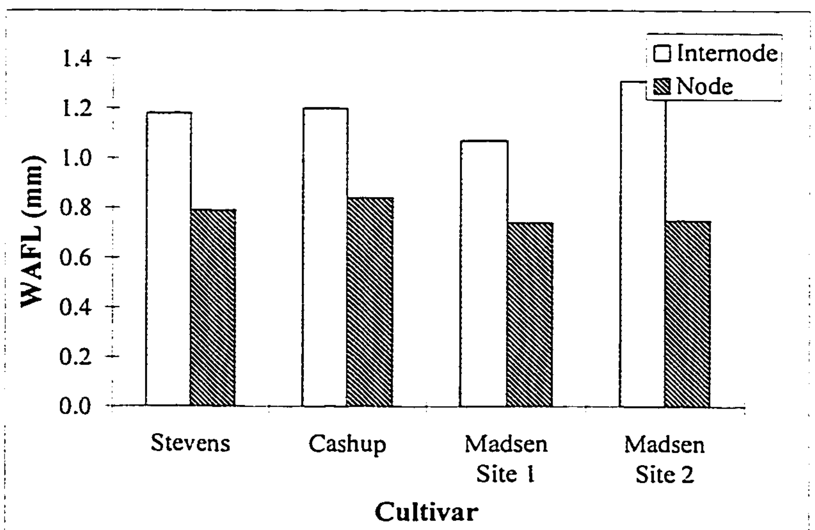


Figure 2.1. Node and Internode WAFL (Kajaani FS-100).

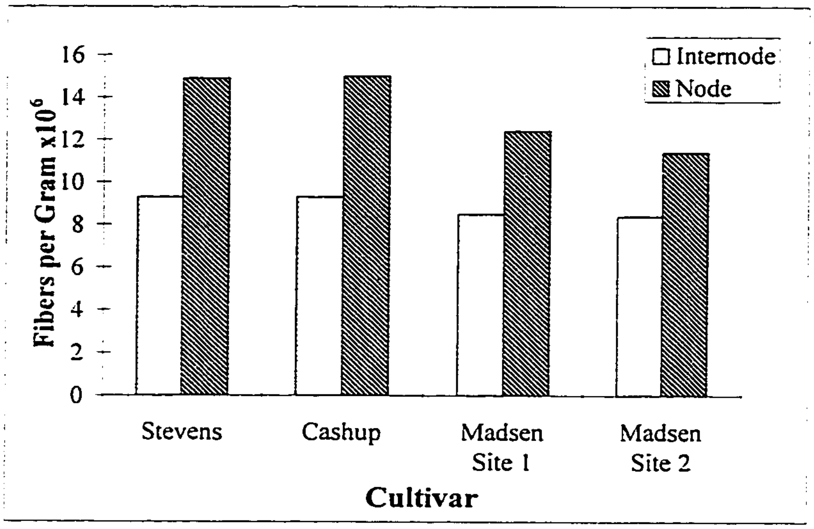


Figure 2.2. Node and Internode Fibers per Gram (Kajaani FS-100).

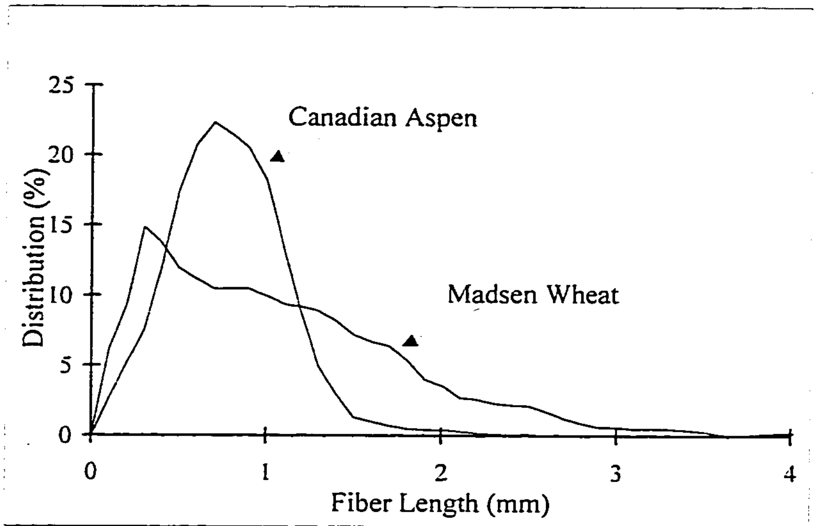


Figure 2.3. Fiber Length Distributions of Madsen Wheat and Canadian Aspen (Kajaani FS-100)

When comparing wheat straw pulp to some of the currently-used hardwood pulps, the average fiber lengths were of the same order of magnitude. However, these two pulps have different fiber length distributions (Figure 2.3). The wheat straw pulp has a much wider fiber length distribution. The straw contains more fines and more long fibers than the aspen pulp. This higher quantity of fines in wheat straw pulp may be plugging the sheet and causing wheat pulp to drain slower than wood pulp. Removal of wheat straw fines and short fibers would result in a pulp with a higher average fiber length than the aspen and possible superior properties. The impact of wheat straw pulp fractionation will be examined in Chapter 6.

The second stage of this study had two objectives: an examination of different PNW cultivars and of the effect of irrigation on fiber morphology. To compare different genotypes, straw was gathered from an ongoing WSU/USDA-ARS genetics study at Pullman, Washington. Twenty-two developmental wheat cultivars (isolines) were sampled from a randomized complete block experiment with identical growing conditions. The stem height of these varieties varied from 30 to 120 cm. Only the internodal pulp was analyzed. As shown in Figures 2.4 and 2.5, the fiber length increased 30% and the numerical average fiber diameter decreased 40% with stem height over that range. Clearly, the different isolines produce fibers of different cell dimensions. These results suggest that stem height is related to fiber morphology and may provide a selection criteria for fiber length.

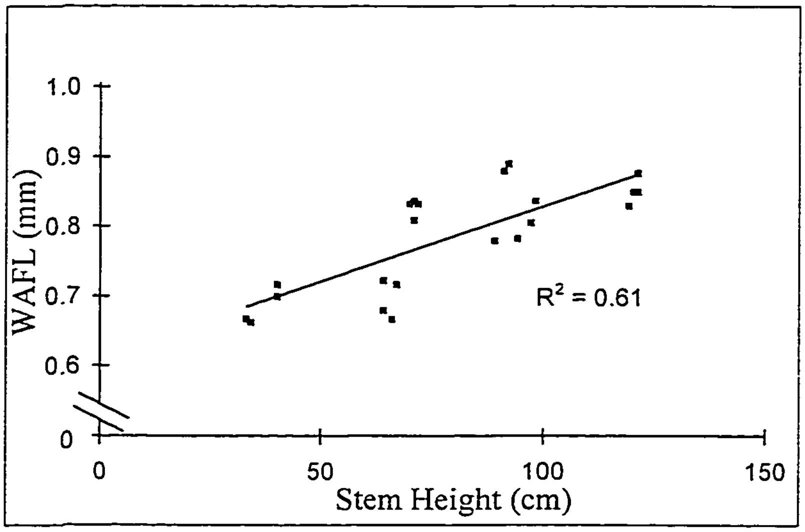


Figure 2.4. Fiber Length of Developmental Cultivars (Kajaani FS-200).

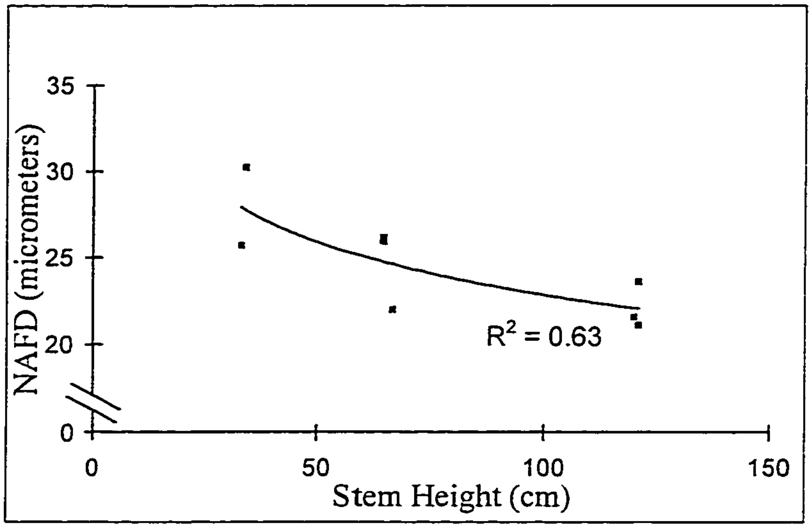


Figure 2.5. Fiber Diameters of Developmental Cultivars

Six of the more common wheat cultivars were examined to assess the affect of irrigation on fiber morphology. These six cultivars represent around half of the winter wheat grown in Washington State (Table 2.1).

Table 2.1. Winter Cultivars in Washington State [Hasslen, 1995]

| ID Number | Cultivar | Percent of Acres |
|---|---|---|
| 1 | Madsen | 26.5 |
| 2 | Stephens | 12.6 |
| 3 | Lewjain | 7.8 |
| 4 | Hatton | 3.0 |
| 5 | Hill 81 | 2.5 |
| 6 | Gene | 0.1 |

Each of the cultivars were sampled on an irrigated and a dryland site. The morphological characteristics of the internodal pulps were examined. Among this limited sampling, the WAFL ranged from 0.73 to 1.04 mm (Kajaani FS-200) and NAFD ranged from 24 to 31 µm. Internodal WAFL values for each pulp are listed in Figure 2.6. Compared to the internodal WAFL listed earlier in Figure 2.1, the lower internodal WAFL reported here was probably due to measurement method. Earlier values were analyzed on a Kajaani FS-100. The values listed in Figure 2.6 are from a Kajaani FS-200 which is better able to

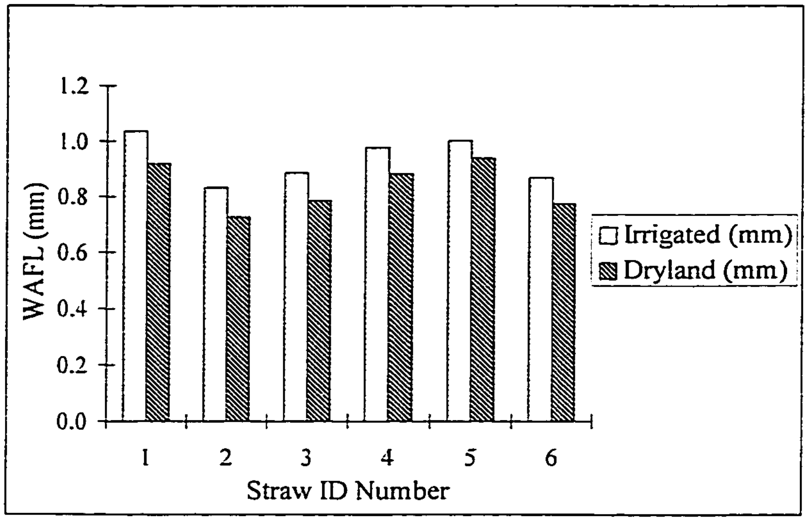


Figure 2.6. Commercial Wheat Fiber Length Comparison (Kajaani FS-200).

determine shorter fibers and fines content thereby resulting in lower average length values [Bichard and Scudamore, 1988].

For the single harvest examined, neither the coarseness nor the NAFD were significantly affected by growing location. However irrigation was found to increase the WAFL of the internodal pulp (Figure 2.6). While the higher WAFL from the irrigated location suggests the potential for stronger paper [Page, 1969], an examination of additional harvests would help describe if the higher fiber length of irrigated locations would be an annual occurrence and will be discussed in Chapter 4.

Variations among commercial cultivars, if any, was also of interest. From the examination of the developmental cultivars, it is clear that fiber properties can be influenced by the genes controlling stem height. However the locally grown commercial cultivars have a narrow range of heights, between 90-105 cm (Figure 2.7). Commercial cultivars have been optimized for disease resistance and grain yield. Shorter cultivars with more of the biomass in the grain are being used [Staniforth, 1979; Misra, 1987]. While these dwarf varieties have higher grain yields, they may eventually decrease the average fiber length of available wheat straw.

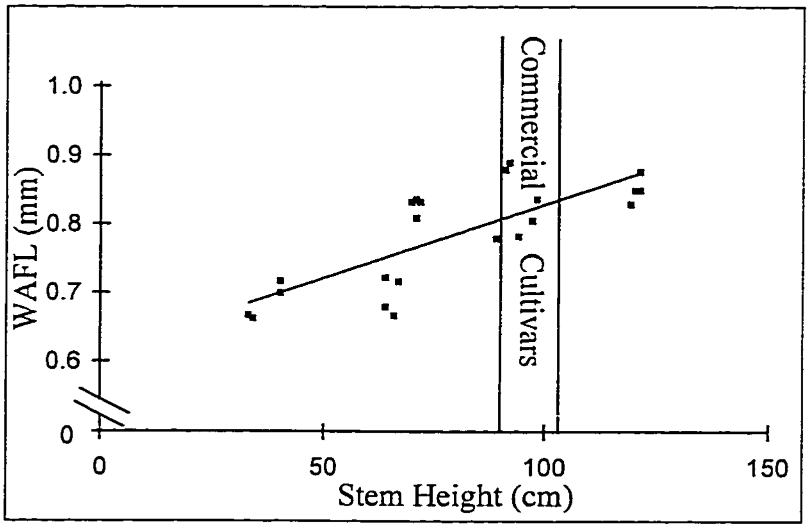


Figure 2.7. WAFL of Developmental Cultivars and the Commercial Cultivar Height Range

Differences are seen in the disease resistance and grain yield of commercial cultivars. Similarly, differences may also exist in the fiber properties of the commercial cultivars. With the limited sampling done in this preliminary study, a statistical evaluation of cultivar variations could not be conducted. A more extensive examination of the influence of plant genetics and growing environment will be discussed in Chapter 4.

The correlations of NAFD are not as easily explained as fiber length. Figure 2.8 shows the NAFD and 95% confidence intervals of the six commercial cultivars at the irrigated and dryland locations. As mentioned earlier, NAFD did not substantially vary by growing location. However, NAFD did vary with cultivar. The Hatton cultivar had a higher NAFD than Hill 81 and Stephens.

The higher NAFD of the Hatton cultivar does not necessarily mean that pulp made from Hatton straw would have wider tracheids. Wheat straw and its resulting pulp contain many cells including tracheids, parenchyma, vessels and epidermal cells. Typical diameters for some of these cells include: tracheids, ~5-24 μm; parenchyma cells, ~58-142 μm; and vessels, ~42-79 μm. When the non-tracheid cells are included in the NAFD determination (Figure 2.9) the NAFD for the sample increases. Differences in NAFD

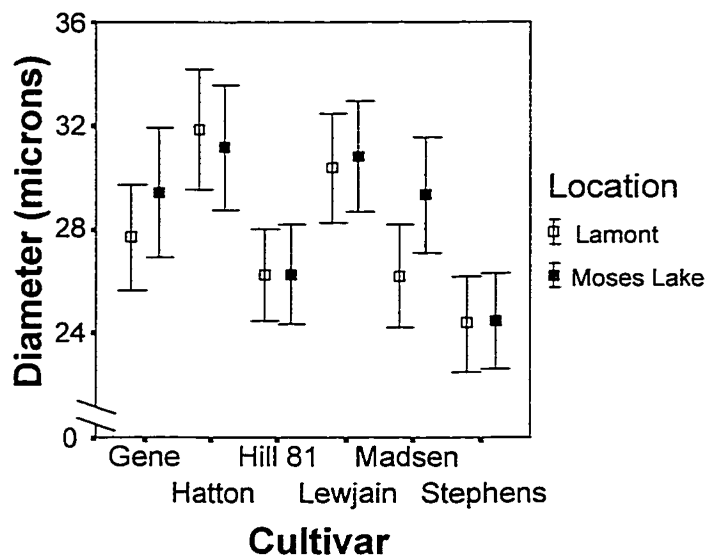


Figure 2.8. Internodal Numerical Average Fiber Diameters
Including All Cells (95% confidence intervals)

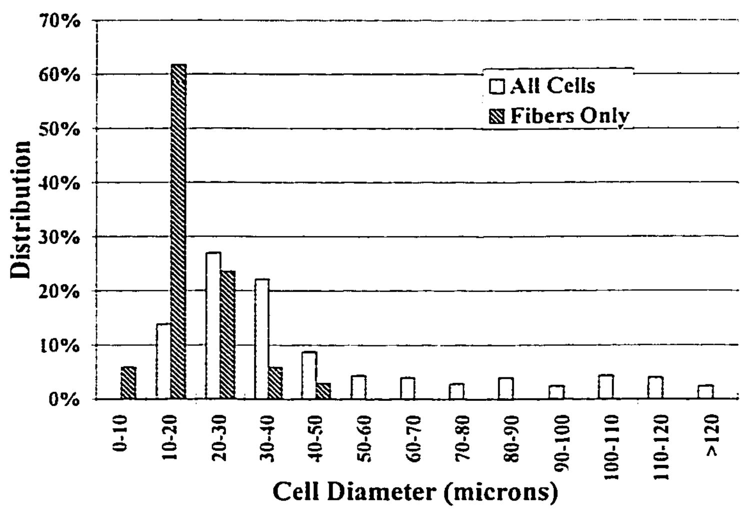


Figure 2.9. Diameter Distributions (1996 Eltan Cultivar)

between Hatton and Hill 81 (Figure 2.10) could be due to differences in tracheid diameters, non-fibrous cell content, or both.

Changes in tracheid diameter and nonfibrous cell content can affect the papermaking properties of the pulp. Differences in fiber dimensions may influence the

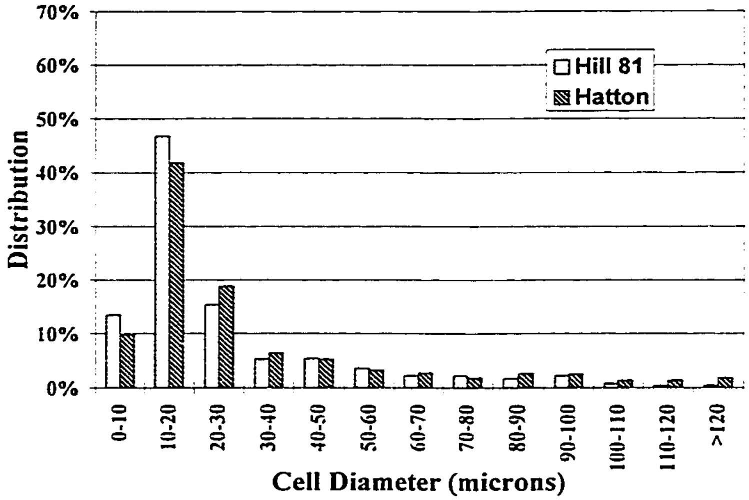


Figure 2.10. Hatton and Hill 81 Cell Diameters

relative bonded area of the sheet and consequently the strength of the sheet [Page, 1969]. The nonfibrous cell content of wheat straw pulp is believed to impair drainage [Watson and Garner, 1997; Misra, 1987]. If the variations in NAFD between Hill 81 and Hatton were due to changes in the nonfibrous cell content, then these pulps may behave differently in papermaking unit operations.

Mansour [1985] described wheat straw pulp as containing 50% tracheids, 15% epidermal cells, 5% vessels, and 30% parenchyma cells. While parenchyma cells are located throughout the internode, a large portion of these cells are located in the pith or innermost parenchymatous layer of the stem (Figure 2.11, #7) [Briggle, 1967].

By comparing photomicrographs, Staniforth [1979] found that the width of the pith layer varied with wheat cultivar and may be used as an identifying characteristic when distinguishing cultivars (Figure 2.12). If other stem dimensions are constant, changes in pith width may correspond with the parenchyma content and therefore the fines and NAFD content of the straw pulp. Microscopic examination of locally grown cultivars may identify similar pith variation in PNW cultivars and, perhaps, between growing locations. Identification of such microscopic variation was beyond the scope of this dissertation but may prove beneficial in the prediction of pulp properties and in the genetic manipulation of the wheat to develop a better papermaking raw material.

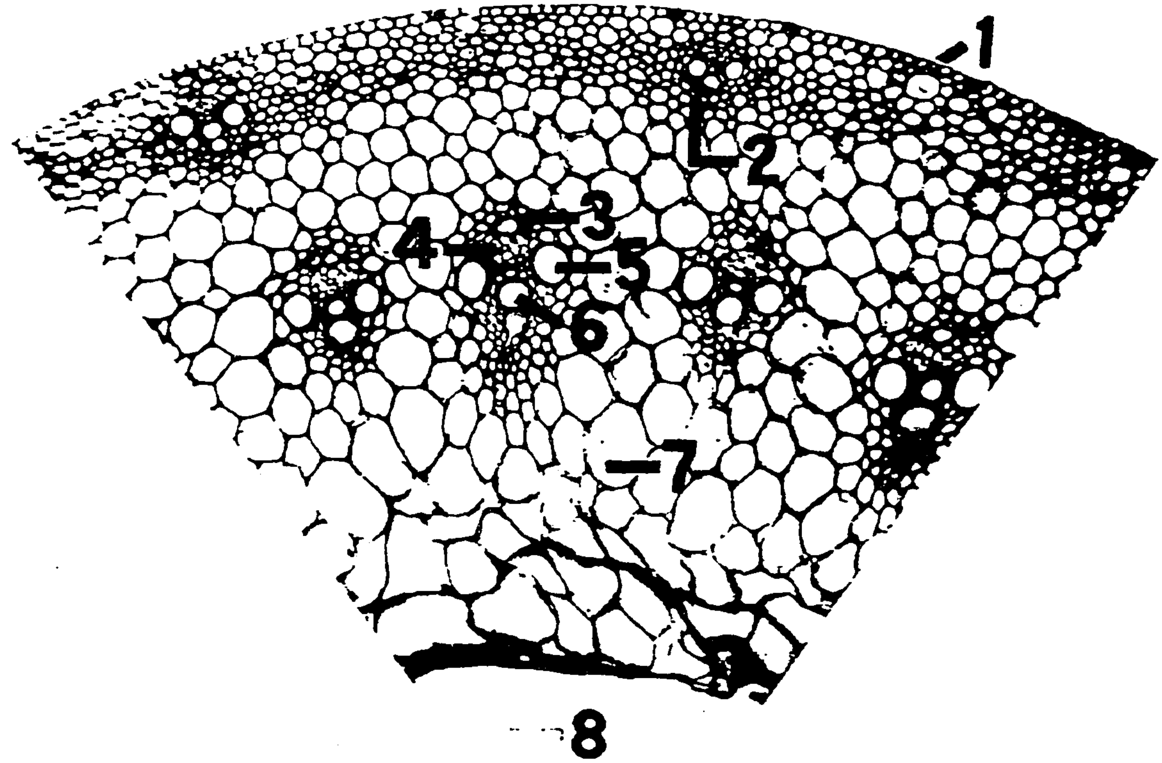


Figure 2.11. Transverse Section of a Wheat Internode Listing Detail of the Tissue: 1. Epidermis. 2 & 3. Mechanical Tissue. 4. Phloem. 5. Metaxylem. 6. Protoxylem. 7. Parenchyma (Pith). 8. Central Cavity of Internode. [from Briggle, 1967].

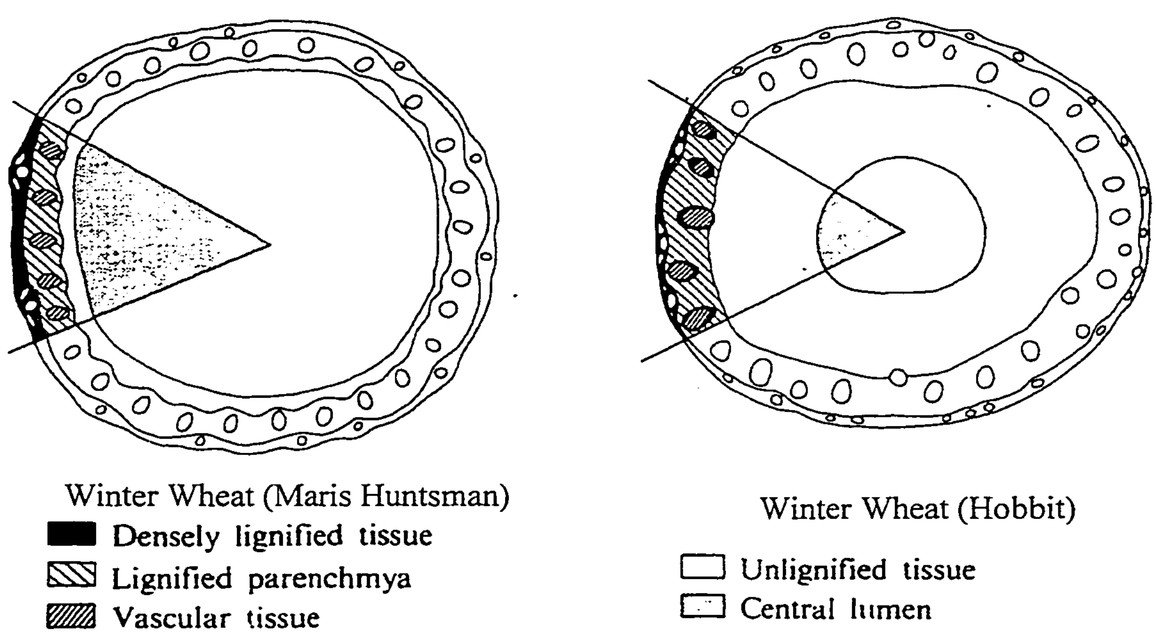


Figure 2.12. Transverse Sections of Wheat Straw Internodes [from Staniforth, 1979]

The fines content of wheat straw pulp may also be reduced by mechanically removing the high-fines containing portions of the internode. Both the epidermal and pith layers are predominantly fines. Mild mechanical action followed by fractionation may permit the removal of these layers. The outermost epidermal layer contains silica and is highly lignified [Staniforth, 1979], and the attached cuticle consists waxy extractives that protect the plant from acid rain [Hua and Xi, 1988]. Removal of this epidermal layer would upgrade the straw resulting in a raw material with a lower fines, lignin, silica, and extractives content.

Pith removal would also reduce the fines content. The innermost pith layer contains little lignin and is predominantly parenchyma cells. The low lignin content may permit this fraction to be easily removed from the rest of the internode. In addition to a lower fines content, pith or epidermal removal may also increase the rate of pulping liquor penetration and result in shorter pulping times.

The mechanism and benefits of epidermal and pith removal were beyond the scope of this dissertation but may be a convenient method of upgrading internodal straw. Determination of a process and quantification of the benefits of such fractionation should be included in future work.

## Conclusions

In this preliminary examination, both genetics and growing conditions affected the fiber morphology of Pacific Northwest wheat straw pulp. Weighted average fiber lengths ranged from 0.66 to 1.04 millimeters (Kajaani FS-200). The taller developmental cultivars (isolines) had higher average fiber lengths. For the harvest year examined, the irrigated straw had a higher average fiber length and may produce a stronger sheet. An examination of additional harvests would determine if these results are annual in nature and elucidate the benefit, if any, of selectively harvesting from irrigated locations. Annual variation will be discussed in Chapter 4.

The limited sampling from this preliminary study prevented the statistical evaluation of the impact of commercial cultivar on fiber length. The average fiber diameter of a wheat straw pulp varied by cultivar suggesting differences in papermaking properties. While these differences in diameter were significant, they did not take into account the within-field or year-to-year variation. A better understanding of these factors would also be warranted before selective harvesting could be justified.

Even though longer fibers are essential for strength, many other factors should be examined to fully understand the implications of adding cereal straw to the Pacific Northwest's raw material supply. Fiber length distributions within the plant and even within the internode may aid in choosing proper harvesting techniques and preprocessing options and is discussed in the next chapter.

# Chapter 3
# Within-the-Plant Variation in Wheat Straw

Wheat straw is a macroscopically heterogeneous papermaking raw material. The structural and chemical variations within the plant allow opportunities for upgrading the straw through improved harvesting practices and preprocessing. The objective of this study was to examine the impact of preprocessing on the most popular Washington State wheat cultivar, Madsen.

The wheat plant consists of leaves (blade and sheath), nodes, internodes (culms) and small amounts of debris (chaff, fines, residual grain) (Figure 1.1, page 4). These different sections have been found to vary in chemical composition and average fiber length (Table 3.1).

Table 3.1. Within-the-Plant Variation of Turkish Wheat Straw [Eroglu and Usta, 1988]

| | Whole Straw | Internodes | Nodes | Leaves and Blades |
|---|---|---|---|---|
| Cellulose, % | 48 | 52.8 | 41.4 | 43.2 |
| Alpha Cellulose, % | 38.9 | 42.2 | 40.5 | 37.1 |
| Pentosans, % | 30.7 | 30.8 | 31.2 | 28.8 |
| Lignin, % | 15.7 | 16.9 | 15.5 | 12.3 |
| Total Ash, % | 4.4 | 2.8 | 7.6 | 11.4 |
| Silica and Silicates, % | 2.6 | 1.7 | 2.1 | 8 |
| Alcohol/Benzene Sol., % | 5.3 | 4.4 | 5.7 | 7.5 |
| Fiber Length, mm | 1.17 | 1.32 | 0.66 | 1.44 |
| Fiber Width, µm | 15.5 | 12.4 | 14.5 | 14.6 |

Removal of the nodes, leaves, and debris has resulted in an upgraded raw material with a higher average fiber length, less fines, less ash (including silica), less protein, and more cellulose [Wisur *et al.*, 1993; Peterson, 1989]. By using this internodal straw instead of whole wheat straw, benefits may be noticed both in the processing of the straw and in product quality through:

- better paper properties [Dhingra *et al.*, 1993; Ali *et al.*, 1991],
- better drainage [Watson and Garner, 1997; Ali *et al.*, 1991],

- higher viscosity [Ali *et al.*, 1991],
- higher yield [Wisur *et al.*, 1993],
- improved pulping and bleaching requirements [Wisur *et al.*, 1993; Dhingra *et al.*, 1993], and
- improved black liquor characteristics [Dhingra *et al.*, 1993].

