

# EVALUATION OF BAST FIBRE RETTING SYSTEMS ON HEMP

**CHRISTOPHER J. HURREN, XUNGAI WANG**

*School of Engineering and Technology, Deakin University, Victoria, Australia 3217*

**HAMISH. G. S. DENNIS, ADRIAN FRANCIS K CLARKE**

*Fibrenova Limited, Victoria, Australia 3136*

## **ABSTRACT**

In this paper the effect of eight different retting methods on decorticated hemp skins were analyzed. The methods were taken from six publications that looked at the retting of different bast fibres such as ramie, flax and kenaf. The fibres were batch retted in a package dye-vat at 100°C under 2 bar of pressure. The rinsed fibres were opened twice using a Shirley cotton trash separator. The opened fibre was assessed for fibre width, fibre width variation, colour and handle. It was found that the fibre that had undergone chemical retting with 7% Sodium Hydroxide (NaOH) and 0.5% Sodium Sulphite (Na<sub>2</sub>SO<sub>3</sub>) had the best diameter (18.39 micron) and the best coefficient of variation of diameter (57.32%). The fibre that was processed with 7% Sodium Hydroxide (NaOH) and 0.5% Sodium Sulphite (Na<sub>2</sub>SO<sub>3</sub>) had the whitest colour (YI D1925 = 31.61). The method treated with 1% Sodium Hydroxide (NaOH) and 1% Sodium Sulphite (Na<sub>2</sub>SO<sub>3</sub>) had the softest handle and the method treated with 1% Sodium Hydroxide (NaOH) and pre-rinsed with 0.3% Hydrochloric Acid (HCl) showed the best conversion of decorticated skins into spin able fibre (33.9% of dry green skins). It was decided that method treated with 1% Sodium Hydroxide (NaOH) and pre-rinsed with 0.3% Hydrochloric Acid (HCl) had the best fineness of fibre micron, conversion to spin able fibre (33.9% of dry green skins), whitest colour (YI D1925 = 36.13) and softest handle for the cost of chemicals involved.

## **INTRODUCTION**

Bast fibres such as Kenaf, Flax, Hemp, Ramie, Jute and Sisal have been used in the manufacture of many varied textile products. Bast fibre needs to be extracted from the plant before it may be used. The first step in the extraction process for Hemp, Kenaf and Ramie is decortication. This is the removal of the bark from the centre woody part of the plant (core part).

The principle of the mechanical decorticator is to subject the stem to a succession of blows to break up the woody core [8]. Dry decortication is quicker and not confined to the harvesting season. However, decortication of fresh stems produces a finer fibre of better quality [10, 11, 13].

The next part of fibre extraction is called retting and is carried out on only the bark portion of the plant. The lignin's, waxes, hemicellulose and pentosans present in the bark need to be broken down to release the fibres.

Traditionally, retting was done in water canals in which the action of anaerobic microorganisms hydrolyzed matrix carbohydrates and freed cellulose fibres. Biological retting can be more closely controlled in tanks where the water temperature, flow rate, and retting microorganisms can be adjusted and monitored [19, 20], but the stench and polluted effluents from fermentation have prevented use of this retting process in Western countries [12].

In most Western countries the method of choice is dew retting, where the bast fibre is spread out in fields for filamentous fungi and to some extent also bacteria and yeast, to degrade pectin and other matrix polysaccharides connecting the fibre cells. However there are several problems associated with this method: fibre quality is often poor or inconsistent, it is climate dependent, there is a risk of over retting, i.e. the microorganisms start to degrade the cellulose and thereby make the fibres weaker, and it is relatively labor intensive. Research has therefore focused on finding alternative methods for retting bast fibres. Attempts have been made to develop both enzymatic [1, 2, 3, 6, 11, 16, 17, 18,] and chemical/physical [3, 5, 7, 9, 11, 12, 16, 17, 18] methods, and combined chemical and enzymatic retting [3, 15, 17] has also been suggested [7].

Chemical/physical retting consist of treatments such as boiling in Sodium Hydroxide [12], boiling in sodium hydroxide in the presence of sodium sulphite [9, 16], boiling in sodium hydroxide in the presence of Sodium Bisulphite [17], boiling in Sodium hydroxide in the presence of sodium chloride, EDTA and Sodium Sulphite [9], boiling in Sodium hydroxide in the presence of EDTA [9], boiling in Sodium hydroxide after soaking in Hydrochloric Acid [5] and boiling in Oxalic Acid at high pH [7] and steam explosion [14]. In this project we looked into the various chemical methods that have been mentioned above to assess their effect on hemp. The methods that are to be analyzed have come from three different bast fibre types. Three of the methods have been previously done on Kenaf [12, 16, 17], four of the methods on Ramie [5, 9] and one on Flax [7].

Hemp (*Canabhis sativa* L) has been selected for this report as it has a high yield for a short growing cycle. The average growing cycle for a hemp fibre crop is 70 to 90 days. Hemp also has the advantage that its short growing time allows it to be share cropped with other crops in the one-year. The hemp plant yields high in the Australian climate and is quite suited to early summer growth. Hemp does not require the fertilizer or pesticide loading used on cotton [22].

