

A preliminary assessment of flax fibre quality during mechanical processing using near infrared spectroscopy

G.J. Faughey^a and H.S.S. Sharma^{a and b}

^aDepartment of Applied Plant Science, School of Agriculture and Food Science, The Queen's University of Belfast and ^bApplied Plant Science Division, Department of Agriculture and Rural Development for Northern Ireland, Newforge Lane, Belfast BT9 5PX, N. Ireland, UK.

Introduction

Many linen spinners purchase their stock fibres as hackled bales of flax which is in the form of a continuous sliver of long fibres. In the hackling process, the fibres are combed out mechanically to remove debris and short fibres left after scutching of the retted flax straw. The hackled stock fibres are blended through the four drawing frames (Figure 1), followed by the roving stage.

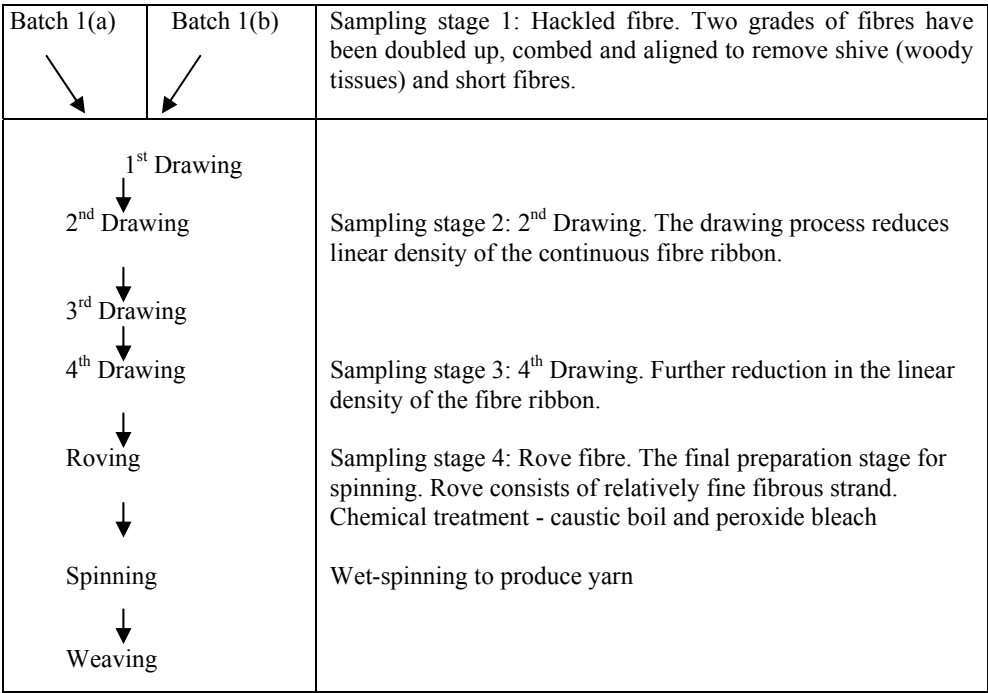


Figure 1. Fibre preparation stages for wet spinning of linen yarn.

After caustic boil and peroxide bleaching treatments, the roves are wet-spun to produce yarn.¹ The objective of flax preparation is to blend and level the continuous slivers to produce a

homogeneous rove suitable for spinning. The process of drawing out the fibre separates the bundles allowing the ultimate fibres to slip past one another to produce a variety of dimensions and degrees of aggregation depending largely on the polysaccharide composition of the fibre.² Major constituents of retted fibre are cellulose (65%), hemicellulose (14–17%), pectin (8–11%), lignin (1–4%), wax (2%), and ash (1–2%).³

Considerable insight into the peculiarities surrounding flax fibre processing has been gained from work carried out in Ireland during the mid 1940s.¹ Unpublished data have shown that the spinning quality of fibre can be judged by the degree of fineness and ease of splitting the fibre bundles during hackling. However, much of the work carried out resided in physical and chemical determinations, which were both tedious and time consuming. Furthermore complex mathematical formulae were used to evaluate relationships and predict responses.^{1,4}

This paper describes a preliminary investigation to evaluate the use of near infrared (NIR) spectroscopy for relating spectral with physical and chemical fibre properties during mechanical processing. Introductory work on the use of NIR for predicting important fibre parameters was recently reported.⁵

Materials and methods

Samples used in this study were obtained from the preparation lines of Herdmans (Sion Mills, Co Tyrone, N. Ireland) during a four-week period. The two hackled batches labelled as 1(a) and 1(b) were dew-retted fibres from Normandy and imported from Belgium (Figure 1). The materials (2 kg × 3 replicates) were removed at the beginning, 2nd, 4th drawing and roving stages before being stored in sealed plastic bags. Samples were not taken from the 1st and 3rd drawing stages. The samples (50) were conditioned and assessed in standard atmosphere for testing textiles (65% relative humidity and 20°C).⁵