Removal of the leaves, nodes, and debris from the whole wheat straw can be done through air density fractionation in the field or at a pulp mill. An alternative method of reducing the collection of nodes and leaves proposes harvesting only the top (uppermost) internodal portion from the field [Watson and Garner, 1997].

Wheat varies in height from 0.5 to 1.5 meters and often contains four to six nodes. The length of the internodal sections usually decrease going down the stem [*ibid.*; Misra, 1987]. Different internodes have been found to vary in fiber properties and chemical composition. Kocon and Krutul [1983] found the bottom internodal section to have the highest cellulose content. Internodal cellulose content decreased from the base up the plant with the top internode having the lowest cellulose content.

Zhang *et al.* [1980] conducted an extensive examination of the chemical composition (Table 3.2) and microscopically determined fiber properties (Table 3.3) of a Chinese wheat plant, Triticum aestivum L. V. C. yang No.4. Like Kocon and Krutul, Zhang *et al.* also found a decrease in internodal cellulose content from the bottom to the top of the plant. However, internodal lignin content also decreased going up the plant. Using the ratio of cellulose to lignin as an estimate of yield, the results of Zhang *et al.*

Table 3.2. Microscopically-Determined Tracheid Properties of Chinese Wheat [Zhang *et al.*, 1990]

| | Mass, % | NAFL, mm | WAFL, mm | Width, µm | Cell Wall Thickness, µm |
|---|---|---|---|---|---|
| Top IN* | 7.98 | 1.22 | 1.29 | 13.1 | 3.45 |
| Middle IN | 30.16 | 1.58 | 1.69 | 15.2 | 4.69 |
| Bottom IN | 13.97 | 1.73 | 1.85 | 16.8 | 5.25 |
| Node | 9.31 | 0.67 | 0.82 | 17.9 | 4.05 |
| Sheath | 15.90 | 1.26 | 1.36 | 14.0 | 3.80 |
| Blade | 7.60 | 1.01 | 1.05 | 13.8 | 3.36 |
| Other | 15.08 | 0.73 | 0.80 | 17.2 | 3.41 |

* Internode

Table 3.3. Within-the-Plant Variation in Chemical Composition [*ibid.*]

| | Ash, % | Ethanol-Benzene, % | Cellulose, % | Pentosans, % | Soluble Lignin, % | Klason Lignin, % | Total Lignin, % |
|---|---|---|---|---|---|---|---|
| Top IN* | 7.20 | 4.28 | 45.24 | 24.66 | 2.14 | 18.27 | 20.41 |
| Middle IN | 6.19 | 3.92 | 46.72 | 23.20 | 2.24 | 19.43 | 21.67 |
| Bottom IN | 5.93 | 4.08 | 47.96 | 23.28 | 2.02 | 20.94 | 22.96 |
| Node | 8.69 | 2.56 | 42.55 | 25.42 | 3.24 | 19.98 | 23.22 |
| Leaves | 11.57 | 5.56 | 43.47 | 23.51 | 2.89 | 15.21 | 18.10 |
| Other | 9.82 | 4.93 | 38.49 | 26.03 | 3.48 | 16.02 | 19.50 |
| Total | 8.28 | 4.37 | 44.38 | 24.04 | 2.64 | 18.12 | 20.76 |

* Internode

suggested similar or slightly improved pulping yields for the higher internodal sections of the wheat straw.

When examining the ash content of the internodal sections, Zhang *et al.* found a 21% increase in internodal ash content from the base to the top of the plant. This variation has two major implications. These internodal differences emphasize the importance of consistent and uniform sampling when examining the internodal ash content of wheat straw. Secondly, the different internodal raw materials would vary in ash content. Using the mass balance and ash contents provided by Zhang *et al.* [1990], the ash content of the combined internodal sections would be 6.27%. However, the ash content of the top internodal section was 7.20%. Collecting all of the internodes could provide a raw material with 15% less ash (relative) than the collection of only the top internode.

These chemical and structural differences in the wheat straw plant may provide an opportunity for fractionation to improve the papermaking potential of this raw material. If similar results are found in the locally available wheat straw, then these variations may further support the removal of leaves and nodes before processing. Differences between the internodal sections could also emphasize the importance of uniform internodal sampling and may suggest preferential internodal harvesting techniques for Pacific Northwest wheat straw.

## Experimental

Madsen wheat straw was hand harvested from an irrigated field near Moses Lake, Washington in 1998. Most of the straw plants had three nodes and four internodal sections (Figure 3.1). Originating at each node is a sheath that wraps up the stem and connects the blade. The blades and corresponding sheath at each node were combined and are referred to as the leaf section. Within-the-plant variation in fiber properties and chemical composition were determined.

Twenty gram samples from each plant part were subjected to peracetic acid delignification. This method was less expensive than the sodium chlorite delignification used in Chapter 2. Two hundred milliliters of hydrogen peroxide and two hundred milliliters of glacial acetic acid were added to each sample and allowed to react at 80°C for 72 hours. Additional peroxide was added as needed to maintain liquid level. Due to limited quantity, only five grams of each node location and the bottom internode were delignified. After delignification, the pulp was washed over a 400 mesh screen, disintegrated, and then dewatered. Fiber properties including numerical average fiber length (NAFL), length weighted average fiber length (WAFL), fines content, and coarseness were determined on a Kajaani FS-200. With the Kajaani, fines content was the percent of tracheids, tracheid fragments, and other cells with lengths less than 0.2 millimeters.

To determine chemical composition, samples from each section of the plant were rinsed and dried. Ash and acid insoluble ash (silica) contents of the rinsed samples were determined according to Tappi Methods 211 and 244 respectively. The remaining sample was ground in a Wiley Mill using two millimeter screens to be used later for the determination of extractives, lignin, and carbohydrate content.

Soxhlet extractors were used to remove the acetone extractives of the rinsed strawmeal. Wheat straw contains large quantities of extractable wax from the protective cuticle [Hua & Xi, 1988; Taiz & Zeiger, 1991; Muller, 1960]. The lignin content was calculated by summing the Klason lignin [Effland, 1977] and soluble lignin contents [Tappi Method UM250] of the extractive-free strawmeal. The carbohydrate content of

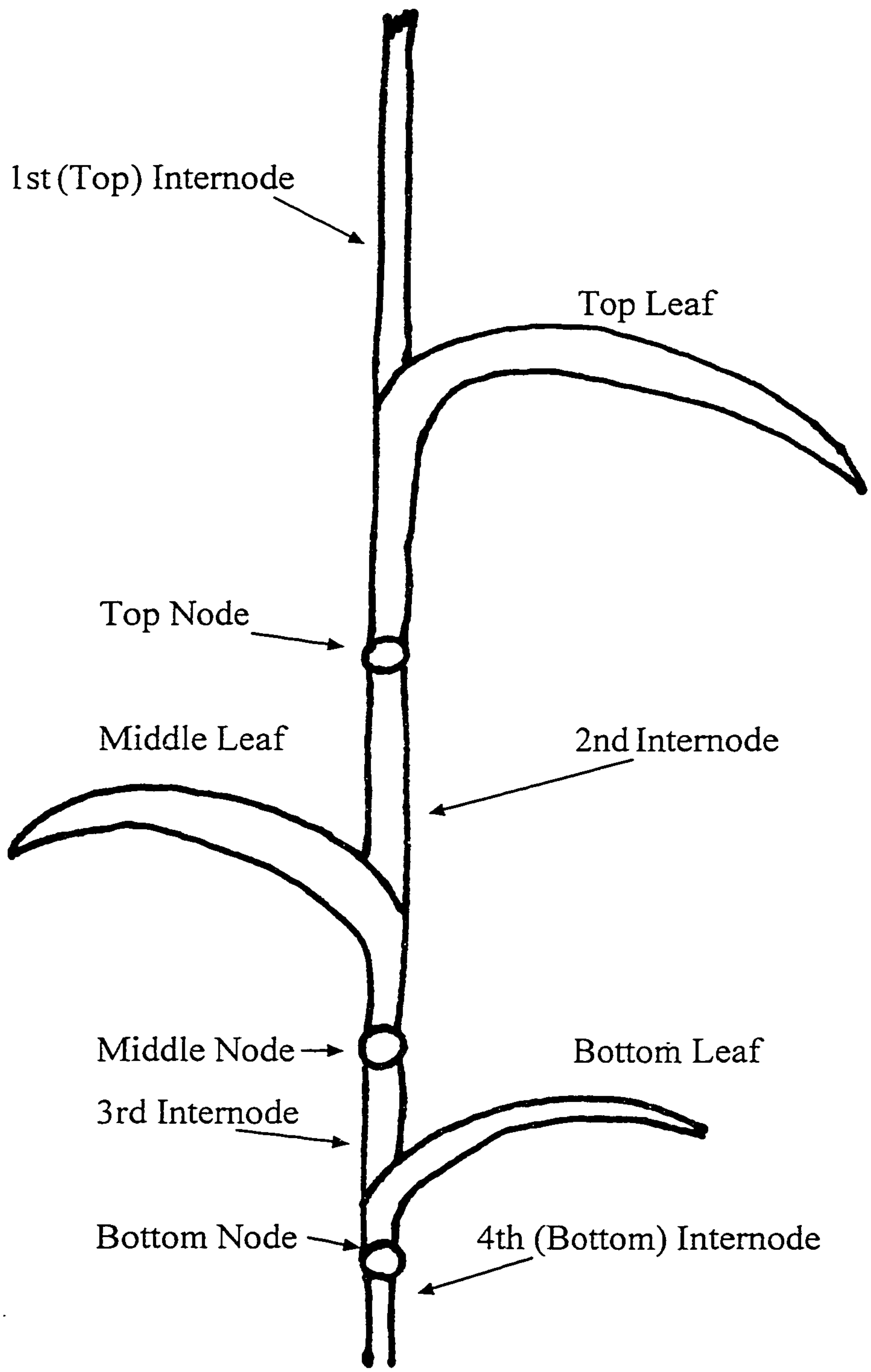


Figure 3.1. Schematic of Irrigated, Madsen Wheat Straw

each section was estimated from the monosaccharide content of the Klason lignin filtrate. A sample of the hydrolyzate was reduced, acetylated and then analyzed on a gas chromatograph using a method adapted from Cao *et al.* [1997] and described in the Appendix. Hemicellulose content was estimated as the sum of the arabinan, xylan, galactan, and mannan content. Glucan content was assumed to originate solely from cellulose [Watson and Garner, 1997].

## Results and Discussion -- Mass Balance

Hand-harvested Madsen wheat straw was comprised predominantly of internodes and leaves (Table 3.4). Within-the-field confidence intervals were determined for the 1996 harvest (Table 3.5). If the 1998 Madsen straw was assumed to have the same within-the-field variation, then the 1998 Madsen straw had a significantly different node, internode, and leaf content than the Madsen straw collected in 1996. Whether these differences were due to annual growing conditions or a difference in agricultural practices (the 1996 samples were grown at the WSU Experimental site and the 1998 straw was collected from a farmer's field) could not be determined from this data. However, if in-field internodal collection is practiced, annual variation in leaf content would have little or no impact on the raw material supply. Table 3.5 also lists the internode, node and leaf contents of other cultivars. Lewjain and Rod had the higher internodal contents for the 1996 harvests. The local cultivars listed in Table 3.5 were hand harvested to preserve complete stems. For industrial processes, the straw will be mechanically harvested which often breaks off leaves [Jeyasingam, 1999]. Baled straw would therefore have a lower

Table 3.4. Mass Balance of Madsen Wheat Straw

| | Mass (%) |
|---|---|
| 1st (Top) Internode | 21.7% |
| 2nd Internode | 18.8% |
| 3rd Internode | 8.0% |
| 4th (Bottom) Internode | 4.6% |
| Top Node | 3.6% |
| Middle Node | 2.8% |
| Bottom Node | 2.7% |
| Top Leaf | 18.5% |
| Middle Leaf | 11.7% |
| Bottom Leaf | 7.5% |
| **Internodes** | **53.2%** |
| **Nodes** | **9.1%** |
| **Leaves** | **37.7%** |

Table 3.5. Wheat Straw Mass Balances

| Wheat Straw | Internode (%) | Node (%) | Leaf & Sheath (%) |
|---|---|---|---|
| Madsen[1] ML[2] 1998 harvest | 53.2 | 9.1 | 37.7 |
| Madsen[1] ML 1996 harvest | 48.1 ± 3.9[3] | 6.3 ± 1.6 | 45.5 ± 4.4 |
| Eltan[1] ML 1996 | 50.5 | 6.7 | 42.8 |
| Stephens[1] ML 1996 | 47.4 | 6.3 | 46.3 |
| Lewjain[1] ML 1996 | 56.0 | 6.8 | 37.2 |
| Cashup[1] ML 1996 | 46.3 | 6.5 | 47.2 |
| Rod[1] ML 1996 | 54.9 | 6.4 | 38.6 |
| Winter [Muller, 1960] | 54.0 | 4.8 | 41.2 |
| Spring [Muller, 1960] | 57.6 | 4.4 | 37.9 |
| Zhang *et al.* [1990] | 52.1 | 9.3 | 23.5 |
| Mansour [1985] | 47.7 | 6.9 | 39.9 |

[1]Pacific Northwest Winter Wheat Cultivar [2]Moses Lake
[3]95% confidence intervals representing within-the-field variation

leaf content and perhaps higher internode content than the hand-harvested straw. Examining mechanically harvested straw would provide more realistic comparisons of cultivars. However, baled straw samples of the different cultivars were not available for this study.

The proportion of each internodal, nodal, and leaf section for the Madsen sampled in 1998 was also determined (Table 3.4). Both the internodal length (Figure 3.2) and the

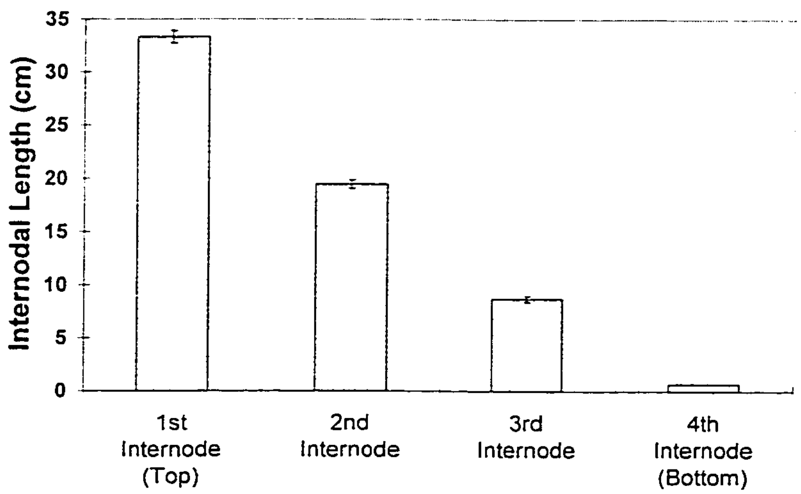


Figure 3.2. Madsen Internodal Lengths (95% confidence intervals)

mass fraction decreased from the top to the base of the plant. Similar results have been reported by Misra [1987] and Watson and Garner [1997]. Having the long top internode may prove advantageous in collection by providing an opportunity for simplified internodal collection. Collection of just this uppermost internode could eliminate the node and leaf removal unit operations in straw preprocessing.

Yield loss is another important factor to examine when comparing internodal collection methods. Based on the Madsen mass balance, collecting all of the internodes would use only 53% of the straw. If the preprocessing of the internodes was done in the field, the unused leaves and nodes could remain on the field for erosion prevention and for retaining organic matter and other nutrients (Mahoney, 1998). However, if all of the straw is baled and shipped to the pulp mill, the 38% yield loss from removing the leaves or 47% loss when removing both nodes and leaves may be less economically feasible. Clearly both the economics balanced with papermaking properties will need to be considered when selecting a raw material source and designing preprocessing methods.

## Results and Discussion -- Fiber Properties

For each of the plant sections, average fiber length, fines content, and coarseness are listed in Table 3.6. Large differences in the fiber properties between the three main botanical components (leaves, nodes, and internodes) were observed. The internodal

Table 3.6. Fiber Properties of Madsen Straw

| Plant Section | NAFL, mm | WAFL, mm | Fines, % | Coarseness, mg/100m |
|---|---|---|---|---|
| 1st (Top) Internode | 0.48 | 0.95 | 34.5 | 8.8 |
| 2nd Internode | 0.61 | 1.26 | 25.4 | 11.5 |
| 3rd Internode | 0.56 | 1.17 | 28.2 | 14.0 |
| 4th (Bottom) Internode | 0.56 | 1.14 | 27.1 | 13.6 |
| Top Node | 0.17 | 0.46 | 71.8 | 13.3 |
| Middle Node | 0.21 | 0.44 | 61.2 | 13.1 |
| Bottom Node | 0.24 | 0.56 | 58.1 | 18.3 |
| Top Leaf | 0.30 | 0.75 | 55.4 | 26.6 |
| Middle Leaf | 0.30 | 0.76 | 57.2 | 23.4 |
| Bottom Leaf | 0.28 | 0.70 | 57.3 | 16.9 |
| Maple (from Chapter 6) | 0.48 | 0.73 | 32.1 | 10.8 |
| Oak (from Chapter 6) | 0.52 | 1.03 | 46.4 | 9.8 |

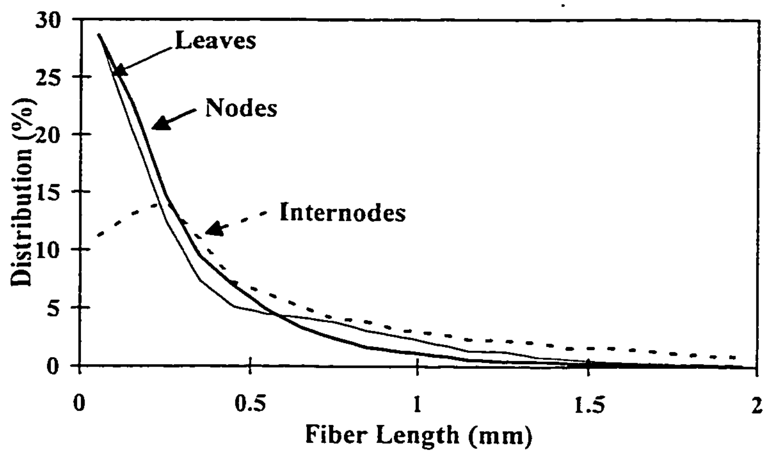


Figure 3.3. Fiber Length Distributions within Wheat

sections had a higher average fiber length and a lower fines content than the nodes and leaves. These variations between internodes, nodes and leaves were also apparent in their fiber length distributions (Figure 3.3). The lower fines content of the internodal pulp could lead to improved drainage. Differences in average fiber length often relate to the strength properties of the pulp [Page, 1969] suggesting that the internodal pulp may produce a stronger sheet than pulp made using the whole plant.

In addition to the variations in fiber properties between the leaves, nodes, and internodes, differences were also observed along the length of the plant. When Zhang *et al.* [1990] microscopically compared the tracheid lengths of the internodal sections, they found longer tracheids at the base (Table 3.2). Their average tracheid length then decreased from the bottom to the top of the plant. While Zhang *et al.* found a clear trend in tracheid length, tracheids only comprise around 35-40% of the cells in wheat straw pulp [Utne and Hegbom, 1992]. Changes in the non-tracheid content along the length of the plant may also impact the processing and product properties of a wheat straw pulp.

For the present study on Madsen straw, all of the plant stem cells were measured and used to determine the averages shown. The different internodal sections did not follow the same trend as found by Zhang *et al.* [1990]. The uppermost internode had the lowest WAFL, and the highest WAFL was found in the second internodal fraction (Table 3.6). These internodal fiber lengths may relate to the vascular bundles in the internodes.

In the wheat internode, tracheid cells are located within vascular bundles (Figure 2.11). These vascular bundles often connect or branch in a node and then go up through the internode to the next node where they will either continue up, branch, or trace to the leaf sheath that comes out at that node [Briggle, 1967]. Of the different leaf sections, the top leaf had more mass (Table 3.4). If this higher mass corresponded to more fiber bundles being present in the second internode and going out to the sheath, such a trend may explain the higher WAFL of the second internode. This hypothesis may also explain the slightly higher WAFL of the third internode when compared to the bottom internode. A detailed microscopic analysis would be useful in verifying this hypothesis by determining the distribution of vascular bundles and their potential impact on WAFL. Different internodal fiber lengths within the plant may also affect the commercial processing of the straw.

Earlier, two approaches to internodal harvesting were suggested, collecting only the top internode or collecting all of the internodes. With the variation in fiber properties between internodes, these two collection methods could result in 25% difference in average fiber length. With other properties were held constant, a 25% decrease in WAFL could decrease pulp tensile strength 10% [Page, 1969]. If this relation is true, then the differences in fiber properties between the two internodal raw materials may be noticeable not only in processing but also in the pure wheat paper and perhaps in blends.

In addition to fiber length, differences in coarseness can also affect straw processing and the properties of the resulting pulp [Seth, 1995; Hatton 1998]. Variations in coarseness may be due to cell wall thickness or fiber diameter [*ibid.*]. Diameters for some of the wheat straw cells were found through microscopic examination to range from ~ 5-24 μm for tracheids, ~58-142 μm for parenchyma, and ~42-79 μm for vessels. If the leaves and nodes contain more parenchyma cells and these cells are wider, then the increased quantity of wider cells could be causing the higher coarseness values for the leaves and nodes. However, cell wall thickness also influences coarseness but was not examined in this study. Comparisons of cell wall thickness or pulp properties would also be useful in determining the implications of the coarseness differences between the nodes, internodes, and leaves.

The lower internodal sections had higher coarseness values than the top internode. This variation correlated with both fines content (Table 3.6) and the tracheid wall thickness reported by Zhang *et al.* [1990]. The lower internodal sections had less fines than the upper internode. If the fines had a higher coarseness than the other cells, then the higher fines content could be increasing pulp coarseness. Thicker cell walls can also increase the coarseness. The cell wall thickness for the Madsen pulp was not determined, but Zhang *et al.* [1990] found the tracheids from the lower internodes to have thicker and wider cell walls than the upper tracheids. Thereby, the tracheids in the lower internode would have a higher coarseness than the upper internode and would contribute to the whole pulp coarseness values.

The impact of these coarseness differences on pulp properties could only be speculated but warrants further investigation. The lower coarseness in the upper internode may result in more collapsible cells. These cells may be easier to refine but may also impair drainage. Comparisons of pulp strength properties would be necessary to quantify the impact of internodal fiber differences on processing and products.

## Results and Discussion -- Chemical Composition

Differences in chemical composition were found both between and within the anatomical parts of the Madsen wheat straw plant. Table 3.7 summarizes the chemical composition for each plant section. Internodes contained more cellulose, less ash, and less extractives than the leaves. This difference in chemical composition between the internodes and leaves was similar to the results of by Muller [1960], Zhang *et al.* [1990], and Eroglu and Usta [1988].

Both ash and extractives are undesirable in a papermaking raw material. The removal of the leaves would benefit pulp processing through reducing the input of these

Table 3.7. Chemical Composition of Madsen Wheat Straw*

| | Extractives, % | Total Lignin, % | Hemicelluloses, % | Cellulose, % | Ash, % | Total, % |
|---|---|---|---|---|---|---|
| 1st (Top) Internode | 2.0 | 20.8 ± 2.7 | 26.8 ± 2.7 | 38.7 ± 0.4 | 6.0 ± 0.1 | 94.4 ± 5.9 |
| 2nd Internode | 2.1 | 21.5 ± 1.0 | 25.3 ± 0.8 | 44.1 ± 1.4 | 4.4 ± 1.0 | 97.4 ± 4.1 |
| 3rd Internode | 1.5 | 23.0 ± 1.9 | 25.3 ± 4.0 | 45.2 ± 6.0 | 3.2 ± 0.3 | 98.2 ± 12.2 |
| 4th (Bottom) Internode | 1.0 | 24.4 ± 0.9 | 24.8 ± 4.0 | 43.1 ± 6.5 | 2.6 ± 0.3 | 96.0 ± 11.7 |
| Top Node | 2.0 | 19.0 | 25.1 | 30.1 | 11.2 | 87.3 |
| Middle Node | 2.1 | 22.6 | 28.2 | 36.8 | 5.9 | 95.5 |
| Bottom Node | 1.3 | 25.1 | 28.1 | 37.9 | 4.3 | 96.7 |
| Top Leaf | 3.7 | 18.7 | 25.9 | 29.4 | 14.9 | 92.6 |
| Middle Leaf | 3.8 | 18.4 | 26.8 | 30.1 | 11.9 | 91.0 |
| Bottom Leaf | 3.1 | 17.8 | 25.4 | 29.6 | 11.8 | 87.7 |
| **INTERNODES** | 1.9 | 21.7 | 25.9 | 42.0 | 4.7 | 96.2 |
| **NODES** | 1.8 | 21.9 | 26.9 | 34.5 | 7.5 | 92.6 |
| **LEAVES** | 3.6 | 18.4 | 26.1 | 29.6 | 13.4 | 91.2 |

* 95% confidence intervals are listed where available

extraneous components. The higher cellulose content of the internodes compared to leaves also suggests an improved pulping yield when the leaves are removed.

In addition to leaf removal, node removal has also been recommended [Ali *et al.*, 1991; Dhingra *et al.*, 1993; Mansour, 1985; Watson and Garner, 1997]. Based on the average composition, nodes have a similar lignin, hemicellulose, and extractives content compared to internodes. However, the nodes also have a higher ash and lower cellulose content than internodes. The lower cellulose content may result in a lower pulping yield; and the higher ash (and fines) content of the nodal fraction would be of no benefit to the pulp mill. Therefore, the removal of the nodal fraction of this Madsen straw may also improve the pulping characteristics of this raw material.

Total mass values determined by the summation of the measured components in Table 3.7 were below 100%. Besides the listed components, wheat straw also contains a significant amount of protein, and the protein content was not measured. Previous studies have found the quantity of protein to vary within the plant with the leaves and nodes containing more than the internodes [Wisur *et al.*, 1993; Billa & Monties, 1995; Dhingra *et al.*, 1993]. If the leaves and nodes contain more protein, then their totals in Table 3.7 should be correspondingly lower. Even though the protein content may be a measurable quantity of the wheat straw, the impact of this component on pulping and processing should predominantly be in yield loss. Therefore the calculated total mass supported the presence of protein especially in the leaves and nodes and supported the potential for higher yield loss from these components.

As was mentioned in the experimental section, the total lignin content was estimated as the sum of the Klason and soluble lignin contents. Arabinan, galactan, mannan, and xylan contents were used to estimate the hemicellulose content, and the wheat straw ash included both silica and an acid soluble fraction. Variations in these components will be discussed in the following sections.

## Lignin

Wheat straw contains less lignin than most woods [Utne & Hegbom, 1992; Ali *et al.*, 1991]. The low lignin content and open structure of this raw material contribute to the rapid delignification often found with wheat straw [*ibid.*]. Lignin contents varied within and along the length of the plant. In this present study, the total lignin contents were not statistically different between the internodes, nodes and leaves (Table 3.8), but the harder to remove, more-condensed Klason lignin contents were higher in the internodes than the leaves. This higher internodal lignin content could hinder

Table 3.8. Madsen Lignin Contents*

| | Klason Lignin, % | Soluble Lignin, % | Total Lignin, % |
|---|---|---|---|
| 1st (Top) Internode | 18.0 ± 2.9 | 2.8 ± 0.8 | 20.8 ± 2.7 |
| 2nd Internode | 19.3 ± 1.1 | 2.1 ± 0.4 | 21.5 ± 1.0 |
| 3rd Internode | 20.9 ± 2.9 | 2.0 ± 1.1 | 23.0 ± 1.9 |
| 4th (Bottom) Internode | 22.3 ± 1.1 | 2.1 ± 0.5 | 24.4 ± 0.9 |
| Top Node | 15.4 | 3.7 | 19.0 |
| Middle Node | 20.0 | 2.6 | 22.6 |
| Bottom Node | 22.2 | 2.9 | 25.1 |
| Top Leaf | 14.1 | 4.7 | 18.7 |
| Middle Leaf | 13.8 | 4.6 | 18.4 |
| Bottom Leaf | 13.8 | 3.9 | 17.8 |
| **INTERNODES** | **19.3** | **2.4** | **21.7** |
| **NODES** | **18.8** | **3.1** | **21.9** |
| **LEAVES** | **14.0** | **4.5** | **18.4** |

* Average and 95% confidence intervals for triplicate determinations

delignification of the internode. Billa and Monties [1995] found similar trends in Klason lignin; and, upon further examination of wheat straw lignin, quantified differences in the lignin structures through the plant. How these differences in lignin structure and content affect delignification has not been examined. However, from a practical point of view, straw mills are able to stop the pulping reactions before the nodes have reached their fiber liberation point [Jeyasingam, 1999; Atchison, 1995]. This practice allows the removal of the fines-containing nodes in the brownstock screens.

