

A New Method of Evaluating the Fineness and Residual Gum Content of Hemp Fibres.

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Abstract

The demand for eco-friendly apparel and technical textiles has led to a resurgence of interests in bast fibres such as hemp. The lack of fast and objective evaluation of the quality attributes of bast fibres has been a major barrier to the advancement of the bast fibre industry. One of the most important quality attributes of a fibre is its fineness. For bast fibres, the fibre fineness measurement can also reflect the degree of fibre separation during retting or degumming. The traditional method of evaluating the fineness and residual gum content of bast fibres is a very tedious process. In this paper, degummed hemp fibres have been measured for fineness on an Optical Fibre Diameter Analyser (OFDA), and the results have been co-related with the residual gum content in the fibre samples. Since hemp fibres do not have a circular cross section, it is the width of the fibre that gets measured by the OFDA instrument, and this width has been used as an indication of the fibre fineness in this paper. The findings from this study suggest that the optical method can provide a fast and objective way of evaluating the fineness of hemp fibres, and that there is a good correlation between the fibre 'width' measurement and the residual gum content.

Keywords: Hemp; Residual gum content; Optical Fibre Diameter Analyser (OFDA); Fibre fineness.

1. Introduction

There has been a renewed interest in bast fibres in the consumer market. The impetus for this renewed interest arises mainly from the ecological and environmental concerns at both government and consumer levels. Another factor is the desirable properties of bast fibres for

both apparel and industrial applications. Figure 1 compares a range of natural fibres with glass fibre in terms of their mechanical properties (Kessler and Kohler, 1996). The excellent mechanical properties of bast fibres such as hemp and the increasing requirements for biodegradability have led to development of hemp plastics from nonwoven hemp mats, and these hemp plastics have been used by some car manufacturers in headliners and door trims etc.

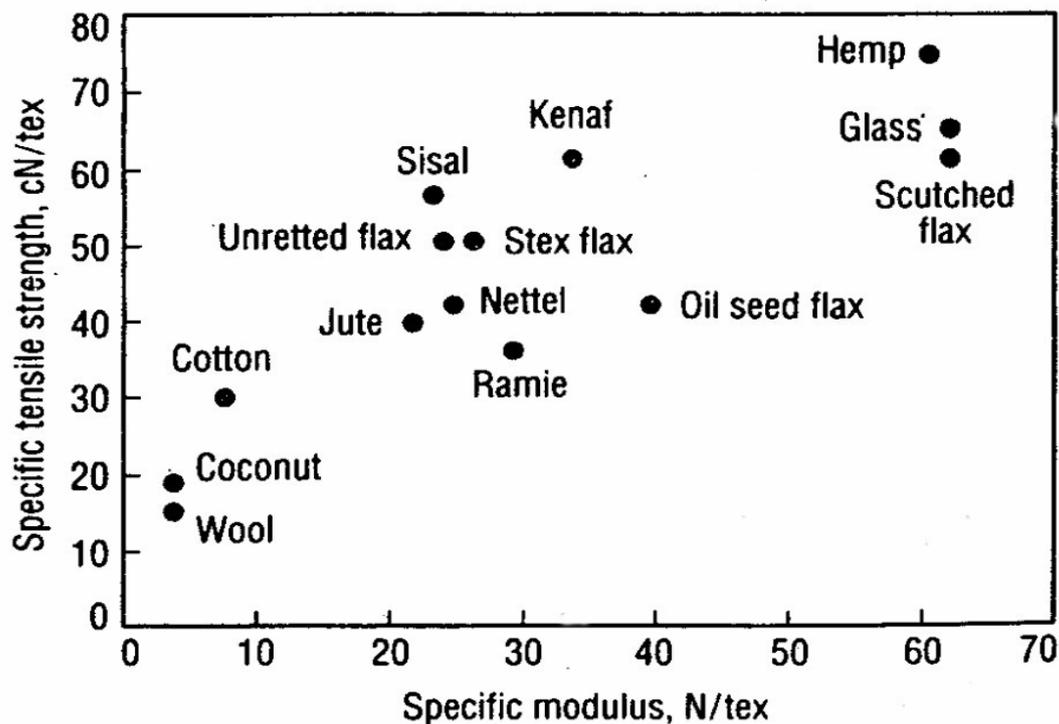


Figure 1: Mechanical properties of natural fibres and glass fibre (Kessler and Kohler, 1996).