Optical Fibre Diameter Analysis (OFDA) was selected as the main choice for evaluation of fibre fineness. The most effective retting systems will separate the fibres into individual fine fibres. OFDA has the ability to analyze the diameter spread of the fibre as well as the coefficient of variation (CV) of the diameter. Diameter and CV of diameter are very important for the manufacture of yarn. For a spun yarn to be perfectly even we need two conditions: (1) The constituent fibres are uniform in diameter. (2) The yarn has the same number of fibres in all cross sections along its length [21]. The number of fibres in the yarn cross section has a decisive effect on yarn evenness [21]. The larger the number of fibres in the cross section the more even the yarn produced from those fibres is.

Colour has always been an important part of raw fibres destined for textile production. The D1925 yellowness index system was developed by the American Society for Testing Materials (ASTM) to give a better system to quantify the yellowness of wool fibres. It is derived from the CIE Tri stimulus values and gives increased emphasis to the colour bands that most influence yellowness [4].

Softness of handle is one of the most important factors in a textile fibre. Handle assessment was compared with cotton as it has a handle that is already accepted and is a cellulose-based fibre like hemp. Handle assessment is subjective and should be carried out by a panel of assessors so that an averaged result can be measured.

## ***RESEARCH PLAN AND METHODOLOGY***

### **Materials and Chemicals**

Fibre hemp variety Zolotonshka (Ukraine) was sourced from plant lots grown at Werribee by Fibrenova in 2000. The plants were decorticated dry using a decorticator specially designed by Fibrenova. The Sodium Hydroxide (NaOH), Sodium Sulphite (Na<sub>2</sub>SO<sub>3</sub>), Oxalic Acid (HOCCOOH), Sodium Acetate (NaCH<sub>2</sub>COOH) and Acetic Acid (CH<sub>3</sub>COOH) were sourced from Redox Chemicals Pty Ltd of Melbourne. The Sodium Chloride (NaCl), Sodium Bi Sulphite (NaHSO<sub>3</sub>), Ethylene Diamine Tetra Acetic Acid

(EDTA) and Hydro Chloric Acid (HCl) were sourced from Vival Chemicals Pty Ltd. of Melbourne. The wetting agent used was Cibaflo CIR and was sourced from Ciba Specialty Chemicals of Switzerland. All of the chemicals used for the processing of the trials were of an industrial grade.

### Batch Chemical Retting

All of the chemical retting was carried out in a Thies Ecobloc LFA package dye kier. The machine is 20 litres in capacity and was packed with 1900 to 1950 grams of decorticated hemp skins. The hemp was loaded into the machine using a perforated loose stock carrier that is 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 is 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. This 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.

Eight different methods were used for the evaluation of retting effectiveness. Each of the methods was taken from a paper published on the retting of bast fibres. The retting steps were carried out at 100°C for 60 minutes. These conditions were the same for all of the processes. Some of the processes had a pretreatment and some had different post rinsing systems as shown in Table 1.

Table 1 – Batch retting methods.

METHOD NUMBER	SOURCE PAPER	PRETREATMENT	RETTING	RINSING	AFTER TREATMENT
1	Das Gumptra et al [5]	0.3% HCl 30 min @ 40°C	1.0% NaOH 60 min @ 100°C	-	1.0% CH <sub>3</sub> COOH 10 min @ 60°C
2	Ramaswamy et al [16]	0.85% Cibaflo CIR 60 min @ 60°C	7.0% NaOH 0.5% Na <sub>2</sub> SO <sub>3</sub> 60 min @ 100°C	10 min @ 60°C	2.0% CH <sub>3</sub> COOH 10 min @ 60°C
3	Ramaswamy et al [17]	0.85% Cibaflo CIR 60 min @ 60°C	7.0% NaOH 0.5% NaHSO <sub>3</sub> 60 min @ 100°C	10 min @ 60°C	0.2% CH <sub>3</sub> COOH 10 min @ 60°C
4	Morrison III et al [12]	0.85% Cibaflo CIR 60 min @ 60°C	7.0% NaOH 60 min @ 100°C	10 min @ 60°C	0.2% CH <sub>3</sub> COOH 10 min @ 60°C
5	Kundu et al [9]	-	1.0% NaOH 1.5% NaCl 0.05% EDTA 0.5% Na <sub>2</sub> SO <sub>3</sub> 60 min @ 100°C	10 min @ 60°C	2.0% CH <sub>3</sub> COOH 10 min @ 60°C
6	Kundu et al [9]	-	1.0% NaOH 0.05% EDTA 60 min @ 100°C	10 min @ 60°C	2.0% CH <sub>3</sub> COOH 10 min @ 60°C

7	Kundu et al [9]	-	1.0% NaOH 1.0% Na <sub>2</sub> SO <sub>3</sub> 60 min @ 100°C	10 min @ 60°C	2.0% CH <sub>3</sub> COOH 10 min @ 60°C
8	Henriksson et al [7]	-	0.63% HOCCOOH 0.22% NaOH 0.5% Cibaflo CIR 60 min @ 100°C	-	1.0% NaCH <sub>2</sub> COOH 10 min @ 60°C

### Rinsing and Drying

After the batch retting process the unheld lignin, wax and colour were removed by two dunks in hot water at 70°C. The fibre was submerged under the water and dunked 10 times by hand before it was removed and hydro extracted to remove the excess water. After the two dunks the fibre was dried using a fan forced oven at 105°C for 2 hours.