The different lignin structures within the plant may also contribute to differences in residual lignin. When pulped under identical conditions, wheat straw fibers had a lower residual lignin content than the wheat straw parenchyma [Zhai and Lee, 1989; Zhang *et al.*, 1988]. The presence of these parenchyma cells lowered brightness and increased bleaching chemical requirements. Therefore, the removal of the higher fines containing portions of the straw (leaves and nodes) may result in reducing chemical consumptions and lower bleaching costs.

In addition to comparing nodes, internodes, and leaves, the variation between the internodal sections was also of interest. While the soluble lignin content did not change between internodes, both the Klason lignin and total lignin content significantly increased from the top to the bottom of the plant. Zhang *et al.* [1990] found a similar trend in the internodal sections of Chinese wheat straw. When examining lignification patterns, Briggle [1967] noted that the parenchyma cells in the lower internodal sections thickened and became lignified. This increased lignification may aid in the structural stability of the plant, but may also influence the pulping and bleaching characteristics of the lower internodes. The impact of these internodal lignin variations may be estimated through a more detailed analysis of the lignin or could be measured through comparisons of pulping and bleaching responses.

## Cellulose

Like lignin, the cellulose content varied between and within the different parts of the wheat plant (Table 3.7). The internodal sections had more cellulose than the leaves.

Both Wisur *et al.* [1993] and Peterson [1989] also found higher cellulose contents in the internodal sections. As mentioned earlier, these increased cellulose contents may be contributing to the higher pulping yields of internodal material when compared to whole straw.

The top internodal section had a lower cellulose content than the lower internode (Table 3.7). This result was unexpected since both Zhang *et al.* [1990] and Kocon and Krutul [1983] reported decreases in cellulose content at each lower internode. Even barley straw has been found to have a higher crude fiber content in the lower internodes [Staniforth, 1979]. With these reported trends in mind, one may speculate that the Madsen straw is either different than other straws or that the analytical technique used to determine the glucose contents of the Madsen straw did not have enough resolving power to measure such differences. The confidence intervals for the cellulose contents of the lower internodes are large and a more precise analytical method may be warranted.

## Hemicellulose

Hemicellulose content and structure are known to affect yield, bleachability, and the specific bond strength of the fibers. While wood contains large quantities of both glucomannan and xylans [McDonough, 1998], arabinoxylan is the predominant hemicellulose found in wheat straw [Timmel, 1957]. This polysaccharide is comprised of a 1-4 linked xylan backbone with arabinose and uronic acid residues attached as single unit side chains [*ibid.*]. Arabinoxylan was the predominant hemicellulose in this straw (Table 3.9).

While the overall hemicellulose content did not significantly change between the sections of the plant, the hemicellulose composition did. The internodal sections had a lower content of arabinose, mannose, and galactose than the leaves and nodes. These differences could be due to varying degrees of substitution within the plant or between different cells (parenchyma, epidermal cells, tracheids, vessels). Even though the different sections of the plant had different hemicellulose contents, the impact of these

Table 3.9. Hemicellulose Content of Madsen Straw

| | Arabinan, % | Xylan, % | Mannan, % | Galactan, % | Hemicelluloses, % |
|---|---|---|---|---|---|
| 1st (Top) Internode | 1.8 ± 0.1* | 24.4 ± 2.6 | 0.2 ± 0.1 | 0.4 ± 0.1 | 26.8 ± 2.7 |
| 2nd Internode | 1.6 ± 0.1 | 23.2 ± 0.8 | 0.2 ± 0.0 | 0.3 ± 0.0 | 25.3 ± 0.8 |
| 3rd Internode | 1.6 ± 0.2 | 23.2 ± 3.6 | 0.2 ± 0.0 | 0.4 ± 0.2 | 25.3 ± 4.0 |
| 4th (Bottom) Internode | 1.6 ± 0.2 | 22.7 ± 3.7 | 0.2 ± 0.0 | 0.4 ± 0.2 | 24.8 ± 4.0 |
| Top Node | 4.7 | 18.6 | 0.7 | 1.1 | 25.1 |
| Middle Node | 4.1 | 22.5 | 0.6 | 1.0 | 28.2 |
| Bottom Node | 3.6 | 23.2 | 0.5 | 0.8 | 28.1 |
| Top Leaf | 3.0 | 21.4 | 0.4 | 1.0 | 25.9 |
| Middle Leaf | 3.4 | 21.9 | 0.5 | 1.1 | 26.8 |
| Bottom Leaf | 3.6 | 20.4 | 0.4 | 1.0 | 25.4 |
| **INTERNODES** | 1.7 | 23.6 | 0.2 | 0.4 | 25.9 |
| **NODES** | 4.2 | 21.2 | 0.6 | 1.0 | 26.9 |
| **LEAVES** | 3.2 | 21.4 | 0.4 | 1.0 | 26.1 |

*95% confidence intervals have been included where available.

variations may be considered minor compared to variations in ash content found within the plant.

## Inorganics

Of the different sections of the plant, the leaves were among the sections with the highest ash content (Table 3.10). Others studies have also found higher ash contents in the wheat straw leaves [Utne and Hegbom, 1982; Zhang *et al.*, 1990; Eroglu and Usta, 1988]. The wheat straw ash was comprised of acid insoluble ash (silica and silicates) and acid soluble ash including, among others, potassium, calcium, and magnesium. Over half of the ash in the leaves was silica. With the potential for silica to interfere with chemical recovery, the removal of this large source of silica may be beneficial. Leaf removal could accomplish a 62% reduction in ash and a 59% decrease in silica at the expense of a 38% loss of plant material.

Table 3.11 lists the estimated silica contents resulting from straw preprocessing. The benefit of leaf removal can be large in terms of silica, but further fractionation of the straw has less of an impact on silica. Therefore, silica removal alone may not be sufficient incentive to install a node removal unit operation.

Table 3.10. Madsen Ash*

| | Ash, % | Silica and Silicates, % | Acid Soluble Inorganics, % |
|---|---|---|---|
| 1st (Top) Internode | 6.0 | 1.7 | 4.3 |
| 2nd Internode | 4.4 | 1.6 | 2.8 |
| 3rd Internode | 3.2 | 1.5 | 1.7 |
| 4th (Bottom) Internode | 2.6 | 1.5 | 1.1 |
| Top Node | 11.2 | 1.8 | 9.4 |
| Middle Node | 5.9 | 1.4 | 4.5 |
| Bottom Node | 4.3 | 1.6 | 2.7 |
| Top Leaf | 14.9 | 8.9 | 6.0 |
| Middle Leaf | 11.9 | 6.5 | 5.4 |
| Bottom Leaf | 11.8 | 6.4 | 5.4 |
| **INTERNODES** | **4.7** | **1.6** | **3.1** |
| **NODES** | **7.5** | **1.6** | **5.9** |
| **LEAVES** | **13.4** | **7.7** | **5.7** |
| **WHOLE STRAW** | **8.2** | **3.9** | **4.3** |

*average of duplicate determinations

Table 3.11. Silica in the Different Fractionated Madsen Straws

| | Silica Content, % | Silica Reduction Compared to Whole Straw, % | Yield After Fractionation, % |
|---|---|---|---|
| Whole Straw | 3.9 | N/A | N/A |
| Straw w/o Leaves | 1.6 | 59 | 62.3 |
| All Internodes | 1.6 | 59 | 53.2 |
| Top Internode | 1.7 | 56 | 21.7/100* |

* only 22% is used but the rest remains on the field and is not a disposal problem

Of the two internodal collection methods, using all of the internodes may result in the lowest silica content. Internodal silica content increased slightly, but significantly, going up the plant (Figure 3.4). Therefore, the collection of just the uppermost internode would also correspond to collecting the internodal fraction with the most silica.

Most of the focus on wheat straw has been on the silica content. However, large amounts of other inorganics are also present. Depending on the fractionation of the straw, the acid soluble ash content could range from 4.3 to 3.1% of the incoming raw material. Acid soluble ash is a combination of many inorganic components (Table 3.12). These results illustrate the anatomical variation in acid soluble ash composition. Note that the leaves are the major contributors of calcium, iron, manganese and phosphorus.

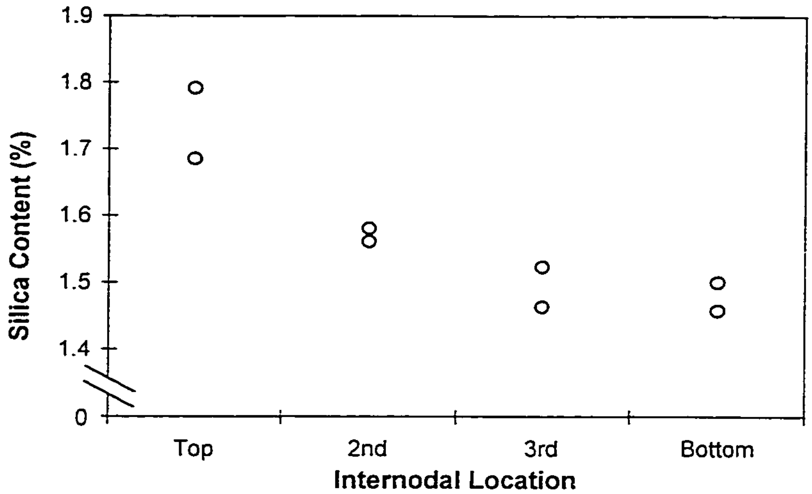


Figure 3.4. Internodal Silica Content (duplicate determinations)

Table 3.12. Acid Soluble Inorganics in 1996 Madsen Grown in Moses Lake

| Metal (ppm) | Internode | Node | Leaf & Sheath |
|---|---|---|---|
| Al | <20 | <20 | 50 |
| Ba | 48 | 68 | 80 |
| Ca | 2000 | 2875 | 7020 |
| Cu | 4 | 6 | 5 |
| Fe | 42 | 38 | 157 |
| K | 23000 | 28500 | 12000 |
| Mg | 1310 | 2350 | 2480 |
| Mn | 15.9 | 16.4 | 82.8 |
| Na | 140 | 362 | 62 |
| P | 830 | 858 | 1300 |
| Sr | 10.0 | 14 | 26.2 |
| Zn | 16 | 24 | 17 |

Removal of the leaves could reduce the load of these non-process elements entering the mill (Table 3.13).

Throughout the wheat straw, potassium was the predominant acid soluble inorganic component. These internodal potassium levels are over one hundred times higher than typically found in wood and may interfere with sodium-based chemical recovery systems [Wong, 1992]. Wong [*ibid.*] speculated that without potassium purges,

Table 3.13. Acid Soluble Ash in the Different Fractionated Madsen Straws

| | Acid Soluble Ash Content, % | Acid Soluble Ash Reduction Compared to Whole Straw, % | Yield After Fractionation, % |
|---|---|---|---|
| Whole Straw | 4.3 | N/A | N/A |
| Straw w/o Leaves | 3.6 | 16 | 62.3 |
| All Internodes | 3.1 | 28 | 53.2 |
| Top Internode | 4.3 | 0 | 21.7 |

a straw pulp mill could accumulate potassium at a rate ten times faster than a corresponding wood mill. In kraft systems, this potassium accumulation could lead to equilibrium shifts, smelt melting point drops, and, in the recovery boiler, lower ash fusion temperatures [Grace, 1985]. While potassium may be undesirable in sodium based pulping systems, other pulping technologies may be more tolerant of this mineral and some potassium based pulping chemistries are being explored [Wong, 1995].

Potassium is based predominantly in the plant stem. Several functional roles for potassium have been proposed including the reduction of lodging (when a the plant falls over), disease resistance, and as a catalytic aid in photosynthesis [Briggle, 1967; Mwrayama, 1964]. Potassium has been found to accumulate in the uppermost internode in rice plants. However, the potassium content was not determined for the different Madsen internodal sections, but higher acid soluble ash contents were observed in the uppermost internode and may correspond to higher potassium concentrations (Figure 3.5).

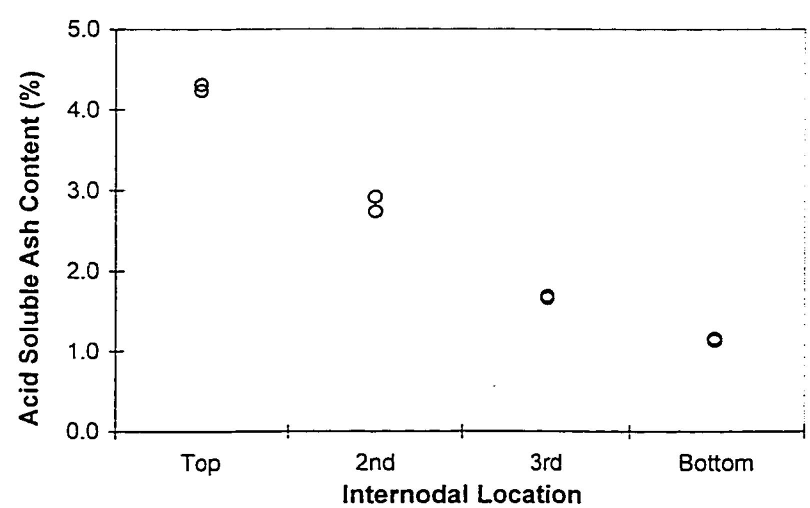


Figure 3.5 Internodal Acid Soluble Ash.

This internodal variation in chemical composition could result in different pulping properties and could have differing impacts on chemical recovery. By knowing these chemical and fiber properties of the fractionation options, more informed decisions may be made in straw selection.

## Conclusions

Wheat straw was found to be a heterogeneous raw material. Differences in fiber properties and chemical composition were seen along the length of the plant and between the leaves, nodes, and internodes. Compared to the node and leaf sections, the internodes had less fines, a higher weighted average fiber length, less ash, and more cellulose. Removal of the nodes and leaves through fractionation could prevent 59% of the silica and 28% of the acid soluble ash from entering the pulp mill. The resulting internodal pulp should have improved drainage, yield, and paper strength properties.

Two methods of collecting internodal straw have been proposed, collecting all of the internodes or collecting just the top internode. These two collection techniques could result in different papermaking raw materials. Compared to a combination of all internodes, the top internode contained more fines, more silica, more acid soluble ash, less cellulose and had a lower average fiber length. This top internodal pulp may have poorer drainage, yield, and paper strength properties. The additional inorganics in the top internode may be an extra burden to chemical recovery systems when compared to collecting all of the internodes.

The heterogeneous nature of Washington-grown wheat straw gives the papermaker several opportunities to upgrade the papermaking potential of this raw material. While these results are based on one local cultivar, similar trends in within-the-plant variation may be found with the other local cultivars. However, the fiber properties and average chemical composition may vary between cultivars and depending on the growing location. Chapter 4 will examine the influence of genetics and growing conditions on the fiber properties of wheat straw pulps.

# Chapter 4
# Genetic and Environmental Influences on the Fiber Properties of Washington State Wheat Straw

Experimental wheat cultivars (isolines) demonstrated a broad range of plant heights that were related to fiber characteristics (Chapter 2). Furthermore, preliminary comparisons of wheat cultivars under the same growing conditions suggested that the fiber properties may be a heritable trait.

Exploitation of wheat straw fiber content in the near future will likely be limited to the use of commercially-grown cultivars. These cultivars have been developed for maximum grain yield, grain quality, and disease resistance. However, little or no information is presently available on differences in fiber morphology among commercial cultivars in Washington State. In the present chapter, six commercial cultivars were examined. Straw from these cultivars were collected for two seasons from three locations. The objective of this study was to define the fiber characteristics (length, fines content, and coarseness) of wheat straw grown in Washington State and to determine the influence of genetic and environmental factors on the papermaking properties of wheat straw fiber. These results will help to quantify the Washington State wheat straw supply in terms of the range in fiber chemistry, morphology, and the potential use in paper and paperboard products.

## Experimental

Six commercial wheat cultivars in Washington State (Table 4.1) were grown at three different locations as part of an ongoing Washington State University Extension program. The wheat grown in Moses Lake was irrigated while Dusty and Pullman are dryland locations.

Table 4.1. Washington Wheat Cultivars [Washington Agric. Statistics Serv., 1998].

| Cultivar | Percent of Wheat Acreage in Washington State |
|---|---|
| Madsen | 23.7 |
| Eltan | 17.5 |
| Stephens | 7.6 |
| Lewjain | 2.5 |
| Rod | 2.3 |
| Cashup | 2.3 |

Pullman is in a more productive, higher-rainfall zone (45-50 cm/year) than Dusty (35-40 cm/year). At each of these three locations, the wheat was grown in a randomized block design with uniform cultivation practices appropriate for the cropping zone and with four replicates of each cultivar at each location. In Dusty, the wheat was planted following bare fallow. Pullman wheat followed spring peas, and Moses Lake wheat followed potatoes. Preplant fertilization in Pullman and Dusty provided 80-90 lb N/acre and 10-12 lb S/acre each year at each location. At Moses Lake, preplant fertilization supplied 100 lb N/acre as ammonium sulfate and an additional 120 lb N/acre through irrigation water as Solution 32. Experimental plot size, row spacing, and, when available, soil analysis are summarized in Table 4.2.

Table 4.2. Plot Size, Row Spacing, and Soil Analysis

| | Dusty (1996,1997) | Moses Lake (1996,1997) | Pullman (1996,1997) |
|---|---|---|---|
| Experimental Plot | 20 ft x 4 ft | 16 ft x 5 ft | 20 ft x 4 ft |
| Row Spacing, in. | 6 | 7 | 6 |
| Soil Analysis | | | |
| Nitrogen*, lb./acre | 271, 374 | not available | 259, 354 |
| Phosphorus, ppm | 33, 33 | " | 31, 33 |
| Potassium, ppm | 340, 516 | " | 350, 223 |
| Soil pH | 6.1, 5.5 | " | 5.4, 5.8 |
| Organic Matter, % | 2.6, 2.0 | " | 3.2, 3.1 |

* sum of tested

To examine the wheat straw, one linear yard samples from these plots were collected at ground level during both the 1996 and 1997 harvests resulting in a total of 144 samples.

During the grain harvest, the mature wheat was collected by hand. Before conversion to fiber by delignification [Franklin, 1945], the head, sheaths, nodes, and leaves were removed. The remaining internodal straw was delignified in peracetic acid for forty-eight hours at 80°C. Fiber properties, including weighted average fiber length (WAFL), fines content, and coarseness, were measured using a Kajaani FS-200 Fiber Analyzer. Additional experimental details are located in Chapter 3. The statistics software, SPSS 7.5, was used to analyze data for the influence of cultivar, location, and year on the fiber properties. Multifactoral analysis of variance was followed by Tukey multiple comparisons when appropriate. Significant differences were defined at the 0.05 level of probability. All of the error bars in this chapter represent 95% confidence intervals.

## Results and Discussion

### Fiber Length

The average fiber lengths of the Washington State wheat straw pulps were comparable to hardwoods. Figure 4.1 shows the 144 Washington wheat samples and some reference hardwoods. The WAFL of the wheat straw pulps examined ranged from 0.8 to 1.2 mm depending on the growing conditions and cultivars. The WAFL of hardwoods also depended on species and growing conditions and have ranged from 0.5 - 1.2 mm [Cisneros *et al.*, 1996; Bichard and Scudamore, 1988]. This similarity supports the potential of wheat straw as a hardwood substitute.

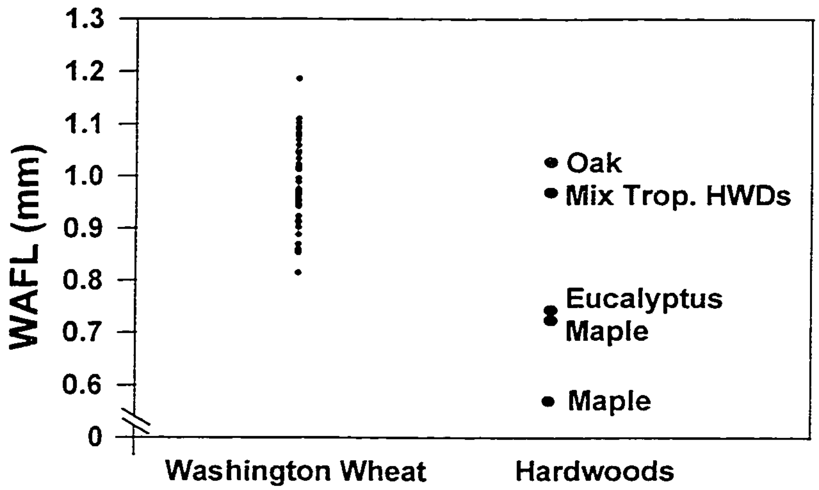


Figure 4.1. Washington Wheat and Hardwood WAFL (Kajaani FS-200) [Bichard and Scudamore, 1988; Tiikkaja, 1994]

However, wheat straw pulps had a wide range of WAFL depending on location, year, and cultivar (Table 4.3). Two significant interactions, location-year and cultivar-location, were present. First the growing location-year interaction will be discussed.

Table 4.3. ANOVA for WAFL

| Variable or Interaction | Significance (P Value) |
|---|---|
| Year | 0.007 |
| Location | 0.000 |
| Cultivar | 0.000 |
| Year * Location | 0.000 |
| Year * Cultivar | 0.539 |
| Location * Cultivar | 0.000 |
| Year * Location * Cultivar | 0.145 |

Extreme values of WAFL ranged from the dryland to irrigated locations. For both harvest years, internodal straw from the irrigated Moses Lake location had a significantly higher WAFL than straw from Dusty (Figure 4.2). Superior fiber lengths from the irrigated location was also observed in the 1994 preliminary study (Chapter 2). In other countries, papermills prefer straw harvested from irrigated locations since this straw produced better paper [Jeyasingam, 1999]. Clearly, irrigated conditions may produce longer fibers which result in stronger pulp [Page, 1969]. However, since other factors (row spacing, fertilization, and soil nutrients) also differed between Dusty and Moses Lake, the exact cause of the superior

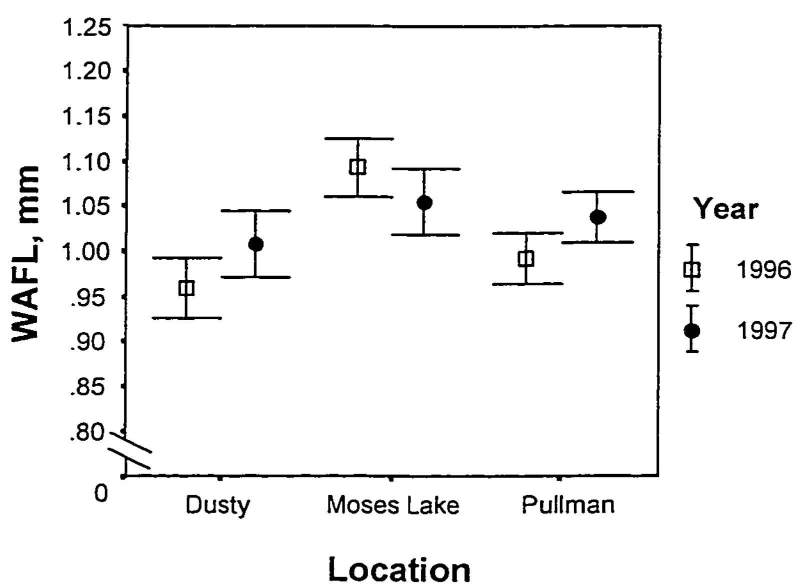


Figure 4.2. WAFL at the Three Locations

WAFL could not be identified. Nevertheless, the higher WAFL of the internodal straw from Moses Lake may lead to a stronger pulp and preferential harvesting from the Moses Lake area.

At all three of the local growing locations, significant year-to-year variations in WAFL were present (Figure 4.2). At both of the dryland sites, the 1997 harvest had a higher average fiber length than the 1996 harvest. The reverse was true for the irrigated site. At Moses Lake, the 1997 straw had a significantly lower WAFL than the 1996.

The cause of these annual variations at each location is not known. Agricultural practices remained relatively constant at each location between years. However, annual differences in precipitation or weather patterns may be influencing WAFL.

Once the harvesting location is selected, the influence of cultivar on fiber properties should not be ignored. The six cultivars responded differently to the three growing locations resulting in a cultivar-location interaction. Tables 4.4a, 4.4b, and 4.4c rank the cultivars at each growing location. Cultivars that do not have statistically different WAFL are in the same group. For example, at Dusty (Table 4.4a), Madsen, Eltan, and Rod do not have significantly different WAFL. However, Lewjain does have a significantly lower WAFL than Madsen. The cultivars rank in a different order at each growing location, but a couple of general trends may be noted. Stephens and Lewjain were among the cultivars with consistently lower WAFL. The Madsen and Eltan cultivars had higher average lengths. However, the only statistically significant difference between cultivars at all three growing locations was between Madsen and Stephens (Figure 4.3). The Madsen cultivar had a higher WAFL than Stephens at all three growing locations, and the average difference was 0.17 mm. This reduction in WAFL could decrease the tensile index of pure wheat paper pulp tensile index around 10% [Page, 1969] and would probably impact the behavior of the wheat straw pulp in a blend. However, the superior average length of the Madsen cultivar is fortuitous in that this cultivar is the most popular wheat grown in Washington State [Washington Agricultural Statistics Service, 1998].

Table 4.4a. Cultivar WAFL (mm) at Dusty

| Cultivar | Group 1 * | Group 2 | Group 3 | Group 4 | Group 5 |
|---|---|---|---|---|---|
| Madsen | 1.08 | | | | |
| Eltan | 1.04 | 1.04 | | | |
| Rod | 1.02 | 1.02 | 1.02 | | |
| Lewjain | | | 0.95 | 0.95 | |
| Cashup | | | | 0.93 | 0.93 |
| Stephens | | | | | 0.87 |

* Cultivars in the same group do not have statistically different WAFL.

Table 4.4b. Cultivar WAFL (mm) at Moses Lake

| Cultivar | Group 1 | Group 2 | Group 3 | Group 4 |
|---|---|---|---|---|
| Madsen | 1.18 | | | |
| Eltan | 1.13 | 1.13 | | |
| Rod | | 1.11 | 1.11 | |
| Cashup | | | 1.04 | 1.04 |
| Lewjain | | | | 1.01 |
| Stephens | | | | 0.98 |

Table 4.4c. Cultivar WAFL (mm) at Pullman

| Cultivar | Group 1 | Group 2 |
|---|---|---|
| Eltan | 1.06 | |
| Cashup | 1.05 | |
| Madsen | 1.05 | |
| Rod | 1.04 | |
| Stephens | | 0.96 |
| Lewjain | | 0.92 |

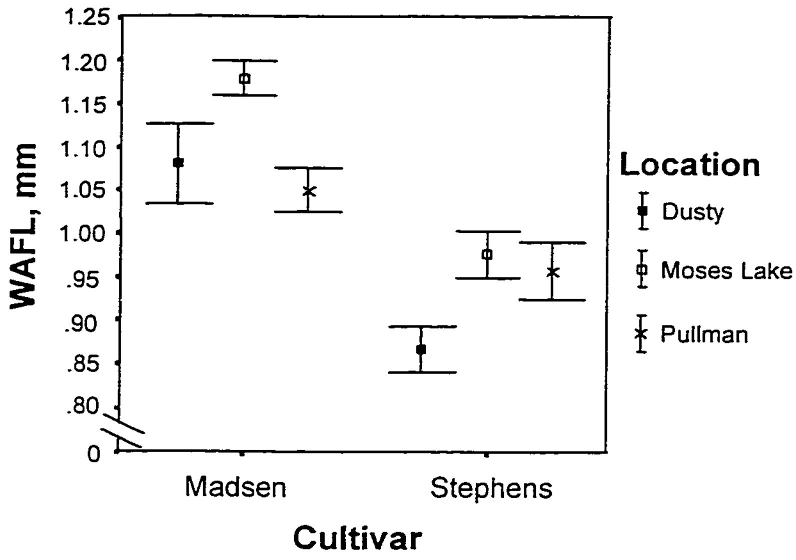


Figure 4.3. Madsen and Stephens WAFL

## Fines Content

The fines contents for the six Washington wheat straw pulps ranged from 32 to 46%. These values were similar to those of Oregon State wheat straw and some hardwood species (Table 4.5).

Table 4.5. Fines Content

| | Fines Content |
|---|---|
| Washington State Wheat | 32-46% |
| Oregon State Wheat [1] | 30-45% |
| Maple | 32% |
| Oak | 46% |
| Eucalyptus [2] | 14% |
| Mixed Tropical Hardwoods [2] | 46% |

Kajaani FS-200, P%
1. Byrd *et al.*, 1997
2. Tiikkaja, 1994

As with WAFL, both location-year and cultivar-location interactions influenced the internodal fines content of the straw. Fines content mirrored WAFL trends. The Madsen cultivar, with the higher WAFL, had a correspondingly and significantly lower fines content than the Stephens cultivar (Figure 4.4). The difference between the Madsen and Stephens cultivars was the only significant difference that held up at all of the growing locations.