Unlike other cellulose fibres such as cotton, hemp fibres must be extracted from the skin or bark of the hemp plants. Beside cellulose the hemp fibre consists of hemicellulose, lignin, pectin and other mixtures that bind fibres together. These impurities must be removed during retting or degumming in order to extract the fibres. Figure 2 gives a simplified view of various stages of hemp fibres and a detailed SEM picture of the degummed hemp fibres. It is worth noting from the SEM photo of the fibre bundle that after degumming, many fibres are still 'glued' together by the residual gum. Improvement in hemp degumming will reduce the residual gum content and hence improve the degree of fibre separation to get very fine hemp fibres.



(a) Dry hemp skin (Chinese variety)

(b) After retting/degumming



(c) Hemp fibres

(d) Cross section of a fibre bundle

Figure 2: Hemp fibres at different stages

The hemp fibre in its individualised form is 10 to 51 micron in width and 5 to 55 mm in length. The most effective ways of reducing the fibre into its individual form are chemical retting [1 2 3 4 5 6 7 8 9], steam explosion [6 10 11], ultrasonic retting [6] and biological retting [5 6 8 9 12 13 14 15]. Chemical and biological retting are commonly used for fibre extraction whereas the other two methods are still in the developmental stages. Hurren et al [3] evaluated the efficiency of degumming hemp fibres with different chemical retting recipes used for other bast fibres. Biological retting uses enzymes and bacteria in aqueous solutions to separate the fibre bundles. It is regarded as a more environment-friendly way of degumming bast fibres.

Regardless of which degumming method is used, it is important to establish the degree of fibre separation and to measure the fibre fineness achieved after degumming. The traditional methods for establishing fibre separation are the Fried test [¹⁵], FTIR spectroscopy [^{13 14 16}] and residual gum content [^{6 8 9 17}].

The Fried test has been used in several papers for the analysis of the degree of retting of flax fibre. It looks at the degree of separation of the fibres after retting. A group of slides showing degrees of separation are used and compared to the results obtained during the retting process. This method is very subjective and does not account for systems that show a better degree of separation than the slides.

FTIR spectroscopy has been used mainly for the research analysis of the degree of retting. Functional groups associated with gums, lignins and waxes can be identified and quantitatively analysed. This method requires very accurate and expensive analysis equipment and takes considerable time and experience to undertake. These constraints limit its use to research applications.

The residual gum content test was used by Das Gumpta et al [¹⁷] in 1976. The test uses a series of caustic degumming to dissolve out any of the residual gum left in the fibre. The residual gum is then expressed as a percentage of the original fibre mass. The test presumes that the more gum removed the better the fibre separation. Unfortunately the test takes considerable time to conduct, which limits its use for industrial analysis applications.

This paper reports a new system for analysing hemp fibre separation is by measurement of the mean fibre width of the retted fibre. The optical fibre diameter analyser (OFDA) measures the width of the fibre snippets by the use of a projection microscope and a digital camera. A determined number of snippets are measured during each test and the mean and coefficient of variation (CV) are calculated using the system software. The test is conducted in less than a minute with minimal sample preparation time. These results can then be correlated with the residual gum content of the hemp specimen.

2. Materials

The hemp variety Zolotonshka (Ukraine) was sourced from plant lots grown at Werribee (Victoria, Australia) by Fibrenova in 2000. The plants were decorticated dry using a decorticator specially designed by Fibrenova. The Sodium Hydroxide (NaOH) and Acetic Acid (CH₃COOH) and Sodium Hydroxide (Na₂CO₃) were sourced from Redox Chemicals Pty Ltd of Melbourne. Hydrochloric Acid (HCl) was sourced from Vival Chemicals Pty Ltd. of Melbourne. Albegal FFA was sourced from Ciba Specialty Chemicals Pty Ltd of Basil Switzerland. All of the chemicals used for the processing of the trials were of an industrial grade.