Physical parameters

Fibre fineness determinations were carried out using a WIRA fibre fineness meter, which had been modified for flax.⁵ Fibre strength determinations were made using a Testometric (Model M250–2.5kN), fixed with a 10 kg load cell.⁵

Sample preparation and chemical analyses

Three sub-samples from each sampling stage were prepared for analysis by cutting to a fibre length of < 1cm prior to being ground using a Cyclotec mill (Foss Tecator 1093). Each sample was analysed for acid detergent fibre (ADF) and neutral detergent fibre (NDF) fractions by refluxing in detergent solutions.⁶ Lignin content was determined by hydrolysis using sulphuric acid followed by refluxing in ADF solution.⁷ Lipid assessments were carried out in accordance with methods recommended by the Society of Analytical Chemists⁸ and caustic weight loss (CWL) was determined by sodium hydroxide boil.⁹ Hemicellulose content was calculated as the difference between NDF and ADF determinations. Assessment of inorganic mineral content was performed in accordance with methods recommended by the Society of Analytical Chemists.⁸ Carbon and nitrogen analysis was carried out using an elemental analyser. All analyses were carried out in triplicate and results were assessed using analysis of variance.

Near infrared spectroscopy

Fibre samples were carefully teased apart and placed in a natural product sample holder of a NIR (Foss 6500). Ten scans were performed for each sample carefully re-packing between each scan to minimise the effects of fibre density and inhomogeneity. Data analyses were carried out using the Unscrambler version 7.0 (Camo, Trondheim Norway). Partial least squares (PLS)

modelling procedures were used to develop relationships between the test parameters and the spectra of the fibre samples.

Results and discussion

Physical assessments

After blending through the drawing frames, the two grades of stock fibres with a fineness value of 60.04 dtex for 1(a) and 57.90 for 1(b), exhibited a significant ($P < 0.001$) reduction in fineness (41.79 dtex) at the 2nd drawing stage (Table 1). The improvement in fineness continued through the 4th drawing (34.85 dtex) and roving (34.06 dtex) stages. The drawing stages separated the fibre bundles, liberating the finer fibrils as much of the cementing material, such as pectin and hemicellulose have been selectively removed by the retting, scutching and hackling processes. The mechanical action can also reduce fibre strength, which has been reported as an important factor for spinning high quality yarn.¹⁰ Mean strength of the two stock fibres was 5.12 g/denier for 1(a) and 5.34 g/denier for 1(b) and after blending fibre strength was further reduced to 4.59 g/denier at the roving stage (Table 1). However, maximum strength of the samples increased from 11.07 g/denier for 1(a) and 11.02 g/denier for 1(b) at the beginning to 12.51 g/denier at the roving stage, due to the removal of immature and short fibres. In contrast minimum strength was reduced from 1.25 g/denier for 1(a) and 0.95 g/denier for 1(b) at the beginning of preparation to 1.03 g/denier at the roving stage. The differences in strength measurements were not statistically significant mainly due to high coefficient of variation within the samples.

Chemical analysis

The mean ADF values of the two stock fibres at the start of processing were 83.75% for 1(a) and 83.48% for 1(b) and increased significantly ($P < 0.05$) to 84.55% at the roving stage of the blended fibre (Table 1). This was probably due to the removal of pectin and hemicellulose during mechanical processing and resulting in a proportional increase of ADF fraction. The mean NDF values for the fibre batches were 89.73% for 1(a) and 90.40% for 1(b) and showed a significant ($P < 0.05$) increase to 90.40% after blending at the roving stage. The results for hemicellulose determinations revealed little statistical significance from the hackled fibres [5.62% for 1(a) and 4.67% for 1(b)] to the roving stage (5.53%). CWL of the stock fibre samples was 21.58 % for 1(a) and 22.98% for 1(b) and after blending, this was significantly ($P < 0.001$) reduced to 20.06% at the roving stage (Table 1). The lignin content of the two stock fibres was 1.29% for 1(a) and 1.54% for 1(b) and this was significantly ($P < 0.001$) reduced to 0.68% at the roving stage. Removal of shives (woody material) and short fibres during processing reduced CWL and lignin fractions of the fibre samples. Lipid assessments of the fibres showed reduction from 2.31% for 1(a) and 2.16% for 1(b) at the start of processing to 1.79% at the roving stage.