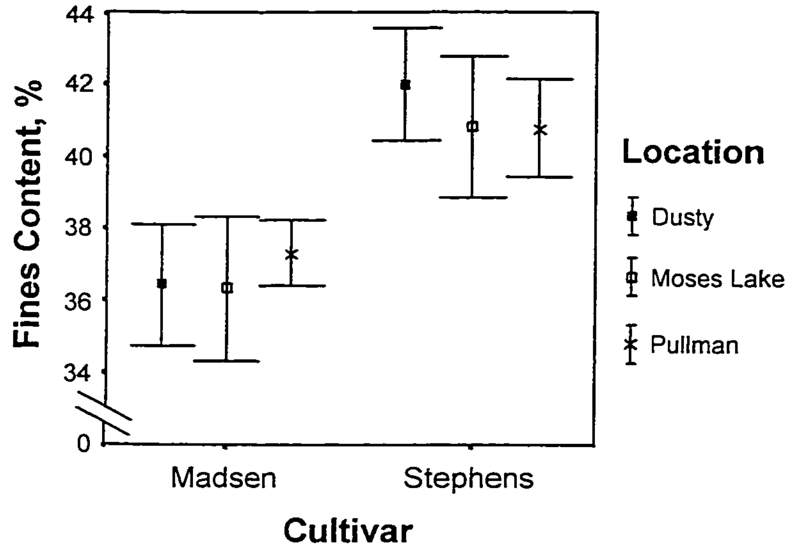


Figure 4.4. Fines Content of the Madsen and Stephens Internodal Pulps

Why the WAFL and fines content of the Madsen cultivar was superior is unknown. Madsen wheat was not found to be consistently taller than Stephens, so another genetic factor may be of influence. In Chapter 2, straw varieties were described that had differing thicknesses in their parenchymatous layers. If the Madsen and Stephens cultivars varied in the thickness of their innermost parenchymatous layers, this difference may help explain the WAFL and fines results. Exploration of this hypothesis could be done with microscopic inspection of Madsen and Stephens internodal cross-sections at similar plant heights. If the parenchyma layer thickness correlates to the fines content, average fiber length, and finally pulp strength properties, then this internodal microscopic examination may prove to be a less labor intensive method of screening for the papermaking properties of straw.

A superior growing location in terms of internodal fines content could not be identified (Figure 4.5). However, the irrigated Moses Lake location had statistically significant annual variation in internodal fines content. If this difference is of the magnitude to noticeably affect pulp drainage or strength properties, then further investigation into the cause could be warranted.

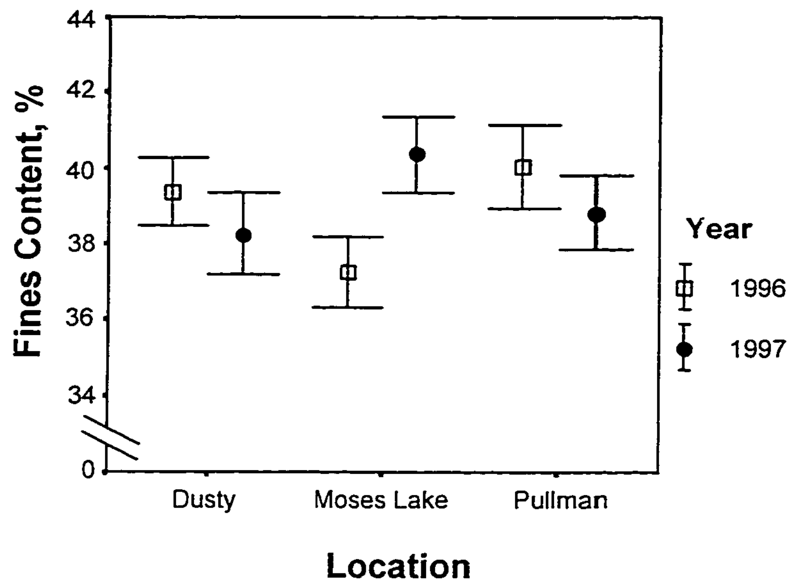


Figure 4.5. Average Fines Contents at the Three Locations in 1996 and 1997

Fines reduction may also be accomplished by other methods. Fines content can be reduced through pulp fractionation (Chapter 6) or by removing the high-fines containing portions of the straw (leaves, nodes, inner and outer layer of the internode) by preprocessing the straw before delignification (Chapter 2 & 3). With these options for fines reduction, many opportunities exist for papermakers to obtain a wheat straw pulp best suited for their process and product.

## Coarseness

Coarseness is a measure of the mass per unit length of fiber. Differences in coarseness could be due to wider fiber diameters or thicker cell walls [Hatton, 1998] (Figure 4.6). An exaggerated example of fibers with the same coarseness would be a fiber with 25 μm diameter and 4 μm wall thickness which would have close to the same coarseness as a fiber with a 45 μm diameter and 2 μm wall thickness. However these two fibers would have different papermaking properties [Rudie, 1998].

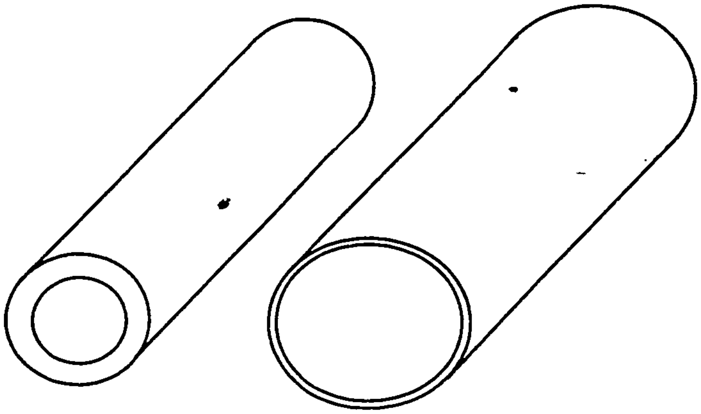

Figure 4.6. Fibers with the Similar Coarseness Values

Wheat straw pulp has many different types of cells including parenchyma, tracheids, epidermal cells, and vessels. Depending which cell is changing coarseness, several scenarios may occur. Tracheids and parenchyma are the predominant cells in wheat straw pulp comprising 50% and 30% of the cells respectively. Since these two cells differ in dimension and role in papermaking, the thickening of their cell walls could have different results. If the cell walls of the parenchyma are becoming thicker, then these cells may be harder to crush during refining; and the resulting pulp may have improved drainage. If the cell walls of the tracheids are thicker, then these tracheids will be less flexible [Gess, 1998]. Such is often the case with softwood fibers. Even in blends, softwood coarseness changes of 15% have significantly affected wet-web strength and papermachine operation [Seth, 1995]. With wheat straw, additional mechanical action may be necessary to fibrillate and collapse the thicker cell-walled tracheids. In the presence of parenchyma cells, this additional mechanical action may crush the parenchyma before noticeably affecting the thick walled-tracheids. This parenchyma crushing may impair drainage and decrease production speeds.

Increases in coarseness due to wider cells could result in an additional set of scenarios. Wider tracheids may collapse easier and result in stronger paper, but wider parenchyma may crush easier and impair drainage.

The Washington State internodal wheat straw pulps had coarseness values ranging from 8 to 14 mg/100m. Both harvest year and growing location influenced the coarseness of the pulp (Figure 4.7).

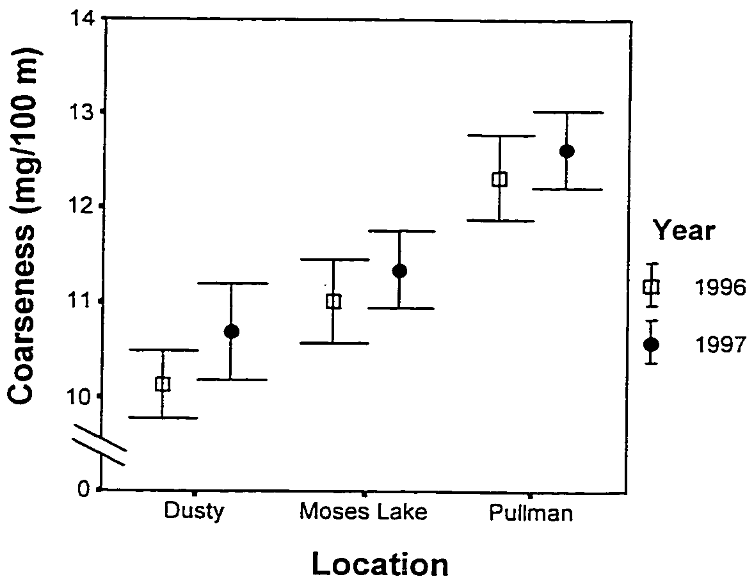


Figure 4.7. Coarseness.

At all three growing locations, the pulp from the 1997 harvests had a higher coarseness than pulp from the 1996 harvests. The reason for this coarseness increase is not obvious. Changes in the weather patterns between 1996 and 1997 are the most likely influencing factors. The examination of additional harvests would aid in determining magnitude of annual variation in coarseness.

The range in internodal wheat straw coarseness between growing seasons (4%) and between growing locations (20%) was statistically significant but less than that found in wood. For example, earlywood fibers have half the coarseness of latewood fibers [Rueckert & Rudie, 1995]; and the coarseness variation between growing locations for a tree can be 20% [Rose & Sultze, 1967].

The Pullman straw was 20% coarser than straw grown in Dusty. Both Pullman and Dusty are dryland locations, but Pullman does receive around 30% more water than Dusty. However, the irrigated Moses Lake wheat gets more water than both of the dryland locations. So, something besides watering technique may be influencing coarseness. The identification of the growing condition influencing coarseness would be of long term interest and may be identified through the examination of additional growing locations. Even without knowing the cause of the coarseness variation, selectively harvesting straw may be a mechanism of collecting the best suited raw material for a paper product.

To fully understand the impact of these coarseness differences, the straws should be further analyzed. The most direct approach would be to pulp both straws and compare their strength and drainage properties through a refining curves. Microscopic examination of the cell widths and cell wall thicknesses would identify the changing factor and permit monitoring in different pulps.

## Conclusions

Washington State wheat straw pulp was found to have similar length-weighted average fiber lengths (WAFL) and fines content to hardwood pulps. These similarities suggest the possible use of wheat straw pulp as a hardwood substitute.

Both environmental factors and cultivar influenced the fiber characteristics of the straw pulps. The Madsen cultivar consistently had higher average fiber lengths than the Stephens cultivar. Changing growing locations also influenced the fiber length. The WAFL of a cultivar grown in irrigated Moses Lake fields was an average of 0.09 mm longer than those grown in the dryland Dusty location. Differences in WAFL between the 1996 and 1997 harvests were not of the same magnitude as the cultivar and location differences. However, with only two years of samples, the potential annual variation on fiber length and the influencing factors could not be determined.

The fines content of the wheat straw was comparable to hardwoods. Chapter 6 will discuss the differences in structure and papermaking impact between wheat straw and hardwood fines.

The predominant influencing factor for wheat straw fiber coarseness was growing location. The difference in coarseness was large with Pullman straw having a 20% higher coarseness than straw grown in Dusty. The influence of these coarseness differences was not rigorously determined. Identification of the mechanism of change would be needed to correlate these coarseness changes to papermaking properties. Having this better understanding of the environmental influences on coarseness may lead to pulps with superior drainage and strength properties.

With cultivar and growing location both influencing the fiber properties of wheat straw pulp, selective straw sourcing should allow the acquisition of pulps better suited for a papermaking product. In addition to fiber properties, other factors are also of importance. The chemical composition including silica content and yield would also be of interest when making informed decisions for selective harvesting. These factors will be discussed in Chapter 5.

# Chapter 5
# Genetic and Environmental Influences on the Chemical Composition of Washington State Wheat Straw

Fiber properties of Washington-grown wheat straw were influenced by growing conditions and cultivar. The objectives of the present study was to determine if the chemical composition of the straw. Differences in chemical composition affect pulping yield, pulp properties, and the amount of non-process elements, like silica and potassium, entering the pulp mill.

## Experimental

Using the same 144 straw samples examined in Chapter 4, six commercial cultivars grown at three locations were compared over two harvest years. For the carbohydrate and lignin content determinations, the leaves and sheaths were removed from whole stems. The remaining node and internode sections were combined and ground in a Wiley Mill (2 mm screen) before lignin and carbohydrate determinations. Chemical composition was determined using a method similar to that described in Chapter 3. The only difference was in the removal of extractives. Since extractives are a minor component of the straw and their removal is time consuming, extractives were not removed prior to the Klason lignin determination. Using extractive containing strawmeal has not been found to alter lignin and carbohydrate results [Bischo, 1997].

In Chapter 3, the leaves and nodes were found to have high ash contents. The possible contamination of the nodes with attached leafy sheaths could lead to erroneously high ash numbers. To eliminate the possibility of this error, only internodal inorganics were determined. Whole stems were selected, and their leaves, sheaths and nodes were discarded. The remaining internodal sections were combined, rinsed (to remove external contaminants), and then analyzed as described in Chapter 3.

The cellulose, hemicellulose, lignin, and inorganics contents of the straw samples are discussed below. As in earlier chapters, significant differences were defined at the 0.05 level of probability; and all error bars represent 95% confidence intervals.

## Results & Discussion--Lignin and Carbohydrates

Chemical pulping processes remove most of the lignin and some carbohydrates resulting in carbohydrate-rich fibers. Differences in either the carbohydrate or lignin content of a raw material will influence pulping yield. This section will describe the carbohydrate (including hemicellulose and cellulose) and lignin contents of the straw.

The cellulose content was not dependent on cultivar; however, annual variation did occur at one growing location. For some reason, the straw from Dusty in 1996 had a lower cellulose content than most of the other straw samples (Figure 5.1) The cellulose contents from the other five harvests were not statistically different from each other.

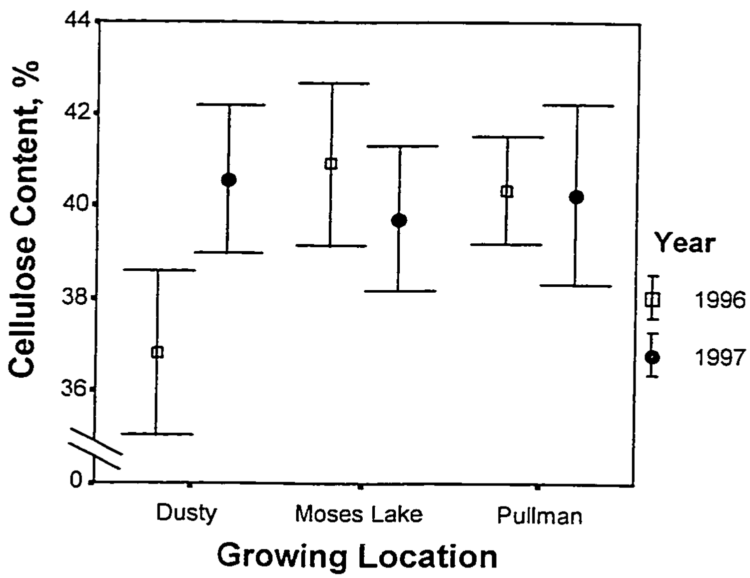


Figure 5.1. Cellulose Content by Growing Location and Harvest Year

The reason for the differences between growing years in Dusty is not clear. This lower cellulose content may lead to a lower pulping yield, but cellulose content is not the only factor that influences pulping yield. The removal of lignin is one of the primary

Table 5.1. Factors Influencing the Lignin Content of Commercial Wheat Straw

| | Klason Lignin | Soluble Lignin | Total Lignin |
|---|---|---|---|
| Location | S* | S | S |
| Cultivar | NS | NS | NS |
| Year | NS | NS | S |
| Location x Year | S | S | S |
| Location x Cultivar | S | NS | NS |
| Cultivar x Year | S | NS | NS |
| Location x Cultivar x Year | NS | NS | NS |

*S = Significant, NS = Not Significant

functions of the pulping process, and differences in lignin content may also affect the yield of a straw.

The total lignin content of each straw sample was estimated as the sum of the Klason lignin and soluble lignin contents. Significant interactions for the lignin contents are listed in Table 5.1. The 1997 Moses Lake straw had a significantly lower total lignin content than 1996 Moses Lake, 1996 Pullman, and both Dusty straw samples (Figure 5.2). Since a lower lignin content straw may be easier to pulp and have a higher yield, the possible agricultural and natural factors that influence lignin content warrant future investigation.

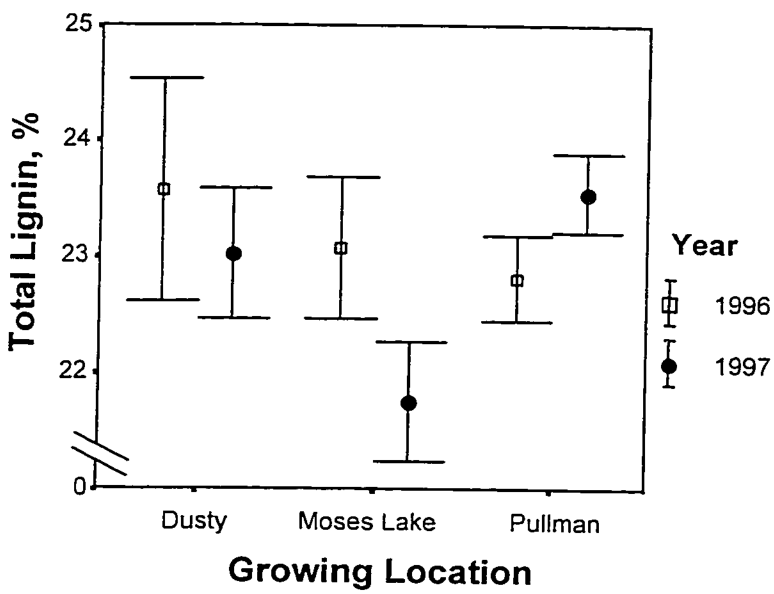


Figure 5.2. Total Lignin Contents

Since the total lignin is the sum of Klason lignin and soluble lignin, the influence of these two values may aid in better describing the straw. The soluble lignin is often made of the lower molecular weight lignin fragments. This portion of the total lignin was low in content, 2-3% of the straw; and, compared to the Klason lignin, should be easier to remove during pulping. Therefore, the impact of the Klason lignin content was of more interest. While both cultivar-growing location and cultivar-year interactions were statistically significant for Klason lignin, neither of these results identified clear advantages in selective cultivar harvest. On the other hand, the three growing locations had interesting Klason lignin results.

At the Moses Lake location, the 1997 harvest had a lower Klason lignin content than the 1996 harvest (Figure 5.3). Similar trends were not found at the dryland locations. In fact, at both Dusty and Pullman, the Klason lignin content of the straw did not significantly change between harvest years.

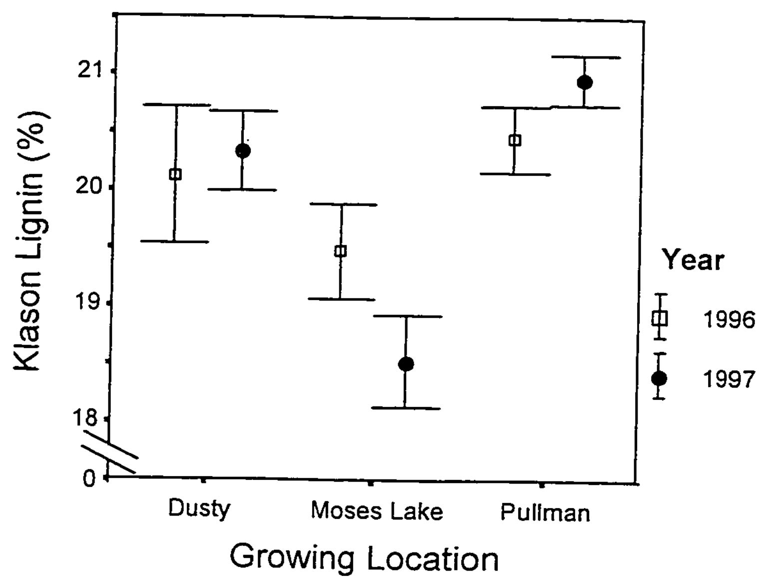


Figure 5.3. Klason Lignin

The identification of the reasons for the Moses Lake lignin content differences would be of interest to papermakers but could not be identified in this study. If the cellulose content remained constant, the lower lignin content may result in higher pulping yields. Therefore, the eventual identification of the reason for the lower Klason lignin content of the 1997 Moses Lake straw, may aid in the development of agricultural practices resulting in lower lignin content.

As mentioned earlier, both cellulose and lignin contents influence yield. In the long term, adjusting agricultural practices to increase cellulose content while decreasing lignin content would be of interest. For the short term, the pulping yield of currently available straw should be examined. Ideally, actual pulping experiments would be preferred. With both sample and time being limited, pulping yield was estimated as the ratio of cellulose to Klason lignin content (Figure 5.4). For this ratio, only the location-year interaction was statistically significant. Even with this interaction, the Tukey post-hoc analysis did not find statistically significant differences between harvest years at any of the growing locations. Therefore, even with the annual differences in cellulose content in Dusty or lignin content in Moses Lake, these may not be of a magnitude to significantly affect pulping yields. However, actual pulping experiments and additional harvests would be needed to verify this hypothesis.

Even though the cellulose to Klason lignin ratio did not predict annual differences, yield variation between locations was predicted. The Moses Lake straw was among those with higher cellulose to Klason lignin ratios (Figure 5.4) suggesting higher yields from Moses Lake straw. The influence of irrigation on cellulose to Klason lignin ratio is interesting to note. Jeyasingam [1999] observed that straw mills prefer wheat straw from irrigated locations due to better pulp properties and yield. Jackson [1977] also reported higher fiber yields from irrigated straw. The Washington straw does not seem to contradict this statement. However, pulping studies comparing straw from the three growing locations would be warranted before definitive statements could be made about pulping yields.

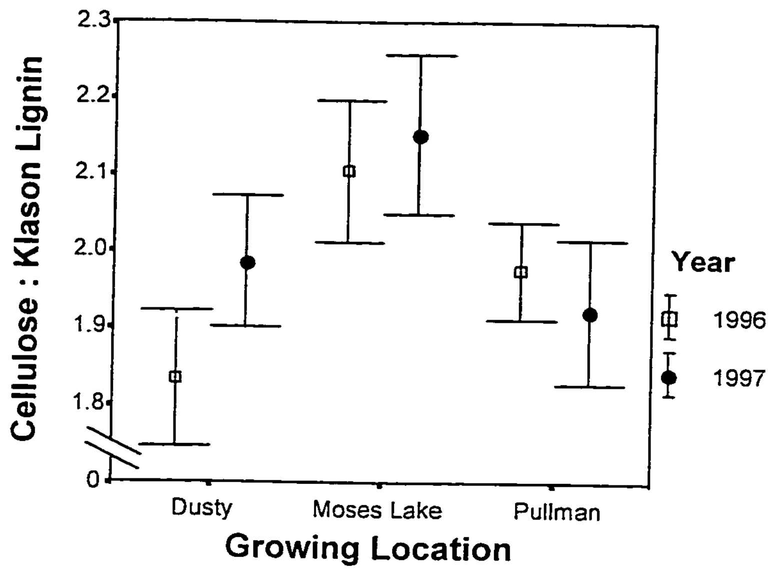


Figure 5.4. Cellulose to Klason Lignin Ratios

Compared to woods, wheat straw contains more hemicelluloses. The hemicellulose content of the locally collected straw ranged from 19 to 28% and was influenced by a growing location - year interaction. The only location with significant annual variation was Dusty (Figure 5.5). If annual variation in the hemicellulose content

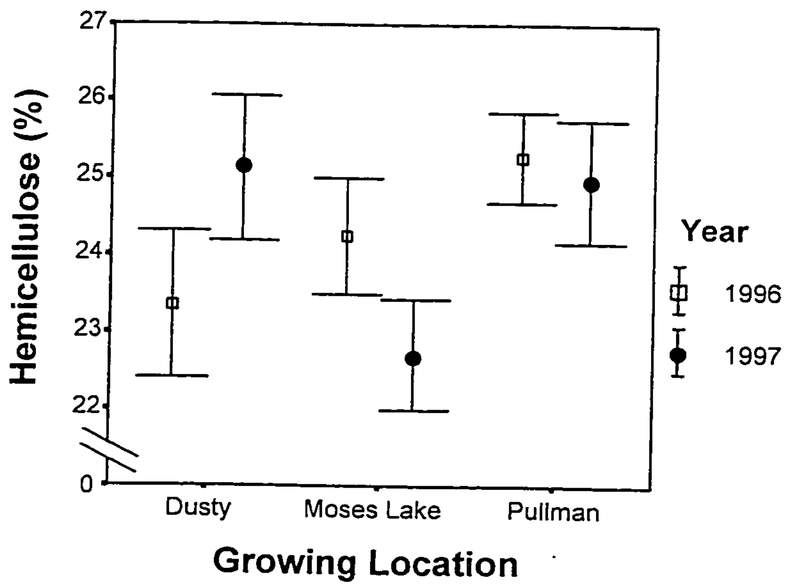


Figure 5.5. Hemicellulose Contents

is regularly found at some locations, then the position of hemicellulose within the plant and impact of these differences would be of interest. The higher hemicellulose content in the 1996 Dusty when compared to the 1997 Dusty did not correlate with a difference in fines content (Chapter 4), and therefore may not be related to differences in amount of smaller cells, like parenchyma and epidermal cells. Hemicellulose content changes could be in the cells thereby influencing their papermaking characteristics.

While the impact of wheat straw hemicellulose content has not been extensively examined, the ash content of wheat straw has been known to cause problems in chemical recovery (Chapter 1). Due to the known impact of inorganics on processing, a few studies have correlated mineral uptake to growing conditions [Casey, 1980; Staniforth, 1979; Eroglu & Deniz, 1993]. These results will be compared to the Washington State findings.

Table 5.2 lists the significant factors on the wheat straw inorganic content. These interactions may present opportunities for selective harvesting by location and by cultivar. The ash content of a sample is the sum of the acid soluble and acid insoluble (silica) contents of the internodes. When comparing ash contents (Figure 5.6) the Moses Lake straw had higher internodal ash content than the dryland sites. There was even some variation between dryland harvests with the 1997 Pullman internodal straw containing more ash than the Dusty samples. Yearly variation in ash content was found with the

Table 5.2. Factors Influencing the Inorganic Content of Commercial Wheat Straw

| | Total Ash | Acid Insoluble Ash (Silica) | Acid Soluble Ash |
|---|---|---|---|
| Location | S* | S | S |
| Cultivar | S | S | S |
| Year | S | S | S |
| Location x Year | S | S | S |
| Location x Cultivar | S | S | NS |
| Cultivar x Year | NS | NS | NS |
| Location x Cultivar x Year | NS | NS | NS |

*S = Significant, NS = Not Significant

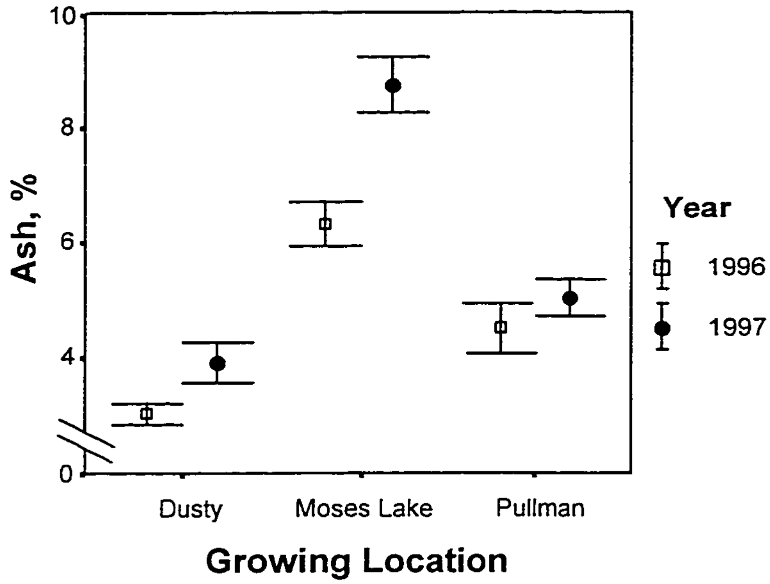


Figure 5.6. Internodal Ash Content at Each Growing Location

Dusty and Moses Lake harvests. Once again agricultural practices and/or environmental factors could be influencing the mineral uptake. Rather than spending too much time on ash contents, it may be useful to the papermaker to examine the minerals of interest.

Jackson [1977] discussed studies where irrigated straw had lower silica contents than dryland sites which differed from the results on Washington wheat straw (Figure 5.7). Instead, the straw from Dusty, a dryland location, was lower in internodal silica content than Moses Lake and Pullman; and it varied from year to year. Perhaps sampling techniques differed between the Washington wheat and that reported by Jackson. Jackson's analysis did not state if leaves were included. A lower leaf content (from growing conditions or harvesting method) could result in a lower wheat straw silica content. Another possibility is that wheat straw silica content is related more to soil and fertilization than to watering practices.