3. Methods

Testing was conducted on Zolotonshka hemp skins that had been chemically retted for 40 minutes at 100°C. Chemical retting was conducted in the presence of Sodium Hydroxide, Sodium carbonate and an anionic wetting agent (Albegal FFA). Variations were made in the amounts of Sodium Hydroxide (0.125-2.0%) and Sodium Carbonate (0.0-2.0%) used to obtain variations in the degree of retting.

Retting was conducted in a Thies Ecobloc LFA package dye kier. The machine was 20 litres in capacity and was packed with 2100 to 2200 grams of decorticated hemp skins. The hemp was loaded into the machine using a perforated loose stock carrier that was 30-cm diameter with a 5-cm diameter perforated pipe up the centre. The flow rate of the machine was controlled so that the difference in pressure between the outside to the inside or vice versa was 0.4 bar. The direction of flow was controlled by timer and was swapped in direction every five minutes.

The machine was controlled using a SEDO PC1000 PLC. The PLC was used for the control of the filling, draining, heating, cooling and hold functions during the batch processing. The use of the PLC enabled the exact replication of each step of the process from trial to trial.

The test specimens consisted of 3g samples. Each of the samples was selected by zoning and random sampling. Nine samples were selected from each of the decorticated un-retted samples and chemically retted fibre samples. The method of analysis of residual gum content was as

per Das Gupta et al [17] with minor modifications. The procedure has been described in full below.

The weighted 3g samples were dried at 105°C so as to remove any moisture from the sample and the dry weight of the sample was recorded. The sample was pre-treated in a bath of 0.3% HCl at 40°C for 30min with a liquor ratio of 1:50. The sample was then rinsed free of acid with water at a liquor ratio of 1:50. Once rinsed the test specimen was treated with 1% NaOH solution at 94-96°C, with a liquor ratio of 1:100 for 60min. The previous step was repeated. The sample was then treated with a 5% sodium hydroxide solution at 94-96°C for 60min at a 1:100 liquor ratio. The final treatment comprised a neutralising step of 1% CH₃COOH at 40°C for 10min at a liquor ratio of 1:50, followed by a thorough washing with hot water. The samples were then dried at 105°C. The fibre samples were then weighed and the weight recorded. The liquor of each of the steps was filtered and the dry filter mass recorded.

The percentage residual gum content was calculated by dividing the weight loss due to the dissolving of gums by the initial dry weight values of the fibre. This following calculation was used for calculating the percentage residual gum content (RGC).

$$\%RGC = \frac{\text{initial weight} - (\text{final weight} + \text{filtrate weight})}{\text{initial weight}} \times 100$$

Five OFDA tests were conducted for each of the batches. Each of the test samples were selected by zoning and random sampling. Each test consisted of the measurement of 4000 fibres. The mean width and CV of width were calculated using the statistical software on the OFDA. An average of the five measurements was tabulated and used for the comparisons.

Figure 3 shows a picture of the OFDA instrument and the slide of fibre snippets under test.



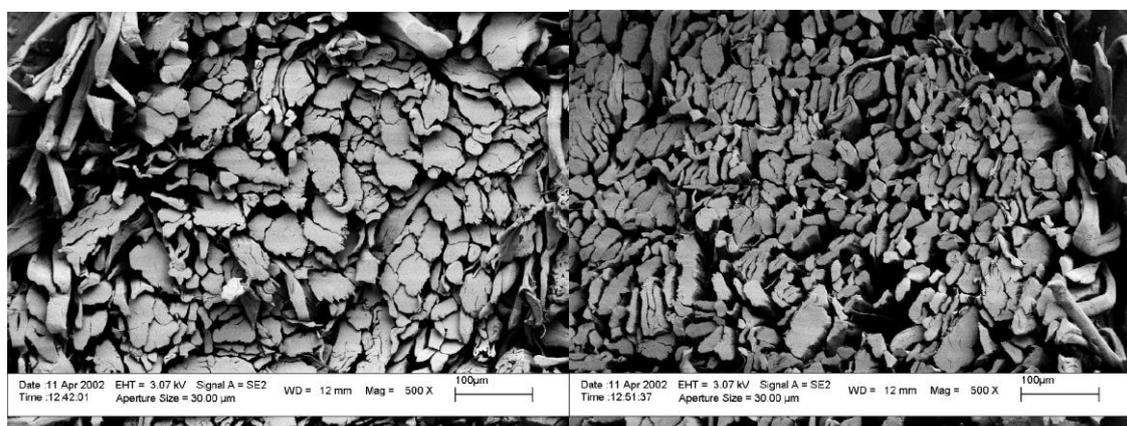
Figure 3: The OFDA instrument (left) and an enlarged view of a slide of fibre snippets (right)