As expected carbon content (42.20%) remained unchanged during all stages of processing. However concentrations of Ca [0.36% for 1(a) and 0.35% for 1(b)], Zn [10.0 ppm for 1(a) and 10.03 ppm for 1(b)] and Fe (121.44 ppm for 1(a) and 74.83 ppm for 1(b)) present in the two stock fibres showed gradual decline at all processing stages and by the roving stage the mineral fractions were reduced to 0.30, 9.70 and 66.48 ppm for Ca, Zn and Fe (Table 2) respectively. This change is due to the removal of coarse fibres and matrix materials especially pectin.¹² The other fractions (P, K, Mg,

Table 1. Comparison of mean fibre fineness, mean fibre strength, minimum (min) and maximum (max) strength, lignin, lipids, carbon, acid detergent fibre (ADF), neutral detergent fibre (NDF), hemicellulose, caustic weight loss (CWL), calcium, zinc, iron at subsequent stages of processing.

Preparation stage	Fibre fineness (dtex)	Fibre strength (g/denier)	Min strength (g/denier)	Max strength (g/denier)	ADF (%)	NDF (%)	Hemicellulose (%)
Raw fibre 1(a)	60.03	5.12	1.25	11.07	83.75	89.73	5.62
Raw fibre 1(b)	57.90	5.34	0.95	11.02	83.48	88.50	4.67
2 nd Drawing stage	41.79	5.19	1.20	12.35	84.25	90.15	5.66
4 th Drawing stage	34.85	4.94	1.14	13.17	84.34	90.65	6.20
Rove fibre	34.06	4.59	1.03	12.51	84.55	90.40	5.53
Standard error	2.651 ***	0.192 ns	0.106 ns	0.591 *	0.576 *	0.531 *	0.569 ns
Preparation stage	Lignin (%)	Lipids (%)	Carbon (%)	CWL (%)	Calcium (%)	Zinc (ppm)	Iron (ppm)
Raw fibre 1(a)	1.29	2.31	42.11	21.58	0.36	10.08	121.44
Raw fibre 1(b)	1.54	2.16	42.31	22.98	0.35	10.03	74.83
2 nd Drawing stage	1.15	2.24	42.01	22.87	0.30	10.00	62.84
4 th Drawing stage	1.07	1.82	41.97	20.45	0.30	9.46	62.53
Rove fibre	0.68	1.79	42.18	20.06	0.30	9.70	66.48
Standard error	0.123 ***	0.166 ns	0.08 *	0.478 ***	0.02 *	0.80 ns	18.64 ns

* significant at P<0.05, ** significant at P<0.01, significant at P<0.001, ns - not significant.

Mn, Na and Cu) exhibited little or no change in concentration during preparation (data not presented). Significant ($P < 0.05$) change in calcium content was detected between the hackled and 2nd drawing stages. This finding agrees with results from other tests.

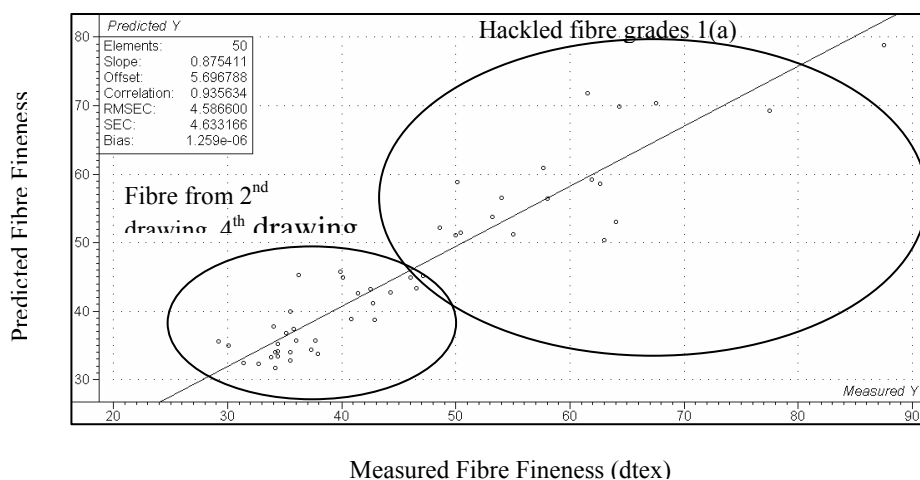


Figure 2. PLS regression analysis of near infrared spectra and fineness of samples (50), segments 700 – 1098, 1400-1420 and 1786-1796 have been excluded ($r^2 = 0.94$, SEC = 4.63).