Soil clay contents have often been correlated to the silica content of a straw. Straw from more humus or heavy soils have higher silica contents than the straw grown in light, sandy soils [Muller, 1960; Casey 1980]. All three of the investigated Washington growing locations had relatively light soils. However the soil in Pullman is

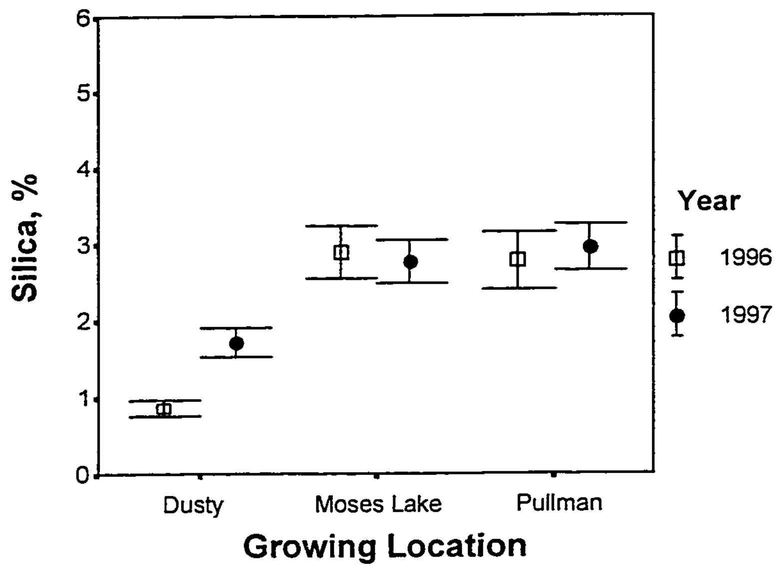


Figure 5.7. Silica Contents at Each Growing Location

heavier than Dusty which is heavier than Moses Lake. Since these silica results do not correlate with the general trends for local soil conditions, once again, other factors must be of influence.

Once a harvesting location has been selected, paying attention to the harvested cultivar could reduce raw material variability. Significant differences were not found among the Dusty grown cultivars, but when these same cultivars were grown in Pullman or Moses Lake, they contained different amounts of internodal silica (Figure 5.8). Similarly, Mueller [1960] found changes in silica content between cultivars grown in same soil conditions. These differences in silica uptake could lead to process upsets when switching raw materials. For example, the silica contents of Pullman grown Lewjain internodal straw (4.2% silica) was almost twice as high as the Pullman grown Cashup internodal straw (2.3% silica). For silica sensitive pulping or chemical recovery systems, sudden unexpected increases in silica may shift equilibriums and result in scaling. Mixing straw supplies and monitoring the incoming cultivars are two approaches that may benefit silica sensitive mills.

Acid soluble inorganics upset some pulping and chemical recovery chemistries [Wong, 1989]. The internodal acid soluble ash content of local straw was found to be

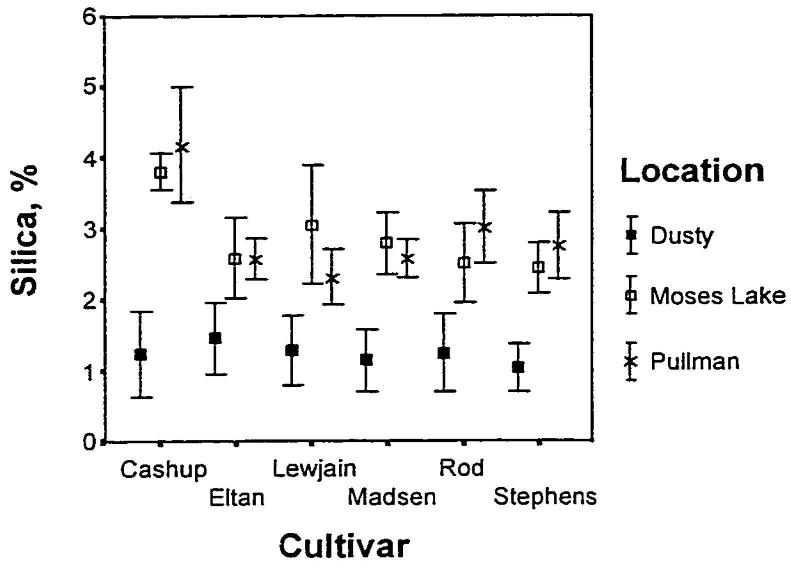


Figure 5.8. Internodal Silica of the Cultivars.

dependent on both cultivar and a location-year interaction. The reason for the location-year interaction (Figure 5.9) is not obvious but is likely to relate to soil conditions and fertilizer application [Staniforth, 1979]. In Moses Lake, wheat followed potatoes which are heavily fertilized with potassium. This is a possible explanation for the higher acid soluble ash contents of the Moses Lake internodal straw.

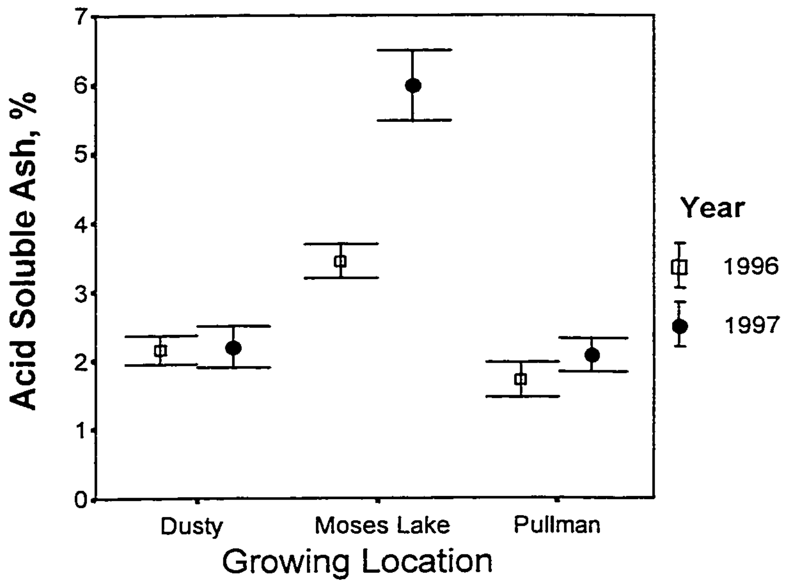


Figure 5.9. Acid Soluble Ash Contents at Each Location

Several acid soluble inorganics are essential for the wheat plant. These nutrients can be divided into macronutrients (nitrogen, phosphorus, potassium, sulfur, calcium, and magnesium) and micronutrients (iron, manganese, boron, zinc, copper, molybdenum and chlorine) [Lamb, 1967]. Most all of these nutrients are found in the wheat straw internode. Table 5.3 lists the acid soluble inorganics content of the 1998 Moses Lake

Table 5.3 Madsen Internodal Inorganics from Moses Lake in 1998 (average of four within the field samples)

| Metal | Average, mg/kg | Standard Deviation, mg/kg |
|---|---|---|
| Al[1] | 70 | 36 |
| Ba | 15.1 | 1.0 |
| Ca | 398 | 37 |
| Cu | 1.8 | 0.5 |
| Fe[1] | 92 | 37 |
| K | 10850 | 880 |
| Mg[1] | 222 | 24 |
| Mn[1] | 44.8 | 2.5 |
| Na | 78 | 38 |
| P | 640 | 120 |
| Sr | 2.8 | 0.6 |
| Zn | 8.7 | 2.7 |

[1]Average of 3 within the field samples

Madsen straw. Potassium was found to be the predominant metal in the 1998 Madsen internodal acid soluble ash. Similarly, the acid soluble ash of the 1996 and 1997 harvests would be predominantly potassium; and the higher acid soluble ash contents of the Moses Lake harvests is likely from increased potassium content. Running a metals analysis on these samples would be necessary to confirm which mineral or minerals are more abundant in the Moses Lake wheat straw. Identification of this mineral will help predict mill impact. If the Moses Lake straw contains a higher quantity of a process element (like sodium in a sodium-based pulping process or potassium in a potassium-based process) then the mill may prefer the Moses Lake straw. However, if the fluctuating inorganic is a non-process element, then the addition of this impurity could upset chemical recovery equilibriums. To minimize the impact of non-process elements, pulp mills could selectively collect straw with the preferential ash contents or operate the recovery system to account for increased non-process elements.

In addition to the location-year interaction, acid soluble ash was also dependent on the cultivar. The Eltan, Lewjain, and Stephens cultivars had significantly more acid soluble ash than Cashup and Rod. Figure 5.10 shows the acid soluble ash for the six cultivars grown in Pullman. For comparison this figure has the same ordinate axis as Figure 5.9. Once again, knowing which mineral was changing content will help a papermaker choose the preferred cultivar or plan ahead for raw material changes.

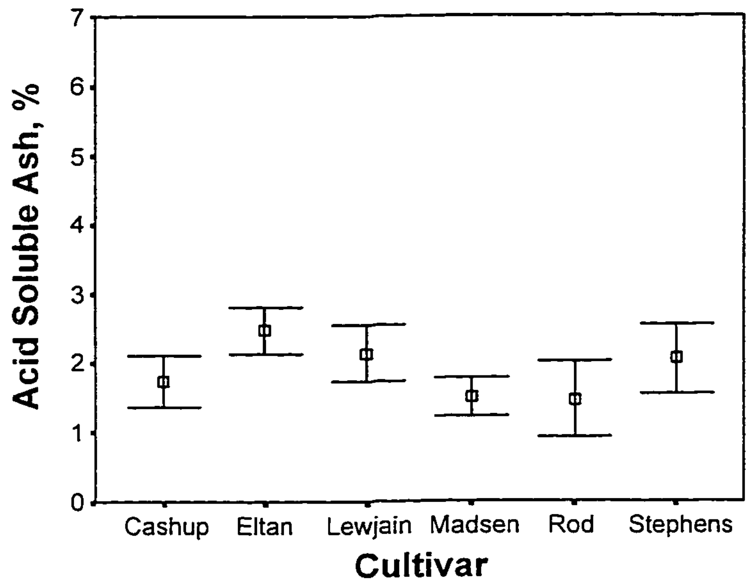


Figure 5.10. Pullman Acid Soluble Ash Contents

A preliminary examination of the 1996 Moses Lake straw determined the internodal mineral content of the six cultivars. Replicate analyses and consistent internodal sampling were not available so statistical comparisons were not possible with the 1996 harvest. To begin an investigation of the true within the field variation in internodal mineral content, 1998 Madsen Moses Lake straw was collected. The standard deviation for the within-the-field variation in mineral content was listed in Table 5.3. To compare the mineral content of the cultivars, a future study would need to analyze replicate samples of each cultivar. Based on the acid soluble minerals from the non-

Table 5.4. Internodal Straw from Moses Lake in 1996: Metals Estimates and Calculated Power[1]

| Metal | Madsen (ppm) | Eltan (ppm) | Stephens (ppm) | Lewjain (ppm) | Cashup (ppm) | Rod (ppm) | | Power |
|---|---|---|---|---|---|---|---|---|
| Ba | 48 | 28 | 30 | 50 | 62 | 58 | | >0.99 |
| Ca | 2000 | 2510 | 2330 | 1960 | 1780 | 2530 | | >0.99 |
| Cu | 4 | 3 | 3 | 4 | 3 | 3 | | <0.30 |
| Fe | 42 | 59 | 41 | 21 | 87 | 37 | | <0.30 |
| K | 22750 | 13000 | 22000 | 20000 | 20000 | 21000 | | >0.99 |
| Mg | 1310 | 730 | 620 | 500 | 680 | 560 | | >0.99 |
| Mn | 16 | 12 | 15.6 | 15.4 | 25.1 | 14.5 | | 0.89 |
| Na | 135 | 430 | 230 | 280 | 390 | 150 | | >0.99 |
| P | 830 | 420 | 480 | 330 | 400 | 440 | | 0.52 |
| Sr | 10.0 | 9.1 | 13.9 | 10.4 | 11.8 | 11.8 | | >0.99 |
| Zn | 16 | 10 | 11 | 7 | 13 | 7 | | 0.35 |

[1] Estimated power for a between cultivar analysis using these cultivars, four within-the-field samples, and $\alpha$=0.05%

replicated 1996 analysis of cultivars and the standard deviations determined from the 1998 harvest (Table 5.3), analysis of four, within-the-field replicates would be able to identify significant differences, if present, for barium, calcium, potassium, magnesium, sodium, and strontium (Table 5.4). However, such a study would not have enough power to identify differences in copper, iron, manganese, phosphorous, or zinc content. Limited sample and the preliminary nature of this investigation prevented cultivar metals comparisons. However, depending on the mineral of interest, future examinations of inorganic content of straw may find these ranges useful in planning their experimental plants.

## Conclusions

The straw harvested at Dusty in 1996 had a lower cellulose content than the other site year. If an agricultural practice is affecting the straw cellulose content, identification of this practice may lead to more uniform and perhaps improved pulping yields.

Likewise, knowing the cause of the lower lignin content in the 1997 straw harvested at Moses Lake would be beneficial. If lignin content could be consistently lowered without influencing cellulose content or grain, this raw material may have superior pulping yields.

Pulping yield was estimated based on chemical composition. Of the currently available straw, the leaf-free Moses Lake straw has slightly higher pulping yields. Examination of additional harvests along with actual pulping studies are recommended to quantify the pulping yield increase.

Potassium and silica were the predominant inorganics found in the local straw internodes. Both soil conditions and fertilization are suspected to be the predominant influences on the inorganic uptake. Current agricultural practices or soil conditions have resulted in less silica in the internode of Dusty-grown straw than from Moses Lake or Pullman. Acid soluble ash, hypothesized to be predominantly potassium, was highest in Moses Lake internodal straw and was likely due to crop rotation.

Internodal mineral uptake was also influenced by cultivar. Cultivar changes in a raw material supply of a papermaker may correspond to variations in the addition of non-process elements to the pulp mill. Depending on the ability of the mill to handle these non-process elements, the monitoring of cultivar changes may be of value.

Knowing that the local wheat straw chemical composition is influenced by growing location and cultivar, papermakers have several options. Pulp mills could aim for a consistent mix of straws to ensure uniform process conditions and product quality. Papermakers could also determine the influence of these variations on their process and yield. With such knowledge, the impact of supply changes may be estimated; and the better raw material may be chosen or developed genetically.

# Chapter 6
# Fractionating and Refining Wheat Straw Pulp

In the paper industry, wheat straw pulp has similar papermaking roles as hardwood pulps [Tabb, 1974; Rab & Szikla, 1992]. However, these two raw materials have different chemical compositions, fibers, and fiber length distributions. Adding wheat straw to a raw material supply would affect pulping, chemical recovery, processing, yield, and paper properties [Watson & Garner, 1997; Papp & Renner, 1990].

Wheat straw chemical composition and fiber properties vary with cultivar, growing conditions, and the harvesting method [Chapters 2-5]. Both selective harvesting and preprocessing of wheat straw are potential methods of collecting the best raw material for a specific product. An additional approach to upgrading this raw material is pulping the straw and then removing the undesired cells. Removal of 20% of the fines from wheat straw pulp has been found to increase the pulp strength 10% [Kuang & Zhang, 1987]. Similar strength improvements may be realized with the Pacific Northwest straw pulp; so, the pulp properties and fractionation potential of mechanically-harvested wheat straw is examined. This quantification of the papermaking properties for locally-available straw, along with comparisons to hardwood pulps, will aid papermakers in choosing the best processes and products for wheat straw pulps.

Most wood furnishes are refined before use to enhance fiber and pulp properties. During refining, fibers can be fibrillated and cut; and the external fibrils are sometimes broken off [Young, 1980; Scott, 1986; Laivins and Scallan, 1996]. This mechanical action improves pulp uniformity, formation, and sheet strength [Young, 1980]. However, the increased surface area and fines content from refining lowers the freeness [*ibid.*]. Papermakers try to balance the strength development with reductions in drainage to produce the optimum pulp for their product lines. The benefit of refining wheat straw pulp is unclear. The refining behavior of Pacific Northwest wheat straw pulp is reported in this chapter.

Strength development from refining wheat straw is often accompanied by a rapid drop in freeness [Watson & Garner, 1997]. Prevention of this freeness drop may open new doors to this raw material. The mechanism of freeness loss is unclear. Wheat straw tracheids may fibrillate, cut, or lose fibrils at a different rate than hardwood tracheids. In addition, wheat straw parenchyma and epidermal cells may react to mechanical action differently than the hardwood fines.

In the present work, the refining curves of two hardwood and two wheat straw pulps are compared to help elucidate the mechanism of freeness drop in these two pulps. Having a better understanding of the refining behavior of wheat straw pulp could be the first step in developing unit operations especially designed for wheat straw pulp and may suggest genetic engineering opportunities for developing a superior papermaking raw material.

## Experimental

Alkaline ammonium sulfite wheat straw pulp was made from bales of Madsen wheat straw grown on irrigated soil (Moses Lake, Washington). The straw was not preprocess and still contained leaves and nodes. The pulp had a kappa of 20. Some of this pulp was fractionated on a Bauer McNett to remove fines and short fibers. All of the fractions long enough to be caught on a 100 mesh screen were combined and referred to as the "long" fiber fraction. This fractionation process had a 69% yield of long fiber pulp. For comparison, the unfractionated wheat straw pulp was referred to as "whole" wheat pulp.

In addition to the wheat straw pulp, maple and oak chips were individually pulped using the kraft process. These pulps also had kappa values of 20.

Refining was done in a PFI mill at 10% consistency, and a portion of each pulp was analyzed on a Kajaani FS-200 before and after refining. Handsheets, freeness, and physical tests were done according to Tappi Standard Methods with the exception of tear which was measured using a single-ply Elmendorf tear tester [Seth, 1991; Sun, 1976].

Two types of fines will be discussed in this chapter. Primary fines are the small (pass 200 mesh), non-tracheid cells in a unrefined pulp [Casey, 1980; Scott, 1986; Laivins and Scallan, 1996]. Both hardwoods and wheat straw will often have parenchyma cells in their primary fines fraction. Wheat straw primary fines also contain epidermal cells from the outer layer of the plant. The second type of fines, secondary fines, are the result of refining and are pieces of fibers and fibrils from the outer layers of fibers that are broken off during refining [Casey, 1980; Scott, 1986; Laivins and Scallan, 1996].

## Unrefined Pulps -- Results and Discussion

To better understand the differences between hardwood and wheat straw, the fiber and paper properties of the unrefined pulps were first examined. All three pulps had different fiber length distributions (Figure 6.1), but the maple pulp had a more bimodal fiber length distribution. The oak and wheat straw pulps contained more of the longer fibers, 1.2 - 1.7 mm, than the maple pulp resulting in higher weighted average fiber lengths (Table 6.1).

The peaks from 0 - 0.2 mm represent the fines content for each pulp. Numeric values for fines content are listed in Table 6.1. All three pulps had high quantities of fines, but the hardwood pulps had narrower fines distributions than the wheat straw pulp.

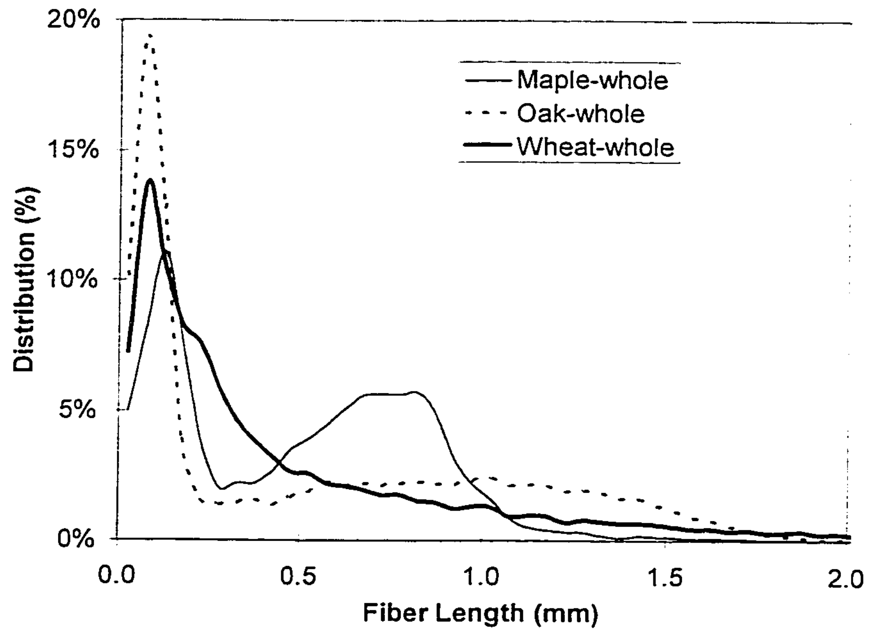


Figure 6.1. Fiber Length Distributions of Unrefined Pulps.

Table 6.1. Unrefined Pulp Properties

| | Whole Maple | Whole Oak | Whole Wheat | Long Wheat |
|---|---|---|---|---|
| NAFL, mm | 0.48 | 0.52 | 0.47 | 0.83 |
| WAFL, mm | 0.73 | 1.03 | 1.09 | 1.24 |
| Fines, P% | 32.1 | 46.4 | 39.5 | 9.4 |
| Coarseness, mg/100 m | 10.8 | 9.8 | 15.5 | 13.2 |
| Freeness, CSF | 665 | 705 | 440 | 690 |
| Density, $g/cm^3$ | 0.51 | 0.51 | 0.53 | 0.53 |
| Tensile, Nm/g | 43.7 | 35.6 | 74.2 | 54.3 |
| Tear, $mNm^2/g$ | 4.06 | 3.89 | 6.72 | 7.19 |
| Water Retention, % | 239 | 165 | 265 | 198 |

Due to their small size, the presence of fines can plug the sheet, decrease freeness [Casey, 1980] and increase water retention value [Abson and Gilbert, 1980]. However, neither the unrefined freeness nor water retention of the three pulps correlated directly with fines content. The fines content of the wheat straw pulp was less than the oak but greater than the maple pulp, but the freeness of the wheat straw pulp was over 200 CSF lower than the hardwoods. Low freeness values are common for unfractionated wheat straw pulps [Rab & Szikla, 1992; Cheng *et al.*, 1994; Wågberg *et al.*, 1990; Watson & Garner, 1997], and this poor drainage will decrease washer efficiencies and reduce papermachine speeds [Cheng *et al.*, 1994].

Differences in character of primary fines may explain the lower freeness of wheat straw pulps. The fines contents of these three pulps were of the same order of magnitude; however, their influence on drainage was not. The primary fines from hardwood pulps contain large quantities of ray cells [Casey, 1980]. These cells are thick-walled and resist deformation [*ibid.*]. Removal of the primary fines and short fibers from the maple pulp using the same fractionation method as the wheat straw pulp resulted in an 18% yield loss but increased the freeness from 680 to 720 CSF. This freeness increase was statistically significant but smaller than with the wheat straw pulp.

Wheat straw primary fines are predominantly thin-walled parenchyma cells. These parenchyma are easily deformed and, when present, also reduce drainage [Cheng *et al.*, 1994]. The presence of the wheat straw primary fines and short fibers reduced the freeness 250 CSF, six times more than the hardwood primary fines. The freeness of the wheat straw pulp increased from 440 CSF to 690 CSF when the primary fines were removed. The resulting long pulp had a freeness comparable to the hardwood pulps but a higher tensile, tear, and average fiber length.

Even though wheat straw fines reduce freeness and increase the water retention, they improve the tensile strength of the unbeaten pulp. The whole unrefined wheat straw pulp had a superior strength to the unrefined hardwood pulps (Table 6.1). The small (4%) differences in sheet density would probably not explain these strength differences; but the fines may. When the wheat straw fines were removed, the tensile index decreased from 74 to 54 Nm/g. The thin-walled wheat straw parenchyma must collapse thereby aiding in bonding and contributing to the tensile strength.

Without the primary fines, the unrefined long wheat straw had a similar freeness, higher tensile and higher tear than the unrefined hardwood pulps. If these properties are maintained through refining, the long wheat straw pulp may have similar or even superior properties when compared to the hardwood pulps. The next section will examine the refining behaviors of the two wheat and two hardwood pulps.

## Refined Pulps -- Results

During refining, the three pulps lost freeness and developed tensile strength at different rates. Fiber length distributions, types of cells, chemical composition, and cell structures could be influencing these pulp properties; so, the strength development and freeness drop of the hardwood and wheat straw pulps will be compared. The influence of tracheid fibrillation and fines on these properties will also be examined.

All of the pulps lost freeness during beating (Figure 6.2). The freeness of the two hardwood pulps decreased at similar rates. However, both wheat straw pulps lost freeness more rapidly and had sudden freeness drops at the beginning of refining. After this initial, rapid drop in freeness, the whole straw pulp continued to rapidly lose freeness while the long pulp behaved more like the hardwoods.

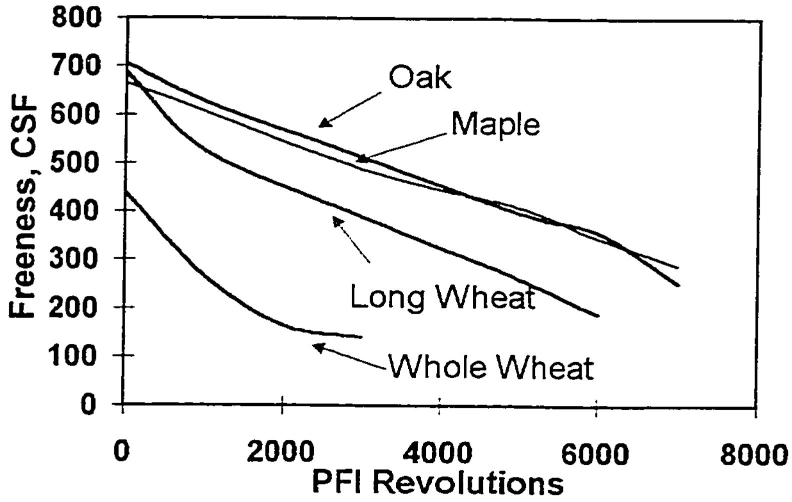


Figure 6.2. Freeness Decrease with Refining

Even with similar freeness curves, the two hardwoods did not develop tensile or tear at the same rate. The maple pulp developed tensile strength more rapidly than the other three pulps (Figures 6.3 and 6.4) and, when compared at 300 CSF [Young, 1980], had a 25-33% higher tensile index. This was not surprising since it is well know that maple is stronger than oak pulp.

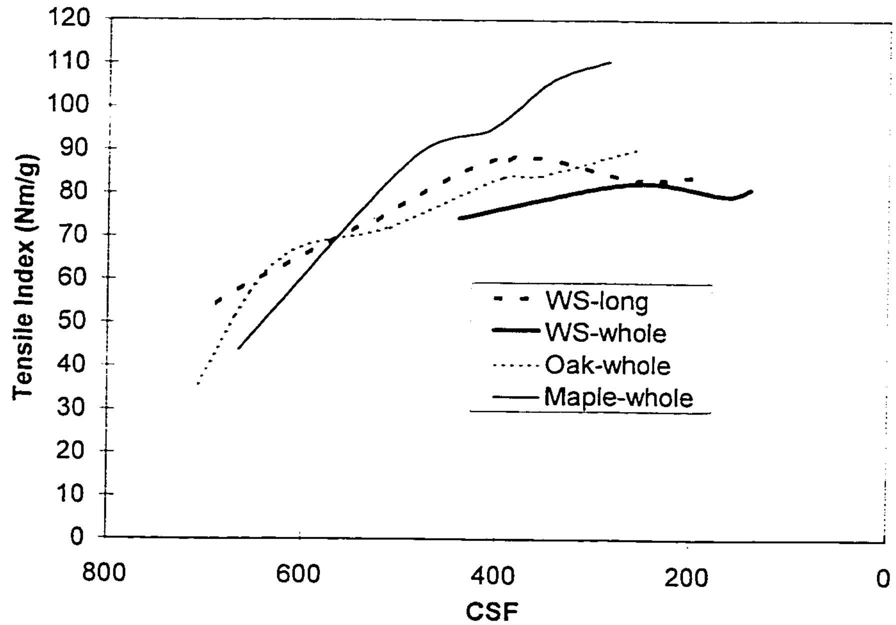


Figure 6.3. Tensile Development at the Different Freenesses

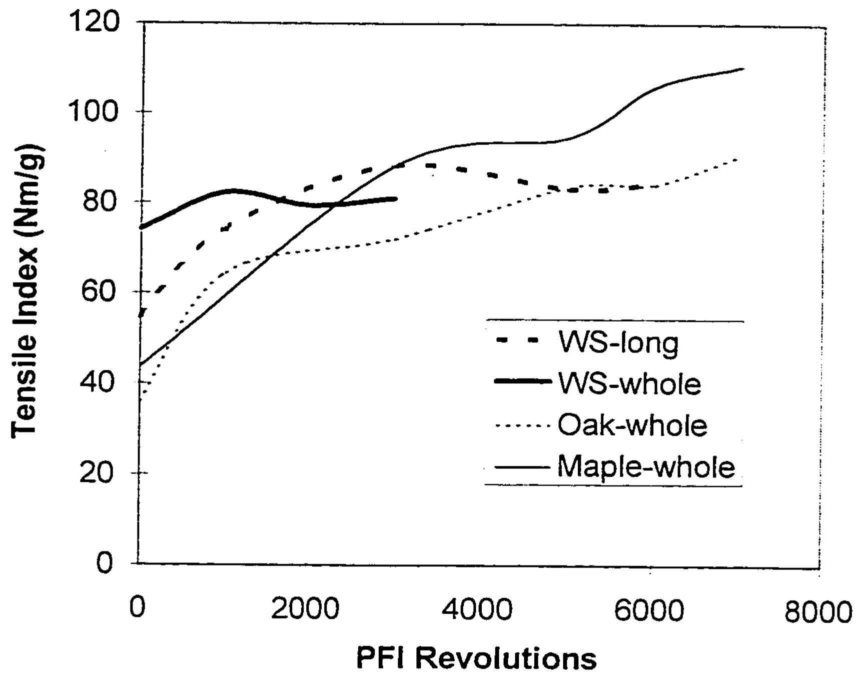


Figure 6.4. Mechanical Energy Needed for Tensile Index Development

At 300 CSF, the two hardwood and the whole wheat pulps were still developing tensile. However, the long wheat pulp had already reached a maximum tensile and was losing strength. The long wheat straw maximum, 88 Nm/g, was not significantly different than the tensile index of the oak pulp, 87 Nm/g, at 300 CSF. But the long wheat pulp reached this maximum at 400 CSF and needed less than half the mechanical action (3000 PFI revolutions compared to 7000 for the oak) to reach this tensile index (Figure 6.4). Clearly, wheat straw pulps will require less refining energy to reach target strengths.