4. Results and Discussion

Figure 4 shows the SEM photo of the fibres that have been chemically retted (trial 4). While the chemically retted sample shows a good degree of fibre separation, there are still bundles of fibres present (Figure 4a). The OFDA measurements for these fibres show that the mean fibre width is 22.16 microns and a CV of fibre width of 72.18 % (Table 3). The CV of the fibre is still very high and this can be explained by the bundles of fibres still present in the sample shown by the SEM.

Figure 4b shows the fibres after the further removal of residual gum. This SEM micrograph shows a far larger degree of fibre separation than the chemically retted one (Figure 4a). The OFDA measurements for these fibres show that the mean fibre width is 18.36 microns and a CV of fibre width of 61.2 %. The mean fibre width has decreased compared to that of the chemically retted fibre. The CV of the fibre has also reduced when compared to the chemically retted fibre sample. Although the CV has reduced it is still considered large for a

natural fibre. This large CV could be explained by the fibre bundles still present in the fibre sample. The fibres that are still bound together could also suggest that the removal of the residual gum is incomplete using the Das Gupta et. al. method [17].



(a) *Chemically retted fibre (trial 4)*

(b) *Fibres after residual gum test (trial 4)*

Figure 4: SEM photos of hemp fibres after retting and residual gum test

Table 2 shows the results of the residual gum content trials on the chemically retted hemp fibres. The results tabled are the average of the six tests that were carried out for each of the trials. The standard deviation of the six values has also been given.

Table 2 – Residual gum content values for the chemically retted fibre.

Trial	Residual Gum Content (%)	Standard Deviation
1	16.40	0.192
2	17.43	0.327
3	15.76	0.430
4	12.72	0.611
5	12.24	0.064
6	11.14	0.553
7	11.71	0.280
8	7.50	0.216
9	6.48	0.581
10	6.96	0.311
11	5.38	0.136
12	6.02	0.326
13	5.00	0.408
14	4.18	0.101
15	3.37	0.131
16	2.64	0.125
17	2.12	0.095

Table 3 shows the results of the OFDA measurements conducted on the decorticated chemically retted fibre. The results tabled are the average of the mean width and coefficient of variation of width of the five tests conducted.

Table 3 – OFDA measurements for the chemically retted fibre.

Trial	Fibre Width (Micron)	CV of Fibre Width (%)
1	22.54	71.02
2	23.73	74.26
3	22.93	72.14
4	22.16	72.18
5	21.22	68.34
6	22.39	74.18
7	22.28	76.13
8	21.66	71.56
9	21.05	71.30
10	20.47	69.00
11	21.29	69.94
12	20.13	64.28
13	20.24	63.30
14	20.68	67.62
15	19.62	63.42
16	19.47	63.52
17	19.68	64.12

Linear regression was then conducted on the results of the fibre width measurements with reference to the residual gum content results. Linear regression was conducted on both the mean fibre width and the coefficient of variation of width. The linear regression equations have been given below in *equations 1 and 2*. *Figure 5* shows the plot of the residual gum content versus the mean fibre width. *Figure 6* shows the plot of the residual gum content versus the coefficient of variation of width. The regression line has been plotted over the top of both charts so as to show the relationships of the regressions to the values.

$$\text{Mean Fibre Width} = 0.233 \times \text{Residual Gum Content} + 19.247$$

Equation 1 – Relationship of mean fibre width to residual gum content.

$$\text{Coefficient of Variation of fibre width} = 0.631 \times \text{Residual Gum Content} + 63.732$$

Equation 2 – Relationship of coefficient of variation of fibre width to residual gum content.