Development of NIR calibrations

Of all the fibre characteristics, fineness has been reported to be one of the most important parameter for assessing quality.^{5,13} The relationship between fibre fineness and NIR has a r^2 of 0.94 for calibration (Figure 2, and Table 2). The regression graph showed differences in fineness, as a result of the mechanical action during drawing and drafting stages. The strength of this relationship infers that NIR could be used to detect deficiencies in processing caused by faulty machine settings, or alternatively to improve such settings, bringing about an overall increase in fibre quality. The score plot in Figure 3 shows clearly the effects of processing on fibre bundle diameter. Successive processing lead to a break down of the fibre strands, allowing fibrils to slip past one another and therefore produced a more uniform sliver. However in this instance the asymmetrical distribution of the samples can be viewed more clearly in terms of the degree of processing. The score plot showed that the sum of the explained variances for the two components was over 90% and therefore the correlation presented in Figure 2 has a high degree of confidence. A clear relationship between strength measurements of the fibre and spectra was not detected possibly due to high co-efficient of variation of individual fibre strength measurements.

PLS regression of chemical parameters with the NIR spectra produced a wide range of calibration values (Table 2). Despite the crude nature of acid and neutral detergent methods, both showed some degree of correlation with spectra (Table 2). Hemicellulose content exhibited little correlation with NIR and this may partially be due to the techniques used. The lipid content of fibre ranged between 1–2% and did not show a relationship with spectra. During processing, lignin and hemicellulose fractions are mechanically stripped from the matrix to allow separation of the fibre bundles, and therefore the percentage of these fractions was reduced. Consequently the cellulose content increased proportionally and it is difficult to ascertain the effects of processing on these

fibre components. The reported r^2 value of 0.74 for calibration could be improved with additional sample sets (Table 2).

Previous studies have shown that lower CWL value was associated with better quality flax fibre, suggesting that the matrix polysaccharides fractions linked to the cellulose fibrils were removed during retting.^{1,9} PLS regression analyses of CWL and NIR had a calibration r^2 of 0.73 (Table 2), although better calibrations have been developed from other sample sets (unpublished data). The validation r^2 for this parameter was poor at 0.49. Examination of the validation plot showed high degree of variation in the hackled fibres, possibly due to the incomplete removal of sprit (woody material) during retting, scutching and hackling operations, with these effects compounded by the small sample size. Carbon plays a structural role in cellulose and cellulose-related components of cell wall in flax, but a role for iron is not clear although phytoferritin complex¹⁴ in fibre cells may be detected by NIR. Mineral fractions are known to absorb NIR radiation poorly and the observed relationship is possibly due to organic fractions associated with the minerals. Calibrations for carbon and iron in processed fibre yielded r^2 values of 0.74 and 0.70 (Table 2).

Previous research has shown that NIR can predict important quality aspects such as fineness of flax fibre.⁵ This study has demonstrated that NIR could detect changes in fibre fineness and composition during processing. However, the cross validation r^2 values of the PLS regression equations need to be improved to maximise accuracy of NIR predictions and increase robustness. Further work based on extending the size and range of the sample set would help in this matter.

Acknowledgement

We wish to thank Fergusons Irish Linen, Banbridge for providing financial support and Herdmans, Sion Mills, for supplying samples from their production facility.

References

1. A.J. Turner, *Quality in Flax*. LIRA, Belfast, UK (1954).
2. R.R. Mukherjee and T. Radhakrishnan, *Text. Prog.* **4**, 1 (1972).
3. H.S.S. Sharma, in *The 4th workshop of the FAO network on flax*, Ed by J. Boyazoglu, R. Kozlowski, H.S.S. Sharma, L. Rosenburg, C. Morvan and C. Sultana. Rouen, France, p. 397 (1996).
4. Anon, *Linen Research Institute Memoirs* No. 3 (1921).
5. G.J. Faughey and H.S.S. Sharma, *J. Near Infrared Spectrosc.* **8**, 61 (2000).
6. Anon, *Standardised and Recommended Methods of Analyses*, SAC, London (1973).
7. P.J. Van Soest and R. Wine, *J. Agri. Chem.* **50**, 50 (1967).
8. K.R. Christian, *Field Station Rec.* CSIRO, **10**, 29 (1971).
9. H.S.S. Sharma and C.J. Gilmore, *Text. Inst.* **80**, 285 (1989).
10. Anon, Monthly notes of the LIRA. *Linen Res.* **3**, 69 (1942).
11. G.T. Grant, E.R. Morris, D.A. Rees, P. Smith and D. Thomas, *FEMS Microbiol. Lett.* **32**, 95 (1973).
12. G.J. Faughey, H.S.S. Sharma and D. McCall, *J. Appl. Poly. Sci.* **75**, 508 (1999).
13. H.F. Beinfait and F. van der Mark, in *Metals and micronutrients: uptake and utilization by plants*, Ed by D.A. Robb and W.S. Pierpoint. Academic Press, New York, USA (1983).