Refining the whole wheat straw pulp to 300 CSF only improved the tensile index 11%, from 74 to 83 Nm/g. This strength increase was lower than was found with the other pulps, but the wheat straw pulp also started at a higher unrefined freeness. The hardwoods improved their tensile 144-150% by 300 CSF. The long wheat straw pulp only increased 62% at the peak and 57% at 300 CSF. Differences between the wheat straw and wood pulps were also apparent in tear index.

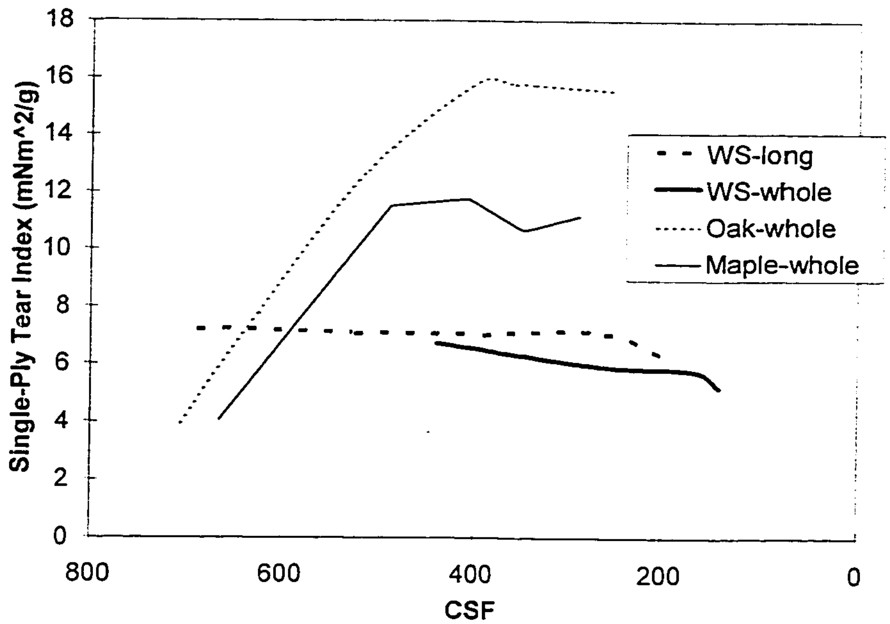


Figure 6.5. Single-Ply Tear

At the beginning of refining, both of the hardwood pulps developed tear strength (Figure 6.5); and, by 300 CSF, tear development seemed to have leveled off or begun to decline. The tear index at 300 CSF for the oak pulp was 43% higher than the maple pulp and 124-153% higher than the wheat straw pulps. This lower tear strength for wheat straw pulps has also been reported by Rab and Szikla [1992]. Even though the unrefined wheat straw pulps had superior tear indexes (Table 6.1), neither of the wheat straw pulps developed tear during the mild PFI beating. The differences in the types of cells present or the beatability of the fibers may explain these differences in strength development and will be discussed in the next section.

## Refined Pulps -- Discussion

The wheat straw and hardwood pulps had different responses to refining. The wheat straw pulp lost freeness more rapidly, did not develop tensile at the same rate, and had a lower tear strength than the hardwood pulps. These refining behaviors can, in part, be explained by the type of cells present in each pulp and the response of these cells to mechanical action.

Primary fines may cause some of the differences in freeness drop. Hardwood primary fines contain ray cells that are thick-walled and resistant to mechanical deformation during refining [Casey, 1980]. Wheat straw primary fines contain large quantities of thin-walled parenchyma cells [Cheng *et al.*, 1994]. These cells are easily crushed with mechanical action [*ibid.*; Hua & Xi, 1988; Zhai & Lee, 1989]. When crushed, wheat straw parenchyma plug the pores of the sheet and reduce drainage. Therefore, these crushed parenchyma may, in part, be lowering the freeness of the whole wheat straw pulp and not contributing to strength development.

The long wheat straw also lost freeness more rapidly than the hardwood, even after the primary fines were removed from this pulp. The initial drop in freeness for the long wheat corresponded to a sudden decrease in average fiber length (Figures 6.6) and increase in fines content (Figure 6.7). Similar rapid average fiber length decreases were not seen with the other pulps.

During the refining of wood pulps, average fiber length decreases through fiber cutting and fibrils being broken off of the fiber. The released fibrils contribute to wet-web strength and the distribution of drying stresses [Scott, 1986; Htun *et al.*, 1978]. Microscopic examination of the long wheat pulp before and after mild refining displayed

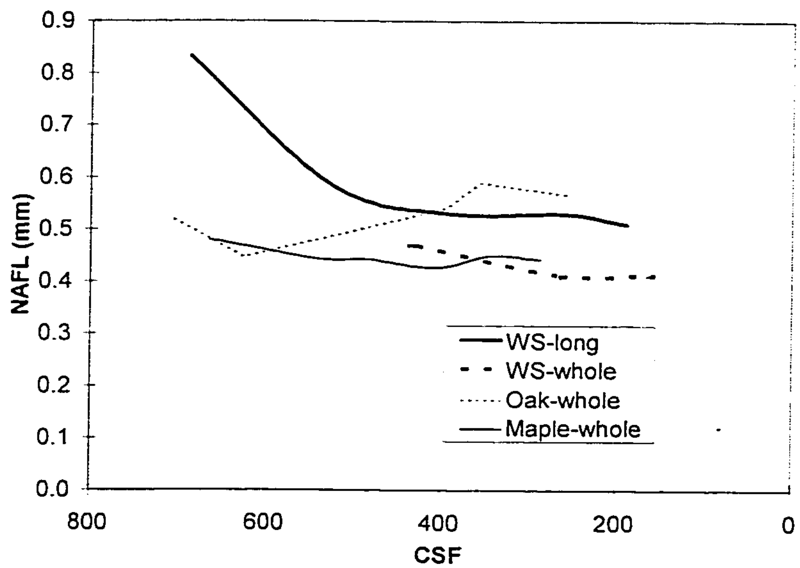


Figure 6.6. Numerical Average Fiber Length

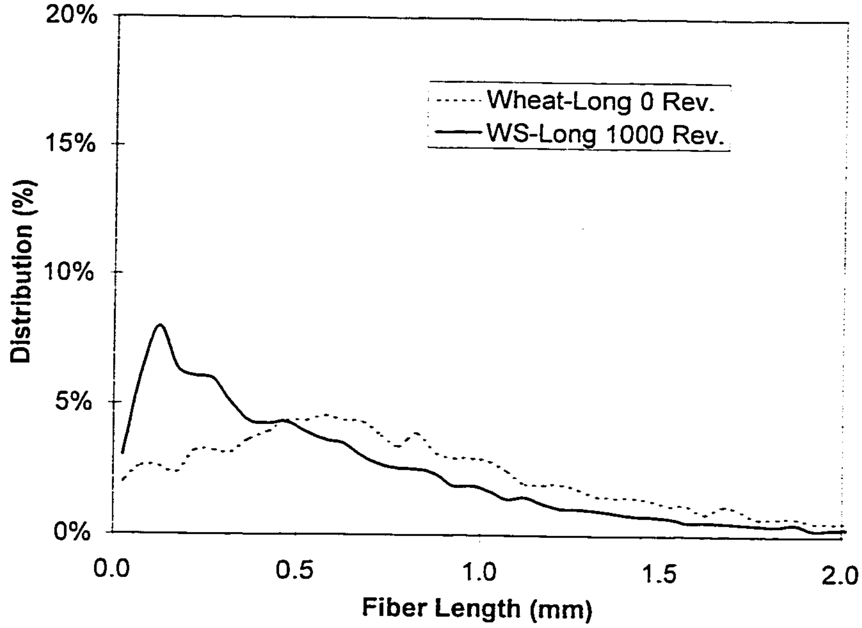


Figure 6.7. Fiber Length Distribution of Long Wheat Pulp

significant differences relative to the wood pulp. For example, large quantities of fiber fragments or released fibrils were not found in the mildly refined wheat straw pulp. Instead the parenchyma content changed. The unrefined long wheat pulp contained very few individual parenchyma and epidermal cells. However, parenchyma and epidermal cells were present in the form of agglomerates (Figures 6.8 & 6.9).

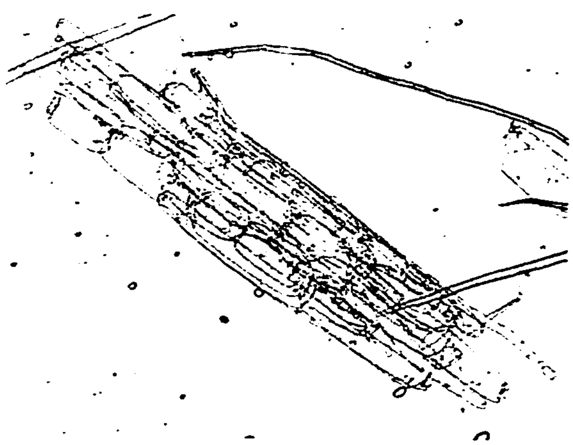

Figure 6.8. Parenchyma Agglomerate

Figure 6.9. Epidermal Agglomerate

After the mild mechanical action on the long wheat pulp, the parenchyma and epidermal agglomerate content was reduced with a corresponding increase in quantity of individual parenchyma cells. This breaking of wheat straw agglomerates during refining has also been observed by Mansour [1985] and Utne and Hegbom [1992]. Increases in individual wheat straw parenchyma cells could be contributing to the rapid loss in freeness and may, in part, explain the differences in strength development between the long wheat straw and hardwood pulps.

Like the primary fines, the wheat straw tracheids may respond differently to refining than the wood tracheids. Typically, mechanical action increases the flexibility, surface area, and bonding of tracheids [Casey, 1980]. Unequal rates of tracheid fibrillation may correlate with the dissimilar tensile responses and lower tear strength of the wheat straw pulp relative to the wood pulps.

To examine tracheid fibrillation, unfractionated wheat straw and maple pulps were beaten in a PFI mill. At several points through the refining period, the freeness of these "unfractionated" pulp samples was measured. Then, the fines and short fibers were removed; and the freeness of the tracheids was determined. These freeness curves are shown in Figures 6.10 and 6.11.

The lower, unfractionated curves in both Figures 6.10 and 6.11 are the same as the whole straw and maple curves in Figure 6.2. However, the "tracheids" in Figure 6.11 is not the same as the "long" wheat curve in Figure 6.2. The primary fines and short fibers were removed from the long wheat straw before refining. During the refining of this long wheat straw pulp, secondary fines may have been generated; and the agglomerates released parenchyma and epidermal cells. These newly released fines were not separated from the tracheids in Figure 6.2. However, the tracheids in Figures 6.10 and 6.11 did not contain fines and short fibers. Both the primary fines and newly released fines were removed before determining the tracheid freeness. So, comparisons of the curves in Figures 6.10 and 6.11 provide some indication of the response of tracheids and fines to mechanical action.

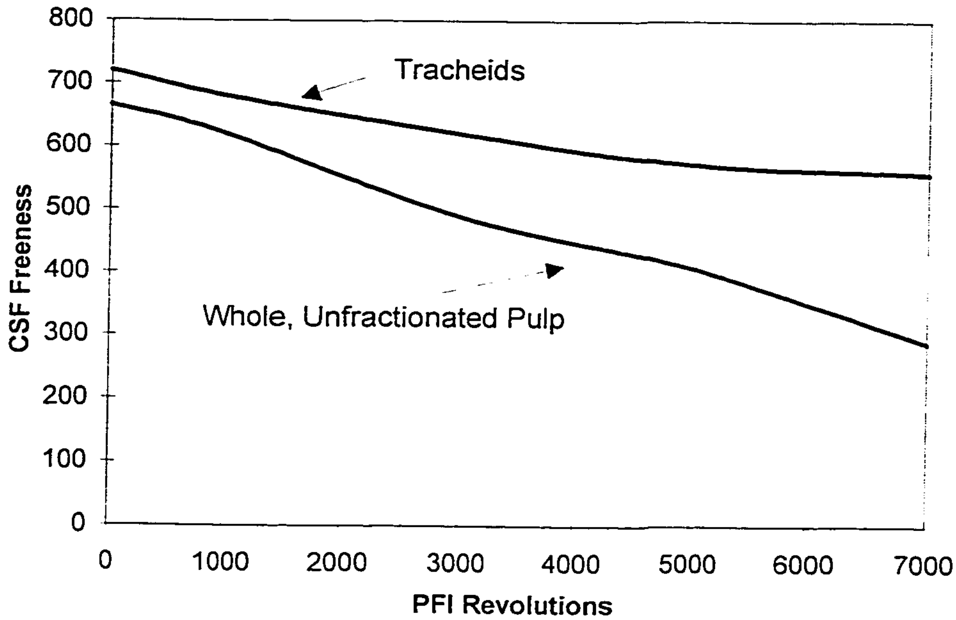


Figure 6.10. Freeness of Maple Tracheids

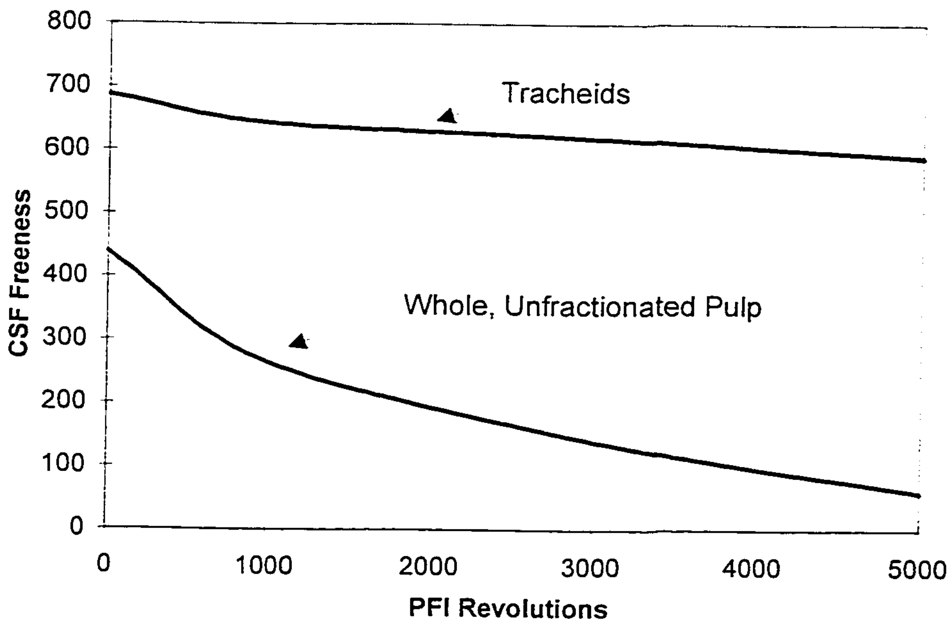


Figure 6.11. Freeness of Wheat Straw Tracheids

During refining, the freeness of the tracheids decreased. After 5000 revolutions, the hardwood tracheids had a freeness of 570 CSF, a 150 CSF reduction from the unrefined freeness of 720. However, the wheat straw tracheids had a freeness of 590 CSF after 5000 revolutions which was only 100 CSF less than the unrefined tracheids (690 CSF). With the same amount of refining, the hardwood tracheids lost 50% more freeness than the wheat straw tracheids. If tracheid freeness drop related directly with fibrillation, then the hardwood tracheids may be more fibrillated and may have different papermaking properties.

Lower wheat straw tracheid fibrillation may be due to tracheid morphology. The structure of wheat straw tracheids is similar to wood, however the S1 layer is thicker [Roelofsen, 1959; Hua and Xi, 1988; Zhai and Lee, 1989]. Roelofsen [1959] found that both the primary and S1 layers of wheat straw fibers were easily detached and speculated that the differences in papermaking properties between wood and wheat straw were probably due to the variations in fiber dimension and chemical composition. However, Hua and Xi [1988], Zhai and Lee [1989], and Zhao *et al.* [1992] state that wheat straw tracheids are harder to fibrillate than wood. Zhao *et al.* describe a thick S1 layer that is tightly bonded to the S2 layer and is difficult to remove thereby making the wheat straw tracheids harder to refine.

Examination of Washington wheat straw also suggests that wheat straw tracheids may be more difficult to fibrillate than hardwood tracheids. This difference in the ability to fibrillate the long fiber fraction may help explain the variations in strength development between hardwood and wheat straw pulps.

During refining, the whole wheat straw pulp developed less tensile than the other three pulps. The tensile increase from unrefined to 300 CSF (74 Nm/g to 83 Nm/g) was only an 11% change. The maple and oak pulps developed 6-9 times more absolute tensile with mechanical action. The thicker S1 layer in wheat straw tracheids may be hindering strength development, but the primary fines content could also be negatively influencing tensile.

During refining, the freeness of the maple pulp decreased from 665 to 410 CSF at 5000 revolutions. The tracheid fibrillation contributed to 150 CSF of the 255 CSF drop. The remaining 105 CSF was from the short fibers and fines. For the refined wheat straw pulp, the short fibers and fines had close to a three times larger contribution to the freeness drop, 280 CSF. This freeness reduction from the fines may be due to several factors including parenchyma crushing, secondary fines generation, and the release of parenchyma and epidermal cells from agglomerates. Quantification of the contributions of each of these factors may be done through fractionation but was beyond the scope of this study. However, the contribution of each of these factors may help explain the tensile and tear development of the wheat straw pulps in comparison to the hardwoods and could lead to improved techniques for wheat straw processing.

Wheat straw primary fines improved the tensile of the unrefined wheat straw pulp. Since primary fines were released from agglomerates, then some of the wheat straw strength development may have come from the newly released primary fines. Without the large quantities of primary fines, the long wheat straw tracheids may have been better able to fibrillate and create secondary fines. If this mechanism contributed to strength development, then primary fines removal may be beneficial before refining.

A better understanding of these mechanisms of strength development and the fractionation and mixing potentials of wheat straw pulp may suggest methods for improving the tensile index of this raw material. Refining only the long fiber fraction could lead to more strength development, and adding the unrefined wheat straw primary fines back to the refined long pulp may further improve tensile. Knowing the role and potential of wheat straw cells could help in the development of improved processing techniques for this raw material.

Removal of the wheat straw primary fines lowered the wheat straw tensile index from 73 to 54 Nm/g. The long wheat straw pulp was able to regain the tensile with mechanical action, but the mechanism of strength development was not identical to the hardwood since primary fines were released from agglomerates during wheat straw refining. However, this long wheat straw pulp did develop tensile comparable to oak using less than half of the energy and resulting in a pulp with a 100 CSF lower freeness. The improved energy requirement may correspond to savings in production costs and the higher freeness may make water removal easier on the papermachine enabling faster production rates.

Mild refining improved the tear of the wood pulps but did not improve the tear strength of the wheat straw pulps. This initial increase in wood tear is believed to be from improved bonding [Young, 1980]. The lesser tracheid fibrillation or increase in parenchyma cell content may be hindering improvements in wheat straw bonding. Young [1980] also stated that tear is limited by fiber length. However, the tear strength of the wheat straw pulp did not improve with primary fines removal. If the primary fines are impairing tear, then their total removal may be necessary to improve tear. This may be done by breaking up the agglomerates with mild refining action and then fractionating.

Another possibility is that the structure or dimensions of the wheat tracheids may be causing the lower tear. Even though the lengths may be the same, the thinner tracheids of wheat straw pulp may reduce bonded area and result in lower tear. Identification of the factor or factors causing the lower wheat straw tear may lead to methods of improving the papermaking properties of this raw material.

## Conclusions and Recommendations

Pacific Northwest wheat straw pulp was compared to two hardwood pulps, maple and oak. The unrefined wheat straw pulp had a lower freeness, higher tensile and higher tear. The primary fines and short fibers in the wheat straw pulp lowered the freeness and contributed to the higher tensile. However, wheat straw fines removal did not create a pulp identical to the hardwoods.

The mechanisms for strength development during refining may be different with these raw materials. Hardwood strength improvements come from tracheids being fibrillated and from the generation of secondary fines. In addition to fibrillation and secondary fines, the refining of wheat straw pulps also leads to parenchyma crushing and the release of additional parenchyma and epidermal cells.

Removal of most of these primary fines and short fibers did not enable this long wheat straw pulp to develop a higher tensile index than the whole wheat straw pulp or the hardwoods. However, the long wheat straw pulp did use less energy than the hardwoods to develop tensile and had a superior freeness than the other pulps. Since the parenchyma agglomerates were not removed in this fractionation, the influence of parenchyma crushing was not completely eliminated. Agglomerate removal would be needed to quantify the impact of parenchyma crushing.

Tracheid morphology could also help explain the variations in strength development. The freeness of the hardwood tracheids decreased 50% more than the wheat straw tracheids suggesting differences in tracheid structure. The higher freeness drop may represent increased fibrillation for the hardwood tracheids. Microscopic examination may confirm variations in fibrillation. Fractionation of the tracheids and comparisons of paper strength properties could help determine the influence of wheat straw tracheids on the strength development of this raw material.

Understanding wheat straw refining response and strength development may lead to improved methods of processing this raw material. For example, if the wheat straw tensile is being hindered by crushed parenchyma, then the quantity of these cells can be reduced through preprocessing the straw by removing leaves, nodes and perhaps even the internodal pith layer. Fines can also be fractionated from the pulp before refining. Then, if the unrefined parenchyma contribute to strength, adding these fines back to the pulp after refining may further improve the tensile index.

This study examined the properties of wheat straw pulp. In most industrial settings, this raw material would be mixed with other pulps and, inorganic fillers. In such a furnish, the impact of the wheat straw pulp may, once again, be different than hardwood. Understanding the role of the various wheat straw cells in blends may also suggest alternative fractionation benefits and may lead to new markets for this raw material.

# Chapter 7
# Conclusions and Recommendations

## Conclusions

Following are the major, novel conclusions from the main body of this study:

1. Wheat straw pulp is a viable hardwood substitute. Pacific Northwest grown wheat straw pulp and hardwood pulps had comparable average fiber lengths, but the straw contained less cellulose, less lignin, more silica, and more potassium. The unrefined whole wheat straw pulp had a lower freeness and higher tensile than maple and oak pulps. Refining responses also differed with the wheat straw pulp losing freeness more rapidly. However, the refined, fines-free wheat straw pulp reached the same tensile index as refined oak with less mechanical energy and a lower freeness suggesting advantages in wheat straw.

2. Developmental wheat cultivars grown at a dryland location had stem heights ranging from 30 to 120 centimeters. Internodal average fiber length was proportional to stem height. Therefore, the use of non-dwarf wheat straw for papermaking may lead to a superior product.

3. The leaves, nodes, and internodes of the wheat plant vary in chemical compositions, fiber length distributions, and have the potential for different papermaking properties. The internodal sections had the more promising papermaking potential since they had less fines, a superior average fiber length, higher cellulose contents, and less ash. These properties could lead to higher yields, better drainage, less non-process elements in chemical recovery, and improved papermaking properties compared to the other parts of the plant or the use of the whole plant. In terms of silica, removal of just the leaves would remove almost two-thirds of the silica entering the mill. The additional removal of nodes did not change the silica content but should improve yield, average fiber length, fines content, and acid soluble ash content.

4. Two internodal collection methods had been proposed: (1) collecting just the top internode and (2) collecting all of the internodes by harvesting all of the straw and

then removing the leaves and nodes by fractionation. Since internodal chemical composition and fiber properties varied along the length of the plant, the properties of these internodal raw materials differed. Compared to collecting all of the internodes, collection of only the top internode could result in lower pulping yields, lower average fiber lengths, and more inorganics including silica and potassium.

5. An additional internodal preprocessing opportunity exists through epidermis and pith removal. The outermost internodal layer is comprised predominantly of epidermal cells and a waxy protective layer, the cuticle. This epidermis is highly lignified, contains the silica, and is predominantly short fibers or fines. Mechanical removal of the epidermal layer from the internode could improve pulping yields, bleaching requirements, and drainage.

Removal of the innermost internodal pith layer could also improve wheat straw pulp properties. The pith contains less-lignified, thin-walled parenchyma cells. Unrefined, these cells may be improving strength properties, but parenchyma also plug the sheet impairing drainage and are easily crushed during refining perhaps causing further drainage problems. Pith removal would reduce the amount of these parenchyma cells in the wheat straw pulp.

6. Selectively harvesting wheat straw may be used to collect the raw material best suited for a process or product. Wheat straw chemical composition and fiber properties were influenced by growing conditions and the commercial cultivar selected:

A. The irrigated, Moses Lake location had wheat straw with higher average fiber lengths than straw grown at the dryland sites.

B. At each location, pulp made from the Madsen cultivar had a higher average fiber length and lower fines content. Conversely, the Stephens cultivar had lower fiber lengths and higher fines contents. Selective harvesting of superior cultivars may lead to improved processing and product properties. Alternatively, controlled mixing of different straw may aid in preventing process upsets.

C. Wheat straw pulp coarseness was predominantly influenced by growing location but did not correlate directly with the amount of water that the plants received. Several factors, including cell wall thickness, types of cells, and cell diameter, could be influencing coarseness. Identification of the reason for the coarseness change and comparisons of the pulp strength properties could aid in determining the impact of these coarseness differences on processing and the products.

D. The benefit of using straw from irrigated locations was further supported by the higher cellulose to Klason lignin ratio for this straw which suggests higher pulping yields.

E. Silica content was influenced both by cultivar and growing location. Only the Cashup cultivar stood out from the rest by having higher silica contents at two of the three growing locations. Comparing the three growing locations, the straw grown in Dusty had lower internodal silica contents. Identification of the reasons for these differences could lead to methods of reducing the silica entering the mill.

F. Potassium was the predominant acid soluble inorganic in the wheat straw. Acid soluble ash contents were higher at the irrigated, Moses Lake growing location, and some cultivars had higher uptakes than others. Since many of these inorganics are non-process elements, both monitoring and a better understanding of the factors influencing the uptake of these metals could help prevent process upsets from raw material variation.

7. Wheat straw fines and tracheids refine and perhaps even contribute to sheet strength differently than hardwood fines and tracheids. These differences in bonding and refining may, in part, explain the low tear, poor refining response, and drainage of wheat straw pulps but may also lead to unique fractionation opportunities.

## Recommendations

The following recommendations follow from the conclusions:

1. The economics of straw preprocessing including the different internodal collection techniques should be examined. Clear documentation of the benefits of leaf and node removal along with comparisons between methods could help in the design of straw pulping facilities. Important technical factors would include bleaching requirements, drainage, inorganics, and sheet properties.

2. The feasibility, benefits, and economics of internodal pith and/or epidermis removal should be determined.

3. Pulping studies and the examination of additional harvests are necessary to verify the impact of the genetic and environmental influences identified in this dissertation. Future studies should also take into account agricultural practices (i.e. seed spacing, fertilization), weather conditions, and soil properties.

4. The impact of these variations in straw properties should be determined through a series of blending studies. The blending behavior of some of the extreme wheat straw pulps (like Moses Lake Madsen and Dusty Cashup) could be compared to hardwood pulps. Refining and fractionation of the wheat straw pulp should also be factored into this study.

5. The development of superior papermaking wheat cultivars would also benefit paper production. Keeping in mind grain production (grain yield, grain quality, disease, lodging), wheat geneticists may be able to engineer cultivars with less pith, higher average fiber lengths, superior yield, leaves that are easily broken off during harvest, and less mineral uptake.