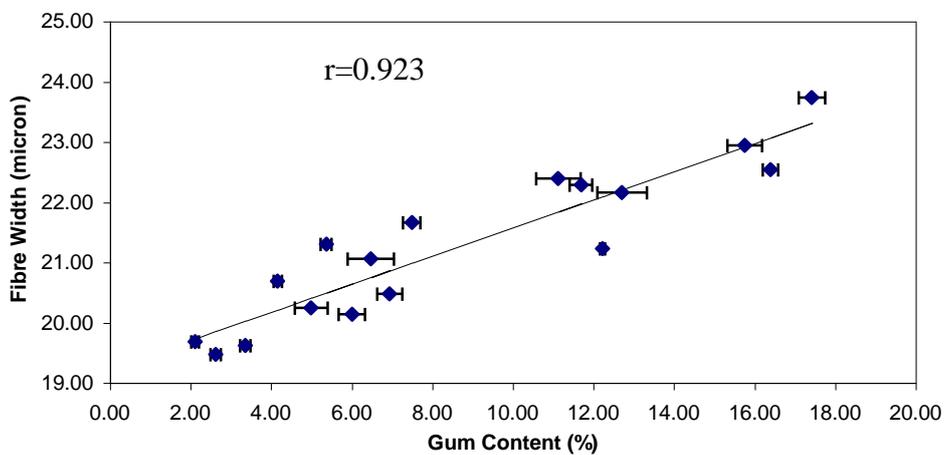


Figure 5: Residual gum content versus the mean fibre width.

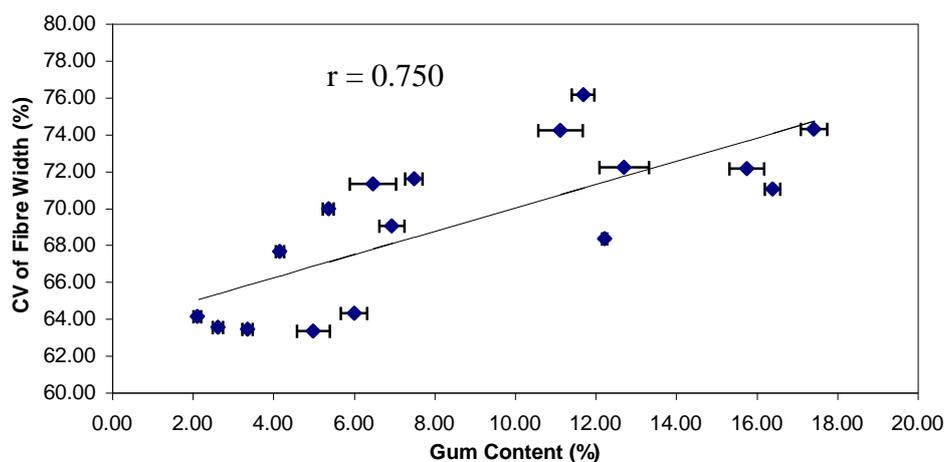


Figure 6: Residual gum content versus the coefficient of variation of fibre width.

The linear regression equations were checked using the correlation coefficient (r). The correlation coefficients given in *figure 4* and *figure 5* show significant correlation between the residual gum content and the mean fibre width ($r = 0.923$) and CV of fibre width ($r = 0.750$).

5. Conclusion

This paper examined the fineness (width) of retted hemp fibres with an Optical Fibre Diameter Analyser (OFDA). The correlation between the residual gum content and the width of chemically retted hemp fibres was investigated in this study. The results show that there is an approximately linear correlation between the residual gum content and fibre width as measured on the Optical Fibre Diameter Analyser (OFDA). This was also the case with the coefficient of variation of fibre width. The significance of these findings is that the tedious evaluation of the fineness and residual gum content of retted bast fibres may be replaced by the much faster measurement of fibre width using an Optical Fibre Diameter Analyser. The SEM results indicate that the residual gum content test procedures do not completely remove the residual gum and some un-separated fibre bundles still exist in the specimens after the test.

6. Acknowledgments

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