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# Appendix 1
# Lignin and Carbohydrate Analysis

Modification of the Paprican Method and Cao [1997]

(UW Revision 6/16/98, RSJ)

*The hydrolysis, soluble lignin, reduction and acetylation should be completed within the same work day.*

**A. Hydrolysis**

1. Record weight of oven-dry test tube
2. Add 350-400 mg of air-dry, milled plant biomass into a test tube. Record weight of air-dry sample and test tube.
3. Oven dry to a constant weight at 105°C (4 hours). Cool in desiccator. Weigh.
4. Add 1 mL of 72% $H_2SO_4$ per 100 mg of meal. Incubate for 1 hour at 30° C stirring occasionally (note: if several samples are done, stagger the start of each sample *e.g. 3 minutes*)
5. After incubation, transfer to a 250 mL Erlenmeyer flask with 28 mL of water per mL of acid added (note: if any sample is lost, start over).
6. "Cap" the flask with an inverted beaker or flask and heat in an autoclave for 1 hour (125°C)
7. Add 5 mL of the internal standard to the flask (20 mg/mL of fucose)
8. Let flasks cool to room temperature (can cool under running water)
9. Filter the precipitate lignin through a tared, dry, medium Gooch crucible.
10. Rinse the precipitated lignin with hot water.
11. Collect and save the filtrate for carbohydrate and acid soluble lignin analysis

    --Dilute the filtrate to 140 mL

    --Use 50 mL of the filtrate for the carbohydrate analysis. The remaining is used for acid soluble lignin
12. Dry the Gooch crucible (with lignin) overnight in an oven (105°C)

**B. Klason Lignin & Acid-Insoluble Ash**

1. Remove the Gooch crucible (with lignin) from oven, cool in a desiccator. Weigh.
2. Ash lignin at 525°C for 3 hours. Cool crucible to ~ 100°C in furnace (to prevent breaking). Transfer to a desiccator. Cool. Weigh

**C. Acid Soluble Lignin**

1. Warm up the spectrophotometer for ~30 minutes
2. Use 3% $H_2SO_4$ for zeroing the equipment
3. Volumetrically dilute the sample as necessary with 3% $H_2SO_4$ (record dilution)
4. Determine the absorbency of the filtrate at 205 nm (record)
5. Turn off the spectrophotometer (do not leave on overnight)

**D. Carbohydrate Content (Reduction and Acetylation)**

1. To the 50 mL of filtrate (hydrolyzate), add 3.95 mL of conc $NH_4OH$. This brings the solution to 1 M with respect to $NH_4OH$.
2. Pipet 2 mL of the filtrate from #1 into a clean dry 125 mL Erlenmeyer flask
3. Add 1 mL of sodium borohydride solution. (1.15 g sodium borohydride/10 mL 3 M $NH_4OH$)
4. Place the flask in a 40±1°C water bath for 90 minutes. Stir the contents occasionally.
5. After reduction, the excess sodium borohydride is decomposed by adding 1 mL of acetic acid dropwise..
6. Pipet in 5 mL of 1-methylimidazole and a magnetic stir bar
7. Immediately add 20 mL of acetic anydride and begin stirring. (Record the time)
8. After 20 minutes add ~30 g of crushed ice and 70 g of distilled water (record time)
9. After at least 20 muntes transfer the misture to a 250 mL separatory funnel
10. Extract the acetylated alditols with successive 10, 5, and 5 mL portions of methylene chloride. Remember to vent. Collect the extractions in a labeled 250 mL beaker
11. Set the beaker aside in the fume hood. The methelene chloride should be allowed to evaporate.

12. Collect the waste from the separatory funnel in a waste bottle. Rinse the separatory funnel with water then a small portion of methylene chloride.
13. Once the methylene chloride from #11 has evaporated, dissolve the acetylated alditol acetates in 2 mL of methylene chloride. (Add 2 mL of methylene choride to the beaker and swirl.) Collect the solution with a new Pasteur pipet. Place the solution in a GC vial.
14. Add a couple crystals of magnesium sulfate to the vial. Seal the vial.

### E. Chromatography

The samples were run on a Perkin-Elmer Chromatograph. A thirty meter DB-225 with 0.25 mm film thickness was used. Injection volumes were one microliter. The injection port temperature was 250°C. The run was isothermal at 220°C. The FID Detector was 250°C. The system was set with a 1:5 split ratio with helium as the carrier gas.

# Appendix 2

# Raw Data Tables for 1996 & 1997 Harvests

Table A.1. Raw Data: Dusty Plant and Fiber Results

| Harvest Year | Cultivar-Replicate | Plant Height (in.) | Stem Mass (g) | NAFL (mm) | WAFL (mm) | MWAFL (mm) | Coarseness (mg/100m) | Fines (%) |
|---|---|---|---|---|---|---|---|---|
| 1996 | Cashup-1 | 39 | 178.2 | 0.412 | 0.915 | 0.105 | 10.45 | 40.04 |
| 1996 | Cashup-2 | 38 | 78.1 | 0.417 | 0.903 | 0.098 | 9.76 | 40.34 |
| 1996 | Cashup-3 | 35 | 179.8 | 0.402 | 0.870 | 0.097 | 9.69 | 39.71 |
| 1996 | Cashup-4 | 33 | 273.5 | 0.391 | 0.853 | 0.102 | 10.19 | 40.30 |
| 1996 | Eltan-1 | 42 | 192.6 | 0.454 | 1.018 | 0.101 | 10.09 | 37.46 |
| 1996 | Eltan-2 | 40 | 120.8 | 0.473 | 1.069 | 0.098 | 9.78 | 36.69 |
| 1996 | Eltan-3 | 33 | 228.2 | 0.457 | 1.034 | 0.108 | 10.82 | 37.06 |
| 1996 | Eltan-4 | 34 | 242.0 | 0.425 | 0.996 | 0.102 | 10.23 | 40.47 |
| 1996 | Lewjain-1 | 35 | 156.9 | 0.415 | 0.911 | 0.090 | 9.01 | 40.35 |
| 1996 | Lewjain-2 | 42 | 181.7 | 0.417 | 0.959 | 0.100 | 9.98 | 41.28 |
| 1996 | Lewjain-3 | 35 | 161.9 | 0.433 | 0.975 | 0.121 | 12.06 | 41.03 |
| 1996 | Lewjain-4 | 33 | 252.3 | 0.449 | 0.948 | 0.102 | 10.19 | 36.63 |
| 1996 | Madsen-1 | 39 | 212.6 | 0.449 | 1.023 | 0.100 | 10.01 | 38.48 |
| 1996 | Madsen-2 | 38 | 209.1 | 0.442 | 1.013 | 0.110 | 11.02 | 38.26 |
| 1996 | Madsen-3 | 36 | 251.3 | 0.463 | 1.084 | 0.088 | 8.77 | 37.93 |
| 1996 | Madsen-4 | 36 | 261.3 | 0.488 | 1.091 | 0.105 | 10.46 | 36.04 |
| 1996 | Rod-1 | 38 | 132.5 | 0.436 | 0.989 | 0.084 | 8.42 | 39.23 |
| 1996 | Rod-2 | 38 | 196.6 | 0.440 | 0.954 | 0.095 | 9.55 | 37.79 |
| 1996 | Rod-3 | 35 | 153.8 | 0.465 | 1.013 | 0.095 | 9.46 | 36.19 |
| 1996 | Rod-4 | 39 | 269.4 | 0.423 | 0.964 | 0.111 | 11.15 | 41.41 |
| 1996 | Stephens-1 | 38 | 229.6 | 0.388 | 0.858 | 0.104 | 10.36 | 43.09 |
| 1996 | Stephens-2 | 37 | 230.2 | 0.394 | 0.860 | 0.113 | 11.35 | 42.02 |
| 1996 | Stephens-3 | 36 | 138.6 | 0.375 | 0.815 | 0.107 | 10.71 | 43.76 |
| 1996 | Stephens-4 | 33 | 257.3 | 0.415 | 0.888 | 0.094 | 9.36 | 39.31 |
| 1997 | Cashup-1 | | | 0.447 | 0.967 | 0.103 | 10.32 | 38.80 |
| 1997 | Cashup-2 | | | 0.430 | 0.945 | 0.103 | 10.28 | 39.33 |
| 1997 | Cashup-3 | | | 0.474 | 1.016 | 0.093 | 9.31 | 36.11 |
| 1997 | Cashup-4 | | | 0.438 | 0.943 | 0.106 | 10.57 | 38.74 |
| 1997 | Eltan-1 | | | 0.475 | 1.045 | 0.104 | 10.36 | 36.74 |
| 1997 | Eltan-2 | | | 0.455 | 1.045 | 0.112 | 11.19 | 39.57 |
| 1997 | Eltan-3 | | | 0.466 | 1.044 | 0.141 | 14.06 | 38.52 |
| 1997 | Eltan-4 | | | 0.467 | 1.078 | 0.108 | 10.81 | 38.72 |
| 1997 | Lewjain-1 | | | 0.444 | 0.971 | 0.102 | 10.19 | 39.13 |
| 1997 | Lewjain-2 | | | 0.433 | 0.923 | 0.099 | 9.89 | 39.51 |
| 1997 | Lewjain-3 | | | 0.437 | 0.954 | 0.098 | 9.80 | 39.80 |
| 1997 | Lewjain-4 | | | 0.434 | 0.952 | 0.119 | 11.87 | 40.19 |
| 1997 | Madsen-1 | | | 0.529 | 1.186 | 0.111 | 11.13 | 33.70 |
| 1997 | Madsen-2 | | | 0.505 | 1.095 | 0.129 | 12.94 | 35.96 |
| 1997 | Madsen-3 | | | 0.509 | 1.102 | 0.102 | 10.17 | 33.34 |
| 1997 | Madsen-4 | | | 0.473 | 1.049 | 0.112 | 11.21 | 37.62 |
| 1997 | Rod-1 | | | 0.477 | 1.059 | 0.104 | 10.37 | 38.19 |
| 1997 | Rod-2 | | | 0.505 | 1.110 | 0.108 | 10.85 | 36.38 |
| 1997 | Rod-3 | | | | | | | |
| 1997 | Rod-4 | | | 0.494 | 1.042 | 0.093 | 9.28 | 35.51 |
| 1997 | Stephens-1 | | | 0.406 | 0.867 | 0.096 | 9.56 | 43.10 |
| 1997 | Stephens-2 | | | | | | | |
| 1997 | Stephens-3 | | | 0.407 | 0.908 | 0.115 | 11.51 | 42.71 |
| 1997 | Stephens-4 | | | 0.420 | 0.868 | 0.094 | 9.40 | 40.00 |

Table A.2. Raw Data: Moses Lake Plant and Fiber Results

| Harvest Year | Cultivar-Replicate | Plant Height (in.) | Stem Mass (g) | NAFL (mm) | WAFL (mm) | MWAFL (mm) | Coarseness (mg/100m) | Fines (%) |
|---|---|---|---|---|---|---|---|---|
| 1996 | Cashup-1 | 37 | 164.7 | 0.511 | 1.132 | 0.106 | 10.58 | 35.55 |
| 1996 | Cashup-2 | 35 | 227.8 | 0.458 | 1.022 | 0.132 | 13.24 | 38.61 |
| 1996 | Cashup-3 | 35 | 144.0 | 0.502 | 1.076 | 0.102 | 10.24 | 36.09 |
| 1996 | Cashup-4 | 34 | 174.6 | 0.478 | 1.024 | 0.111 | 11.07 | 37.58 |
| 1996 | Eltan-1 | 35 | 157.3 | 0.461 | 1.075 | 0.109 | 10.86 | 40.43 |
| 1996 | Eltan-2 | 36 | 124.5 | 0.521 | 1.157 | 0.136 | 13.63 | 35.96 |
| 1996 | Eltan-3 | 40 | 180.1 | 0.484 | 1.118 | 0.110 | 10.95 | 39.27 |
| 1996 | Eltan-4 | 40 | 150.6 | 0.528 | 1.179 | 0.113 | 11.28 | 36.49 |
| 1996 | Lewjain-1 | 36 | 151.0 | 0.486 | 1.096 | 0.121 | 12.08 | 38.36 |
| 1996 | Lewjain-2 | 36 | 148.0 | 0.449 | 0.983 | 0.120 | 12.04 | 39.02 |
| 1996 | Lewjain-3 | 36 | 239.8 | 0.475 | 1.067 | 0.112 | 11.25 | 37.70 |
| 1996 | Lewjain-4 | 36 | 175.7 | 0.484 | 1.018 | 0.101 | 10.11 | 36.57 |
| 1996 | Madsen-1 | 35 | 195.7 | 0.570 | 1.221 | 0.113 | 11.33 | 32.10 |
| 1996 | Madsen-2 | 36 | 273.2 | 0.517 | 1.150 | 0.106 | 10.61 | 34.39 |
| 1996 | Madsen-3 | 36 | 233.5 | 0.508 | 1.155 | 0.115 | 11.48 | 36.16 |
| 1996 | Madsen-4 | 36 | 206.5 | 0.510 | 1.164 | 0.113 | 11.32 | 36.26 |
| 1996 | Rod-1 | 40 | 185.7 | 0.533 | 1.178 | 0.108 | 10.76 | 34.28 |
| 1996 | Rod-2 | 38 | 152.4 | 0.496 | 1.130 | 0.111 | 11.12 | 37.09 |
| 1996 | Rod-3 | 38 | 166.6 | 0.526 | 1.205 | 0.098 | 9.81 | 34.98 |
| 1996 | Rod-4 | 37 | 197.5 | 0.492 | 1.102 | 0.107 | 10.74 | 38.78 |
| 1996 | Stephens-1 | 37 | 211.9 | 0.492 | 1.045 | 0.104 | 10.39 | 36.51 |
| 1996 | Stephens-2 | 36 | 196.6 | 0.456 | 0.994 | 0.092 | 9.16 | 39.63 |
| 1996 | Stephens-3 | 33 | 180.9 | 0.442 | 0.956 | 0.110 | 11.03 | 40.98 |
| 1996 | Stephens-4 | 35 | 197.7 | 0.443 | 0.978 | 0.091 | 9.15 | 41.12 |
| 1997 | Cashup-1 | | | 0.473 | 1.063 | 0.111 | 11.08 | 36.57 |
| 1997 | Cashup-2 | | | 0.463 | 1.023 | 0.101 | 10.07 | |
| 1997 | Cashup-3 | | | 0.411 | 0.956 | 0.112 | 11.22 | 43.10 |
| 1997 | Cashup-4 | | | 0.480 | 1.058 | 0.114 | 11.43 | 36.36 |
| 1997 | Eltan-1 | | | 0.465 | 1.118 | 0.112 | 11.19 | 41.65 |
| 1997 | Eltan-2 | | | 0.468 | 1.114 | 0.116 | 11.55 | 40.90 |
| 1997 | Eltan-3 | | | 0.483 | 1.174 | 0.122 | 12.19 | 40.91 |
| 1997 | Eltan-4 | | | 0.460 | 1.072 | 0.116 | 11.55 | 40.96 |
| 1997 | Lewjain-1 | | | 0.409 | 0.948 | 0.130 | 13.04 | 43.66 |
| 1997 | Lewjain-2 | | | 0.436 | 0.952 | 0.105 | 10.49 | 39.93 |
| 1997 | Lewjain-3 | | | 0.450 | 1.024 | 0.105 | 10.48 | 38.89 |
| 1997 | Lewjain-4 | | | 0.434 | 1.000 | 0.121 | 12.12 | 42.21 |
| 1997 | Madsen-1 | | | 0.525 | 1.201 | 0.109 | 10.86 | 35.98 |
| 1997 | Madsen-2 | | | 0.485 | 1.170 | 0.112 | 11.19 | 39.84 |
| 1997 | Madsen-3 | | | 0.493 | 1.182 | 0.136 | 13.57 | 38.66 |
| 1997 | Madsen-4 | | | 0.500 | 1.192 | 0.108 | 10.80 | 37.20 |
| 1997 | Rod-1 | | | 0.442 | 1.056 | 0.116 | 11.65 | 41.46 |
| 1997 | Rod-2 | | | 0.472 | 1.117 | 0.102 | 10.25 | 39.05 |
| 1997 | Rod-3 | | | 0.446 | 1.028 | 0.126 | 12.55 | 41.31 |
| 1997 | Rod-4 | | | 0.437 | 1.031 | 0.118 | 11.76 | 41.37 |
| 1997 | Stephens-1 | | | 0.400 | 0.967 | 0.110 | 11.01 | 44.78 |
| 1997 | Stephens-2 | | | 0.438 | 0.971 | 0.101 | 10.10 | 40.59 |
| 1997 | Stephens-3 | | | 0.433 | 0.955 | 0.096 | 9.64 | 40.58 |
| 1997 | Stephens-4 | | | 0.417 | 0.942 | 0.123 | 12.34 | 42.46 |

Table A.3. Raw Data: Pullman Plant and Fiber Results

| Harvest Year | Cultivar-Replicate | Plant Height (in.) | Stem Mass (g) | NAFL (mm) | WAFL (mm) | MWAFL (mm) | Coarseness (mg/100m) | Fines (%) |
|---|---|---|---|---|---|---|---|---|
| 1996 | Cashup-1 | 35 | 73.2 | 0.445 | 0.977 | 0.127 | 12.69 | 37.71 |
| 1996 | Cashup-2 | 33 | 79.8 | 0.519 | 1.099 | 0.125 | 12.48 | 35.43 |
| 1996 | Cashup-3 | 33 | 110.9 | 0.437 | 0.979 | 0.120 | 12.04 | 40.22 |
| 1996 | Cashup-4 | 36 | 72.1 | 0.497 | 1.112 | 0.103 | 10.28 | 35.57 |
| 1996 | Eltan-1 | 41 | 142.8 | 0.461 | 1.053 | 0.120 | 12.00 | 39.91 |
| 1996 | Eltan-2 | 41 | 125.8 | 0.464 | 1.049 | 0.125 | 12.48 | 39.86 |
| 1996 | Eltan-3 | 38 | 108.6 | 0.450 | 1.007 | 0.111 | 11.10 | 40.26 |
| 1996 | Eltan-4 | 40 | 112.7 | 0.443 | 1.002 | 0.133 | 13.25 | 41.03 |
| 1996 | Lewjain-1 | 32 | 89.6 | 0.398 | 0.918 | 0.114 | 11.37 | 44.30 |
| 1996 | Lewjain-2 | 37 | 148.8 | 0.423 | 0.968 | 0.117 | 11.69 | 42.25 |
| 1996 | Lewjain-3 | 36 | 143.7 | 0.374 | 0.849 | 0.114 | 11.39 | 46.16 |
| 1996 | Lewjain-4 | 33 | 103.9 | 0.412 | 0.903 | 0.132 | 13.16 | 42.19 |
| 1996 | Madsen-1 | . | 109.8 | 0.477 | 1.050 | 0.115 | 11.49 | 37.57 |
| 1996 | Madsen-2 | 34 | 116.4 | 0.476 | 1.005 | 0.128 | 12.82 | 36.53 |
| 1996 | Madsen-3 | 35 | 160.4 | 0.480 | 1.072 | 0.139 | 13.93 | 38.12 |
| 1996 | Madsen-4 | 35 | 124.4 | 0.467 | 1.025 | 0.123 | 12.34 | 38.72 |
| 1996 | Rod-1 | . | 90.0 | 0.448 | 1.033 | 0.130 | 12.98 | 41.29 |
| 1996 | Rod-2 | 41 | 162.9 | 0.448 | 0.991 | 0.128 | 12.78 | 39.70 |
| 1996 | Rod-3 | . | 118.6 | 0.460 | 1.018 | 0.119 | 11.91 | 38.88 |
| 1996 | Rod-4 | 37 | 138.2 | 0.453 | 0.991 | 0.097 | 9.72 | 39.18 |
| 1996 | Stephens-1 | 36 | 135.1 | 0.448 | 0.968 | 0.134 | 13.40 | 40.49 |
| 1996 | Stephens-2 | 35 | 89.1 | 0.408 | 0.913 | 0.136 | 13.65 | 43.96 |
| 1996 | Stephens-3 | 33 | 98.4 | 0.410 | 0.894 | 0.129 | 12.92 | 42.52 |
| 1996 | Stephens-4 | 36 | 121.9 | 0.440 | 0.931 | 0.139 | 13.93 | 39.30 |
| 1997 | Cashup-1 | | | 0.471 | 1.022 | 0.139 | 13.86 | 38.47 |
| 1997 | Cashup-2 | | | 0.505 | 1.051 | 0.126 | 12.55 | 35.31 |
| 1997 | Cashup-3 | | | 0.490 | 1.063 | 0.122 | 12.23 | 37.09 |
| 1997 | Cashup-4 | | | 0.537 | 1.168 | 0.095 | 9.47 | 33.00 |
| 1997 | Eltan-1 | | | 0.451 | 1.035 | 0.138 | 13.77 | 40.94 |
| 1997 | Eltan-2 | | | 0.498 | 1.161 | 0.135 | 13.50 | 38.09 |
| 1997 | Eltan-3 | | | 0.470 | 1.066 | 0.135 | 13.45 | 40.07 |
| 1997 | Eltan-4 | | | 0.474 | 1.108 | 0.112 | 11.23 | 39.76 |
| 1997 | Lewjain-1 | | | 0.407 | 0.901 | 0.133 | 13.34 | 43.70 |
| 1997 | Lewjain-2 | | | 0.422 | 0.920 | 0.128 | 12.77 | 41.50 |
| 1997 | Lewjain-3 | | | 0.451 | 0.991 | 0.128 | 12.79 | 40.47 |
| 1997 | Lewjain-4 | | | 0.456 | 0.947 | 0.137 | 13.68 | 38.47 |
| 1997 | Madsen-1 | | | 0.475 | 1.031 | 0.118 | 11.75 | 37.35 |
| 1997 | Madsen-2 | | | 0.497 | 1.105 | 0.127 | 12.71 | 36.94 |
| 1997 | Madsen-3 | | | 0.471 | 1.062 | 0.138 | 13.81 | 38.06 |
| 1997 | Madsen-4 | | | 0.493 | 1.051 | 0.123 | 12.29 | 35.19 |
| 1997 | Rod-1 | | | 0.463 | 1.056 | 0.126 | 12.59 | 40.38 |
| 1997 | Rod-2 | | | 0.455 | 1.026 | 0.128 | 12.77 | 39.94 |
| 1997 | Rod-3 | | | 0.464 | 1.070 | 0.120 | 11.99 | 40.08 |
| 1997 | Rod-4 | | | 0.498 | 1.114 | 0.123 | 12.28 | 37.45 |
| 1997 | Stephens-1 | | | 0.454 | 0.977 | 0.133 | 13.30 | 40.02 |
| 1997 | Stephens-2 | | | 0.452 | 0.982 | 0.124 | 12.35 | 39.84 |
| 1997 | Stephens-3 | | | 0.455 | 0.998 | 0.123 | 12.25 | 39.69 |
| 1997 | Stephens-4 | | | 0.450 | 0.993 | 0.122 | 12.19 | 40.56 |

Table A.4. Raw Data: Dusty Inorganics and Lignin Data

| Harvest Year | Cultivar-Replicate | Ash (%) | Acid Insoluble Ash [Silica] (%) | Acid Soluble Ash [K] (%)[1] | Klason Lignin (%) | Soluble Lignin (%) | Total Lignin (%)[2] |
|---|---|---|---|---|---|---|---|
| 1996 | Cashup-1 | 2.10 | 1.09 | 1.01 | 20.59 | | |
| 1996 | Cashup-2 | 2.90 | 1.20 | 1.69 | 20.21 | | |
| 1996 | Cashup-3 | 2.87 | 0.63 | 2.24 | 20.07 | 3.72 | 23.79 |
| 1996 | Cashup-4 | 2.75 | 0.70 | 2.06 | 21.29 | 3.32 | 24.61 |
| 1996 | Eltan-1 | 3.42 | 0.77 | 2.65 | 19.56 | 2.85 | 22.41 |
| 1996 | Eltan-2 | 3.84 | 1.41 | 2.42 | 17.25 | 4.13 | 21.38 |
| 1996 | Eltan-3 | 2.99 | 0.85 | 2.14 | 20.16 | 2.78 | 22.94 |
| 1996 | Eltan-4 | 3.29 | 1.16 | 2.13 | 20.59 | | |
| 1996 | Lewjain-1 | 3.20 | 0.71 | 2.49 | 20.06 | 2.34 | 22.39 |
| 1996 | Lewjain-2 | 2.40 | 0.60 | 1.80 | 24.47 | 3.57 | 28.04 |
| 1996 | Lewjain-3 | 2.63 | 1.06 | 1.58 | 20.02 | | |
| 1996 | Lewjain-4 | 3.45 | 1.09 | 2.35 | 21.42 | 2.58 | 23.99 |
| 1996 | Madsen-1 | 2.79 | 0.64 | 2.15 | 19.29 | 3.09 | 22.37 |
| 1996 | Madsen-2 | 2.63 | 0.70 | 1.93 | 18.84 | | |
| 1996 | Madsen-3 | 2.76 | 0.44 | 2.32 | 18.26 | | |
| 1996 | Madsen-4 | 3.34 | 0.89 | 2.45 | 20.44 | 2.31 | 22.75 |
| 1996 | Rod-1 | 3.18 | 0.56 | 2.63 | 18.41 | | |
| 1996 | Rod-2 | 3.37 | 0.70 | 2.67 | 19.19 | 2.88 | 22.07 |
| 1996 | Rod-3 | 2.82 | 1.07 | 1.74 | 20.47 | | |
| 1996 | Rod-4 | 3.12 | 0.83 | 2.28 | 20.99 | 3.56 | 24.55 |
| 1996 | Stephens-1 | 3.40 | 0.93 | 2.47 | 19.99 | | |
| 1996 | Stephens-2 | 2.89 | 1.17 | 1.72 | 20.44 | 3.75 | 24.19 |
| 1996 | Stephens-3 | 2.38 | 0.83 | 1.55 | 21.78 | 2.83 | 24.61 |
| 1996 | Stephens-4 | 4.00 | 0.72 | 3.29 | 19.21 | | |
| 1997 | Cashup-1 | | | | 21.89 | 2.29 | 24.18 |
| 1997 | Cashup-2 | 3.99 | 1.70 | 2.29 | 19.88 | 1.88 | 21.76 |
| 1997 | Cashup-3 | | | | 20.41 | | |
| 1997 | Cashup-4 | 3.46 | 2.10 | 1.36 | 20.74 | 2.40 | 23.14 |
| 1997 | Eltan-1 | 4.47 | 1.78 | 2.68 | 19.89 | | |
| 1997 | Eltan-2 | 4.89 | 2.22 | 2.67 | 21.69 | | |
| 1997 | Eltan-3 | 4.19 | 1.89 | 2.30 | 20.61 | 2.13 | 22.74 |
| 1997 | Eltan-4 | | | | 21.16 | 3.00 | 24.17 |
| 1997 | Lewjain-1 | | | | 20.40 | | |
| 1997 | Lewjain-2 | 5.01 | 1.79 | 3.22 | 19.91 | | |
| 1997 | Lewjain-3 | 4.53 | 1.74 | 2.80 | 20.41 | 2.36 | 22.77 |
| 1997 | Lewjain-4 | 3.25 | 1.86 | 1.39 | 20.19 | 2.50 | 22.69 |
| 1997 | Madsen-1 | 4.00 | 1.48 | 2.53 | 19.69 | | |
| 1997 | Madsen-2 | 3.25 | 1.50 | 1.75 | 20.46 | | |
| 1997 | Madsen-3 | 4.19 | 1.81 | 2.37 | 19.47 | | |
| 1997 | Madsen-4 | 3.22 | 1.62 | 1.61 | 20.75 | | |
| 1997 | Rod-1 | 3.76 | 1.20 | 2.56 | 20.14 | 2.27 | 22.42 |
| 1997 | Rod-2 | 3.83 | 1.70 | 2.14 | 21.00 | | |
| 1997 | Rod-3 | 4.33 | 2.56 | 1.77 | 20.79 | | |
| 1997 | Rod-4 | 2.32 | 1.27 | 1.05 | 20.87 | 2.89 | 23.77 |
| 1997 | Stephens-1 | 4.52 | 1.61 | 2.91 | 17.81 | | |
| 1997 | Stephens-2 | | | | 19.80 | | |
| 1997 | Stephens-3 | | | | 19.99 | | |
| 1997 | Stephens-4 | 3.08 | 0.92 | 2.16 | 20.18 | 2.44 | 22.62 |

[1] Acid Soluble Ash = Ash - Acid Insoluble Ash [2] Total Lignin = Soluble Lignin + Klason Lignin

Table A.5. Raw Data: Moses Lake Inorganics and Lignin Data

| Harvest Year | Cultivar-Replicate | Ash (%) | Acid Insoluble Ash [Silica] (%) | Acid Soluble Ash [K] (%)[1] | Klason Lignin (%) | Soluble Lignin (%) | Total Lignin (%)[2] |
|---|---|---|---|---|---|---|---|
| 1996 | Cashup-1 | 6.90 | 3.42 | 3.48 | 18.65 | | |
| 1996 | Cashup-2 | 6.67 | 4.09 | 2.58 | 18.00 | | |
| 1996 | Cashup-3 | 7.90 | 4.13 | 3.77 | 19.44 | 2.88 | 22.32 |
| 1996 | Cashup-4 | 6.14 | 3.79 | 2.35 | 19.41 | | |
| 1996 | Eltan-1 | 4.22 | 1.36 | 2.86 | 19.79 | | |
| 1996 | Eltan-2 | 6.51 | 3.09 | 3.42 | 19.88 | 3.05 | 22.93 |
| 1996 | Eltan-3 | 7.26 | 3.10 | 4.16 | 17.76 | | |
| 1996 | Eltan-4 | 7.13 | 2.90 | 4.23 | 18.91 | 2.47 | 21.38 |
| 1996 | Lewjain-1 | 7.32 | 2.48 | 4.83 | 19.65 | 3.27 | 22.92 |
| 1996 | Lewjain-2 | 7.44 | 4.36 | 3.09 | 19.44 | 3.18 | 22.62 |
| 1996 | Lewjain-3 | 7.32 | 4.09 | 3.23 | 18.83 | 3.84 | 22.67 |
| 1996 | Lewjain-4 | 7.16 | 3.26 | 3.90 | 17.69 | | |
| 1996 | Madsen-1 | 5.59 | 2.24 | 3.35 | 19.12 | | |
| 1996 | Madsen-2 | 6.35 | 3.25 | 3.09 | 20.00 | | |
| 1996 | Madsen-3 | 6.38 | 2.93 | 3.45 | 19.28 | | |
| 1996 | Madsen-4 | 5.89 | 2.33 | 3.55 | 19.53 | 3.52 | 23.06 |
| 1996 | Rod-1 | 5.10 | 1.79 | 3.31 | 21.19 | 3.03 | 24.21 |
| 1996 | Rod-2 | 4.88 | 1.81 | 3.07 | 21.76 | 2.95 | 24.71 |
| 1996 | Rod-3 | 6.10 | 3.23 | 2.87 | 21.10 | 2.69 | 23.79 |
| 1996 | Rod-4 | 5.76 | 2.44 | 3.32 | 18.99 | | |
| 1996 | Stephens-1 | 6.80 | 2.35 | 4.45 | 19.88 | | |
| 1996 | Stephens-2 | 6.40 | 2.97 | 3.43 | 19.82 | 3.44 | 23.26 |
| 1996 | Stephens-3 | 5.32 | 1.86 | 3.47 | 19.73 | | |
| 1996 | Stephens-4 | 5.38 | 2.12 | 3.26 | 19.61 | | |
| 1997 | Cashup-1 | 9.12 | 3.81 | 5.31 | 19.89 | | |
| 1997 | Cashup-2 | | | | 18.75 | 2.87 | 21.62 |
| 1997 | Cashup-3 | 8.71 | 3.90 | 4.81 | 17.88 | | |
| 1997 | Cashup-4 | 8.19 | 3.48 | 4.71 | 17.52 | | |
| 1997 | Eltan-1 | 7.64 | 1.88 | 5.76 | 19.98 | | |
| 1997 | Eltan-2 | 8.66 | 2.23 | 6.43 | 20.44 | 2.53 | 22.97 |
| 1997 | Eltan-3 | 9.69 | 2.70 | 6.99 | 18.59 | 3.48 | 22.08 |
| 1997 | Eltan-4 | 9.01 | 3.33 | 5.69 | 19.52 | 2.28 | 21.80 |
| 1997 | Lewjain-1 | 7.97 | 2.01 | 5.95 | 17.43 | 3.82 | 21.25 |
| 1997 | Lewjain-2 | 10.38 | 2.33 | 8.05 | 17.58 | | |
| 1997 | Lewjain-3 | | | | 17.48 | | |
| 1997 | Lewjain-4 | 9.85 | 2.73 | 7.12 | 18.23 | | |
| 1997 | Madsen-1 | 9.83 | 2.52 | 7.32 | 17.79 | 3.56 | 21.35 |
| 1997 | Madsen-2 | 10.23 | 3.75 | 6.49 | 19.26 | 2.92 | 22.17 |
| 1997 | Madsen-3 | 9.21 | 2.77 | 6.44 | 17.98 | | |
| 1997 | Madsen-4 | 6.24 | 2.47 | 3.77 | 18.28 | | |
| 1997 | Rod-1 | 9.67 | 2.13 | 7.54 | 19.15 | 3.45 | 22.59 |
| 1997 | Rod-2 | | | | 18.64 | | |
| 1997 | Rod-3 | 7.41 | 3.13 | 4.28 | 18.95 | | |
| 1997 | Rod-4 | 8.45 | 2.93 | 5.52 | 19.10 | | |
| 1997 | Stephens-1 | 8.93 | 2.19 | 6.75 | 18.16 | 2.97 | 21.14 |
| 1997 | Stephens-2 | 7.77 | 2.62 | 5.15 | 19.35 | | |
| 1997 | Stephens-3 | 9.49 | 3.08 | 6.41 | 17.76 | | |
| 1997 | Stephens-4 | 7.58 | 2.27 | 5.31 | 16.80 | 3.77 | 20.57 |

[1] Acid Soluble Ash = Ash - Acid Insoluble Ash   [2] Total Lignin = Soluble Lignin + Klason Lignin

Table A.6. Raw Data: Pullman Inorganics and Lignin Data

| Harvest Year | Cultivar-Replicate | Ash (%) | Acid Insoluble Ash [Silica] (%) | Acid Soluble Ash [K] (%)[1] | Klason Lignin (%) | Soluble Lignin (%) | Total Lignin (%)[2] |
|---|---|---|---|---|---|---|---|
| 1996 | Cashup-1 | 5.39 | 3.02 | 2.37 | 19.76 | | |
| 1996 | Cashup-2 | 7.34 | 5.51 | 1.83 | 20.27 | 2.94 | 23.22 |
| 1996 | Cashup-3 | 5.72 | 4.50 | 1.22 | 20.86 | 1.56 | 22.42 |
| 1996 | Cashup-4 | | | | 20.24 | | |
| 1996 | Eltan-1 | 5.11 | 2.29 | 2.83 | 20.02 | | |
| 1996 | Eltan-2 | 5.31 | 2.48 | 2.82 | 20.19 | 2.42 | 22.61 |
| 1996 | Eltan-3 | 4.41 | 2.74 | 1.67 | 20.49 | 2.11 | 22.60 |
| 1996 | Eltan-4 | 4.41 | 2.09 | 2.32 | 20.33 | 1.26 | 21.59 |
| 1996 | Lewjain-1 | 5.03 | 3.27 | 1.76 | 20.19 | 2.39 | 22.59 |
| 1996 | Lewjain-2 | 4.39 | 2.23 | 2.16 | 21.42 | | |
| 1996 | Lewjain-3 | 3.95 | 1.89 | 2.06 | 20.23 | 2.60 | 22.83 |
| 1996 | Lewjain-4 | 3.73 | 1.64 | 2.09 | 20.77 | 2.21 | 22.98 |
| 1996 | Madsen-1 | 4.27 | 2.44 | 1.83 | 19.97 | 2.19 | 22.16 |
| 1996 | Madsen-2 | 3.86 | 2.79 | 1.08 | 19.31 | | |
| 1996 | Madsen-3 | 3.47 | 2.45 | 1.02 | 20.55 | | |
| 1996 | Madsen-4 | 4.13 | 2.43 | 1.70 | 20.81 | 1.31 | 22.12 |
| 1996 | Rod-1 | 5.43 | 3.92 | 1.52 | 21.09 | | |
| 1996 | Rod-2 | 3.44 | 2.46 | 0.97 | 21.52 | 2.52 | 24.03 |
| 1996 | Rod-3 | 3.69 | 2.73 | 0.97 | 19.39 | | |
| 1996 | Rod-4 | 3.47 | 2.65 | 0.82 | 20.91 | 2.22 | 23.14 |
| 1996 | Stephens-1 | 4.66 | 2.67 | 1.98 | 20.90 | 1.74 | 22.65 |
| 1996 | Stephens-2 | 4.91 | 2.85 | 2.06 | 20.66 | 2.63 | 23.28 |
| 1996 | Stephens-3 | 2.84 | 1.80 | 1.04 | 21.70 | 2.34 | 24.04 |
| 1996 | Stephens-4 | 4.47 | 3.07 | 1.40 | 19.15 | | |
| 1997 | Cashup-1 | 5.58 | 4.15 | 1.43 | 21.04 | 2.31 | 23.35 |
| 1997 | Cashup-2 | 4.72 | 3.32 | 1.41 | 20.24 | | |
| 1997 | Cashup-3 | 6.81 | 4.92 | 1.89 | 20.38 | | |
| 1997 | Cashup-4 | 5.87 | 3.82 | 2.05 | 20.72 | 2.27 | 22.99 |
| 1997 | Eltan-1 | 5.36 | 3.20 | 2.16 | 20.62 | 2.67 | 23.29 |
| 1997 | Eltan-2 | 5.03 | 2.49 | 2.54 | 21.00 | 2.62 | 23.61 |
| 1997 | Eltan-3 | 5.60 | 2.88 | 2.72 | 21.19 | 2.27 | 23.47 |
| 1997 | Eltan-4 | 5.09 | 2.34 | 2.75 | 21.09 | 2.54 | 23.63 |
| 1997 | Lewjain-1 | 3.79 | 2.28 | 1.51 | 20.81 | | |
| 1997 | Lewjain-2 | 4.30 | 2.44 | 1.87 | 20.89 | | |
| 1997 | Lewjain-3 | 4.88 | 2.31 | 2.57 | 20.92 | 2.93 | 23.84 |
| 1997 | Lewjain-4 | 5.46 | 2.40 | 3.06 | 20.63 | 2.68 | 23.31 |
| 1997 | Madsen-1 | 3.94 | 2.37 | 1.57 | 19.64 | 1.63 | 21.27 |
| 1997 | Madsen-2 | 3.59 | 2.32 | 1.27 | 21.42 | 2.83 | 24.25 |
| 1997 | Madsen-3 | 4.21 | 2.44 | 1.77 | 20.90 | 2.14 | 23.03 |
| 1997 | Madsen-4 | 5.03 | 3.27 | 1.76 | 21.32 | | |
| 1997 | Rod-1 | 5.04 | 3.62 | 1.42 | 21.42 | 2.38 | 23.81 |
| 1997 | Rod-2 | 3.91 | 2.62 | 1.29 | 21.86 | 2.44 | 24.29 |
| 1997 | Rod-3 | 5.57 | 3.67 | 1.90 | 21.67 | 2.60 | 24.27 |
| 1997 | Rod-4 | 5.27 | 2.43 | 2.84 | 21.36 | 2.68 | 24.04 |
| 1997 | Stephens-1 | 6.01 | 3.70 | 2.31 | 21.32 | 2.43 | 23.75 |
| 1997 | Stephens-2 | 5.29 | 2.40 | 2.89 | 21.34 | 2.69 | 24.03 |
| 1997 | Stephens-3 | 5.30 | 3.00 | 2.30 | 20.06 | | |
| 1997 | Stephens-4 | 4.95 | 2.51 | 2.44 | 21.30 | 2.48 | 23.78 |

[1] Acid Soluble Ash = Ash - Acid Insoluble Ash [2] Total Lignin = Soluble Lignin + Klason Lignin

Table A.7. Raw Data: Dusty Carbohydrate Data

| Harvest Year | Cultivar-Replicate | Cellulose (Glucan), % | Arabinan, % | Xylan, % | Mannan, % | Galactan, % | Hemicel-lulose, %[1] |
|---|---|---|---|---|---|---|---|
| 1996 | Cashup-1 | 40.9 | 2.3 | 22.9 | 0.6 | 0.8 | 26.5 |
| 1996 | Cashup-2 | 37.0 | 2.6 | 20.2 | 0.7 | 0.8 | 24.3 |
| 1996 | Cashup-3 | 35.3 | 2.1 | 18.1 | 1.4 | 0.7 | 22.3 |
| 1996 | Cashup-4 | 40.2 | 2.1 | 23.6 | 0.5 | 0.6 | 26.8 |
| 1996 | Eltan-1 | 36.0 | 2.2 | 19.2 | 0.8 | 0.9 | 23.1 |
| 1996 | Eltan-2 | 34.4 | 1.8 | 16.8 | 2.1 | 0.7 | 21.4 |
| 1996 | Eltan-3 | 38.0 | 2.1 | 20.1 | 0.9 | 0.7 | 23.8 |
| 1996 | Eltan-4 | 35.9 | 2.6 | 18.5 | 0.6 | 1.0 | 22.6 |
| 1996 | Lewjain-1 | 35.5 | 2.0 | 18.8 | 0.7 | 0.7 | 22.2 |
| 1996 | Lewjain-2 | 36.5 | 1.9 | 20.0 | 0.5 | 0.7 | 23.1 |
| 1996 | Lewjain-3 | 29.5 | 2.4 | 18.4 | 0.8 | 0.7 | 22.3 |
| 1996 | Lewjain-4 | 37.1 | 2.3 | 20.8 | 0.6 | 0.7 | 24.5 |
| 1996 | Madsen-1 | 38.4 | 1.6 | 16.9 | 1.7 | 0.4 | 20.5 |
| 1996 | Madsen-2 | 34.3 | 2.5 | 17.7 | 1.0 | 0.9 | 22.1 |
| 1996 | Madsen-3 | 31.2 | 2.1 | 15.3 | 1.1 | 0.7 | 19.2 |
| 1996 | Madsen-4 | 42.0 | 2.1 | 21.2 | 1.0 | 0.6 | 24.9 |
| 1996 | Rod-1 | 38.5 | 1.8 | 15.3 | 1.7 | 0.8 | 19.7 |
| 1996 | Rod-2 | 36.0 | 2.6 | 20.1 | 0.5 | 0.8 | 24.0 |
| 1996 | Rod-3 | 47.5 | 1.2 | 24.0 | 0.7 | 1.0 | 26.9 |
| 1996 | Rod-4 | 36.7 | 2.1 | 20.1 | 1.0 | 0.7 | 23.8 |
| 1996 | Stephens-1 | 37.4 | 2.1 | 21.3 | 0.7 | 0.7 | 24.7 |
| 1996 | Stephens-2 | 42.0 | 2.1 | 23.4 | 0.3 | 0.5 | 26.4 |
| 1996 | Stephens-3 | 36.2 | 2.5 | 22.0 | 0.4 | 0.7 | 25.6 |
| 1996 | Stephens-4 | 27.2 | 2.2 | 15.3 | 1.4 | 0.8 | 19.6 |
| 1997 | Cashup-1 | 45.0 | 2.1 | 24.5 | 0.3 | 0.6 | 27.5 |
| 1997 | Cashup-2 | 39.9 | 2.2 | 22.2 | 0.3 | 0.5 | 25.2 |
| 1997 | Cashup-3 | 45.0 | 2.5 | 21.9 | 0.4 | 0.9 | 25.7 |
| 1997 | Cashup-4 | 43.2 | 2.1 | 24.0 | 0.3 | 0.5 | 26.8 |
| 1997 | Eltan-1 | 42.1 | 2.3 | 19.6 | 0.8 | 1.0 | 23.7 |
| 1997 | Eltan-2 | 40.4 | 2.5 | 19.3 | 0.3 | 0.9 | 23.0 |
| 1997 | Eltan-3 | 38.8 | 2.2 | 21.1 | 0.5 | 0.7 | 24.5 |
| 1997 | Eltan-4 | 40.6 | 2.3 | 23.1 | 0.3 | 0.7 | 26.4 |
| 1997 | Lewjain-1 | 35.2 | 2.1 | 17.7 | 0.3 | 0.7 | 20.7 |
| 1997 | Lewjain-2 | 39.8 | 2.4 | 22.6 | 0.3 | 0.7 | 25.9 |
| 1997 | Lewjain-3 | 39.8 | 2.1 | 21.2 | 0.4 | 0.8 | 24.6 |
| 1997 | Lewjain-4 | 40.3 | 2.1 | 23.7 | 0.5 | 0.7 | 27.0 |
| 1997 | Madsen-1 | 47.0 | 2.1 | 20.1 | 0.5 | 0.6 | 23.3 |
| 1997 | Madsen-2 | 41.8 | 2.4 | 22.1 | 0.5 | 0.7 | 25.6 |
| 1997 | Madsen-3 | 41.5 | 2.2 | 20.0 | 0.5 | 0.6 | 23.3 |
| 1997 | Madsen-4 | 34.9 | 2.1 | 19.0 | 0.4 | 0.5 | 21.9 |
| 1997 | Rod-1 | 37.9 | 2.0 | 21.4 | 0.3 | 0.4 | 24.2 |
| 1997 | Rod-2 | 30.7 | 2.0 | 18.7 | 0.4 | 0.4 | 21.6 |
| 1997 | Rod-3 | 42.7 | 2.1 | 22.6 | 0.7 | 0.8 | 26.1 |
| 1997 | Rod-4 | 39.6 | 2.3 | 23.0 | 0.4 | 0.7 | 26.3 |
| 1997 | Stephens-1 | 39.8 | 2.5 | 22.4 | 0.4 | 0.6 | 25.9 |
| 1997 | Stephens-2 | 38.6 | 2.5 | 23.1 | 0.4 | 0.6 | 26.5 |
| 1997 | Stephens-3 | 45.7 | 3.0 | 22.5 | 1.5 | 1.3 | 28.2 |
| 1997 | Stephens-4 | 42.9 | 2.4 | 26.2 | 0.3 | 0.6 | 29.5 |

[1] Calculated as Hemicellulose = Galactan + Mannan + Arabinan + Xylan

Table A.8. Raw Data: Moses Lake Carbohydrate Data

| Harvest Year | Cultivar-Replicate | Cellulose (Glucan), % | Arabinan, % | Xylan, % | Mannan, % | Galactan, % | Hemicellulose, %[1] |
|---|---|---|---|---|---|---|---|
| 1996 | Cashup-1 | 42.0 | 2.0 | 19.9 | 0.3 | 0.5 | 22.7 |
| 1996 | Cashup-2 | 38.0 | 2.0 | 22.2 | 0.2 | 0.4 | 24.7 |
| 1996 | Cashup-3 | 29.3 | 1.6 | 17.5 | 0.1 | 0.2 | 19.4 |
| 1996 | Cashup-4 | 43.5 | 1.8 | 22.5 | 0.2 | 0.4 | 25.0 |
| 1996 | Eltan-1 | 46.0 | 2.3 | 22.6 | 0.4 | 0.8 | 26.0 |
| 1996 | Eltan-2 | 36.6 | 2.1 | 19.8 | 0.2 | 0.4 | 22.5 |
| 1996 | Eltan-3 | 35.6 | 1.9 | 21.5 | 0.3 | 0.6 | 24.2 |
| 1996 | Eltan-4 | 41.6 | 1.8 | 22.4 | 0.2 | 0.4 | 24.8 |
| 1996 | Lewjain-1 | 41.2 | 1.9 | 21.9 | 0.1 | 0.5 | 24.5 |
| 1996 | Lewjain-2 | 40.9 | 1.9 | 21.3 | 0.3 | 0.5 | 24.0 |
| 1996 | Lewjain-3 | 39.5 | 2.1 | 21.4 | 0.2 | 0.5 | 24.1 |
| 1996 | Lewjain-4 | 36.6 | 2.0 | 21.0 | 0.2 | 0.4 | 23.6 |
| 1996 | Madsen-1 | 46.7 | 2.0 | 21.5 | 0.3 | 0.7 | 24.4 |
| 1996 | Madsen-2 | 41.8 | 2.0 | 18.3 | 0.3 | 0.6 | 21.2 |
| 1996 | Madsen-3 | 42.5 | 1.9 | 21.5 | 0.2 | 0.4 | 23.9 |
| 1996 | Madsen-4 | 40.2 | 1.8 | 21.4 | 0.2 | 0.5 | 23.8 |
| 1996 | Rod-1 | 43.5 | 2.1 | 24.2 | 0.4 | 0.5 | 27.2 |
| 1996 | Rod-2 | 38.9 | 1.9 | 21.9 | 0.2 | 0.5 | 24.5 |
| 1996 | Rod-3 | 39.3 | 1.7 | 21.3 | 0.2 | 0.4 | 23.6 |
| 1996 | Rod-4 | 46.6 | 2.4 | 22.3 | 0.4 | 0.9 | 25.9 |
| 1996 | Stephens-1 | 42.5 | 2.1 | 20.7 | 0.3 | 0.6 | 23.8 |
| 1996 | Stephens-2 | 40.6 | 1.7 | 21.8 | 0.2 | 0.4 | 24.1 |
| 1996 | Stephens-3 | 49.0 | 2.4 | 24.5 | 0.4 | 0.9 | 28.3 |
| 1996 | Stephens-4 | 40.1 | 2.4 | 21.9 | 0.5 | 0.7 | 25.4 |
| 1997 | Cashup-1 | 42.1 | 2.0 | 18.5 | 0.4 | 0.7 | 21.5 |
| 1997 | Cashup-2 | 44.3 | 1.9 | 23.1 | 0.1 | 0.5 | 25.6 |
| 1997 | Cashup-3 | 44.3 | 1.9 | 22.1 | 0.2 | 0.5 | 24.7 |
| 1997 | Cashup-4 | 41.3 | 1.9 | 19.5 | 0.3 | 0.6 | 22.3 |
| 1997 | Eltan-1 | 36.1 | 1.6 | 18.6 | 0.1 | 0.4 | 20.7 |
| 1997 | Eltan-2 | 36.9 | 1.7 | 18.3 | 0.1 | 0.3 | 20.5 |
| 1997 | Eltan-3 | 39.4 | 1.9 | 22.1 | 0.2 | 0.5 | 24.6 |
| 1997 | Eltan-4 | 40.1 | 1.6 | 20.4 | 0.2 | 0.4 | 22.6 |
| 1997 | Lewjain-1 | 37.8 | 1.7 | 19.7 | 0.1 | 0.4 | 22.0 |
| 1997 | Lewjain-2 | 33.7 | 2.0 | 19.7 | 0.2 | 0.4 | 22.2 |
| 1997 | Lewjain-3 | 45.2 | 2.3 | 21.2 | 0.3 | 0.8 | 24.5 |
| 1997 | Lewjain-4 | 38.1 | 1.8 | 19.8 | 0.2 | 0.4 | 22.2 |
| 1997 | Madsen-1 | 42.4 | 1.9 | 21.7 | 0.2 | 0.5 | 24.3 |
| 1997 | Madsen-2 | 44.9 | 1.7 | 20.8 | 0.2 | 0.4 | 23.1 |
| 1997 | Madsen-3 | 37.5 | 1.8 | 20.8 | 0.1 | 0.3 | 23.0 |
| 1997 | Madsen-4 | 37.9 | 1.8 | 17.5 | 0.3 | 0.5 | 20.0 |
| 1997 | Rod-1 | 39.8 | 1.8 | 21.5 | 0.1 | 0.5 | 23.8 |
| 1997 | Rod-2 | 37.8 | 2.3 | 18.1 | 0.2 | 0.6 | 21.2 |
| 1997 | Rod-3 | 42.6 | 2.2 | 20.5 | 0.3 | 0.7 | 23.7 |
| 1997 | Rod-4 | 34.5 | 1.7 | 18.4 | 0.1 | 0.3 | 20.4 |
| 1997 | Stephens-1 | 41.8 | 2.0 | 22.5 | 0.2 | 0.5 | 25.2 |
| 1997 | Stephens-2 | 31.2 | 1.8 | 17.5 | 0.2 | 0.5 | 19.9 |
| 1997 | Stephens-3 | 43.2 | 2.1 | 20.0 | 0.3 | 0.7 | 23.0 |
| 1997 | Stephens-4 | 41.0 | 1.7 | 21.7 | 0.2 | 0.4 | 24.0 |

[1] Calculated as Hemicellulose = Galactan + Mannan + Arabinan + Xylan

Table A.9. Raw Data: Pullman Carbohydrate Data

| Harvest Year | Cultivar-Replicate | Cellulose (Glucan), % | Arabinan, % | Xylan, % | Mannan, % | Galactan, % | Hemicellulose, %[1] |
|---|---|---|---|---|---|---|---|
| 1996 | Cashup-1 | 44.4 | 2.4 | 22.2 | 0.3 | 0.8 | 25.7 |
| 1996 | Cashup-2 | 39.8 | 1.9 | 22.3 | 0.1 | 0.5 | 24.7 |
| 1996 | Cashup-3 | 39.5 | 2.1 | 22.1 | 0.2 | 0.4 | 24.8 |
| 1996 | Cashup-4 | 42.6 | 2.5 | 22.6 | 0.3 | 0.8 | 26.2 |
| 1996 | Eltan-1 | 32.8 | 2.0 | 20.4 | 0.5 | 0.5 | 23.4 |
| 1996 | Eltan-2 | 41.5 | 1.9 | 21.4 | 0.7 | 0.5 | 24.5 |
| 1996 | Eltan-3 | 39.0 | 2.1 | 21.5 | 0.6 | 0.5 | 24.8 |
| 1996 | Eltan-4 | 42.0 | 2.2 | 22.8 | 0.6 | 0.7 | 26.2 |
| 1996 | Lewjain-1 | 41.0 | 2.1 | 22.9 | 0.3 | 0.5 | 25.8 |
| 1996 | Lewjain-2 | 42.6 | 2.8 | 22.5 | 0.4 | 1.1 | 26.8 |
| 1996 | Lewjain-3 | 39.8 | 2.3 | 25.0 | 0.3 | 0.5 | 28.1 |
| 1996 | Lewjain-4 | 35.7 | 2.0 | 19.6 | 0.9 | 0.7 | 23.2 |
| 1996 | Madsen-1 | 43.8 | 1.9 | 22.2 | 0.3 | 0.4 | 24.8 |
| 1996 | Madsen-2 | 44.9 | 2.3 | 22.6 | 0.6 | 0.8 | 26.2 |
| 1996 | Madsen-3 | 39.0 | 2.0 | 21.6 | 0.4 | 0.4 | 24.3 |
| 1996 | Madsen-4 | 37.9 | 2.0 | 19.9 | 0.4 | 0.4 | 22.6 |
| 1996 | Rod-1 | 41.6 | 1.9 | 23.0 | 0.2 | 0.4 | 25.5 |
| 1996 | Rod-2 | 42.3 | 2.1 | 23.5 | 0.3 | 0.5 | 26.4 |
| 1996 | Rod-3 | 39.1 | 1.8 | 21.4 | 0.3 | 0.5 | 24.0 |
| 1996 | Rod-4 | 42.0 | 2.1 | 23.9 | 0.3 | 0.5 | 26.9 |
| 1996 | Stephens-1 | 39.5 | 1.9 | 21.9 | 0.3 | 0.3 | 24.4 |
| 1996 | Stephens-2 | 40.4 | 2.0 | 22.2 | 0.2 | 0.3 | 24.6 |
| 1996 | Stephens-3 | 40.6 | 2.4 | 24.3 | 0.4 | 0.7 | 27.7 |
| 1996 | Stephens-4 | 36.8 | 2.0 | 21.6 | 0.6 | 0.6 | 24.9 |
| 1997 | Cashup-1 | 38.7 | 1.9 | 20.1 | 0.5 | 0.5 | 23.0 |
| 1997 | Cashup-2 | 43.5 | 1.9 | 24.1 | 0.3 | 0.3 | 26.6 |
| 1997 | Cashup-3 | 42.9 | 1.8 | 22.3 | 0.6 | 0.4 | 25.2 |
| 1997 | Cashup-4 | 40.6 | 1.8 | 21.9 | 0.3 | 0.3 | 24.4 |
| 1997 | Eltan-1 | 42.5 | 2.0 | 22.1 | 0.9 | 0.4 | 25.4 |
| 1997 | Eltan-2 | 42.2 | 1.7 | 22.5 | 1.0 | 0.4 | 25.7 |
| 1997 | Eltan-3 | 38.3 | 2.0 | 21.1 | 1.0 | 0.5 | 24.6 |
| 1997 | Eltan-4 | 36.2 | 1.7 | 19.0 | 1.4 | 0.4 | 22.5 |
| 1997 | Lewjain-1 | 47.7 | 2.6 | 22.6 | 1.4 | 1.0 | 27.6 |
| 1997 | Lewjain-2 | 36.5 | 1.8 | 21.7 | 1.2 | 0.4 | 25.1 |
| 1997 | Lewjain-3 | 42.3 | 2.2 | 24.1 | 0.3 | 0.5 | 27.1 |
| 1997 | Lewjain-4 | 40.8 | 1.8 | 24.6 | 1.0 | 0.4 | 27.8 |
| 1997 | Madsen-1 | 40.7 | 1.9 | 22.4 | 0.5 | 0.3 | 25.1 |
| 1997 | Madsen-2 | 44.7 | 1.9 | 22.6 | 0.3 | 0.4 | 25.2 |
| 1997 | Madsen-3 | 44.7 | 1.9 | 23.0 | 0.3 | 0.4 | 25.7 |
| 1997 | Madsen-4 | 39.4 | 1.7 | 19.8 | 0.5 | 0.5 | 22.6 |
| 1997 | Rod-1 | 30.4 | 1.8 | 19.6 | 0.4 | 0.4 | 22.2 |
| 1997 | Rod-2 | 42.4 | 1.9 | 23.9 | 0.8 | 0.4 | 27.0 |
| 1997 | Rod-3 | 39.6 | 2.0 | 21.9 | 1.0 | 0.6 | 25.5 |
| 1997 | Rod-4 | 43.1 | 1.8 | 22.5 | 0.9 | 0.3 | 25.5 |
| 1997 | Stephens-1 | 42.7 | 1.9 | 23.0 | 0.6 | 0.4 | 25.9 |
| 1997 | Stephens-2 | 37.2 | 1.8 | 20.3 | 1.2 | 0.4 | 23.7 |
| 1997 | Stephens-3 | 26.3 | 2.0 | 16.7 | 0.7 | 0.5 | 19.8 |
| 1997 | Stephens-4 | 43.2 | 1.7 | 22.4 | 1.0 | 0.4 | 25.6 |

[1] Calculated as Hemicellulose = Galactan + Mannan + Arabinan + Xylan

# Biographical Note

Roberta S. Jacobs was born in Spokane, Washington and graduated from Columbia River High School in Vancouver, Washington. She obtained a Bachelor of Science degree in Pulp and Paper Science and Engineering from the University of Washington in 1992 and a Master of Science degree in Chemical Engineering from the University of Maine in 1995.

# IMAGE EVALUATION TEST TARGET (QA-3)

150mm

6"