### Opening

The fibre was opened twice after drying using a Platt Shirley trash analyzer. The fibre that was carried over into the clean fibre box was set aside and kept. The fibre that was dropped out into the trash box of the Shirley trash separator was processed a second time. The fibre carried over into the clean fibre box on the second run was mixed with the first lot of fibre collected from the same box. The fibre removed from the trash box was set aside. The testing was then carried out on the fibre removed from the clean fibre box from both runs. The opened fibre was conditioned in a standard atmosphere laboratory for 2 days before any testing was carried out.

### Optical Fibre Width Analysis

Width testing and variation of Width testing were carried out on an Optical Fibre Diameter Analysis Machine (OFDA100). Five tests were conducted for each of the methods. The average width (micron) and the coefficient of variation (%CV) was recorded for each test and averaged over the 5 tests.

### Colour Testing

The opened fibre was measured for colour difference using a Datacolor Spectraflash SF600. The CIE tri stimulus values X, Y, Z and D1925 Yellowness Index for each of the samples was recorded. The measurement was done using instrument averaging where the sample is measured 10 times and then averaged by the datacolor software. Each measurement was carried out after the sample had been moved and rotated through 90 degrees. A 30mm diameter aperture was used and the light source was D65.

### Handle Assessment

The opened fibre was assessed for softness of handle by a panel of 10 students. A sample of middling cotton was inserted as a known handle comparison. The samples were dried for 2 hours at 105°C in a fan forced oven then conditioned for 24 hours before assessment. Each sample was graded in two ways

Method 1 (position) was assessed using a ranking system of 1 to 9 where 1 was the softest and 9 the harshest. Each sample was handled and then put in order of softest to harshest. The appropriate number was assigned to the sample.

Method 2 (rank) was assessed using a ranking system of 1 to 5 where 1 was the softest and 5 the harshest. Each sample was assessed and the softest was assigned 1 and the harshest sample was assigned 5. All of the other samples were assigned a value with reference to these two samples.

## ***RESULTS AND DISCUSSION***

### **Fibre Conversion Rates**

The initial retting loss, opening loss, total loss and skin to fibre conversion rate for each of the trials are given in Table 2. There was a large amount of hurd present in the skins that was due to ineffective decortication of the plant stem. This must be taken into account when interpreting the loss and conversion data given in Table 2.

Although method 8 had the lowest retting loss it suffered from quite drastic losses during the opening stage. This brought it to a level of loss similar to all of the other processes. Method 1 underwent the second lowest retting and second lowest opening loss, which gave it the best skins to fibre conversion rate of all of the method. Method 4 underwent the lowest losses during opening. This, when combined with the retting loss, gave the second best skins to fibre conversion rate of all of the trials.

*Table 2 - Losses incurred during processing.*

METHOD NO	RETTING LOSS (%)	OPENING LOSS (%)	TOTAL LOSS (%)	SKINS CONV (%)
1	44.4	39.1	66.1	33.9
2	52.5	39.8	71.4	28.6
3	51.9	42.4	72.3	27.7
4	53.8	33.2	69.1	30.9
5	46.1	44.7	70.2	29.8
6	45.8	45.0	70.2	29.8
7	46.8	47.2	71.9	28.1
8	32.2	58.6	71.9	28.1

### **Colour**

The CIE tri-stimulus values (X, Y, Z) and the D1925 Yellowness Index values for the trials are given in Table 3. Figure 1 gives the D1925 Yellowness Index data compared to a middling cotton sample (9). Method 2 has the best value for the D1925 Yellowness index (31.61). Visually this sample showed the best purity. Method 8 had a D1925 Yellowness Index value of 34.77. The purity of this sample did not concur with the measured value. The purity of the sample was by far the worst of all of the methods. The colour of the sample was a pale green when compared to the slight red/yellow tinge of all of the other samples. The cotton sample was far lighter than the hemp trials and this has been indicated by the far lower D1925 Yellowness Index given in Table 3 value 9.

Table 3 – CIE Tri-stimulus values and D1925 Yellowness Index values

METHOD NO	X	Y	Z	YI D1925
1	46.53	48.69	39.59	36.13
2	51.90	54.40	46.45	31.61
3	52.43	54.62	43.59	38.27
4	52.50	54.57	43.25	39.13
5	49.23	51.69	41.69	36.42
6	49.66	51.85	41.92	36.89
7	52.90	55.51	45.52	35.06
8	48.74	51.05	42.11	34.77
COTTON	76.20	80.04	75.36	22.06

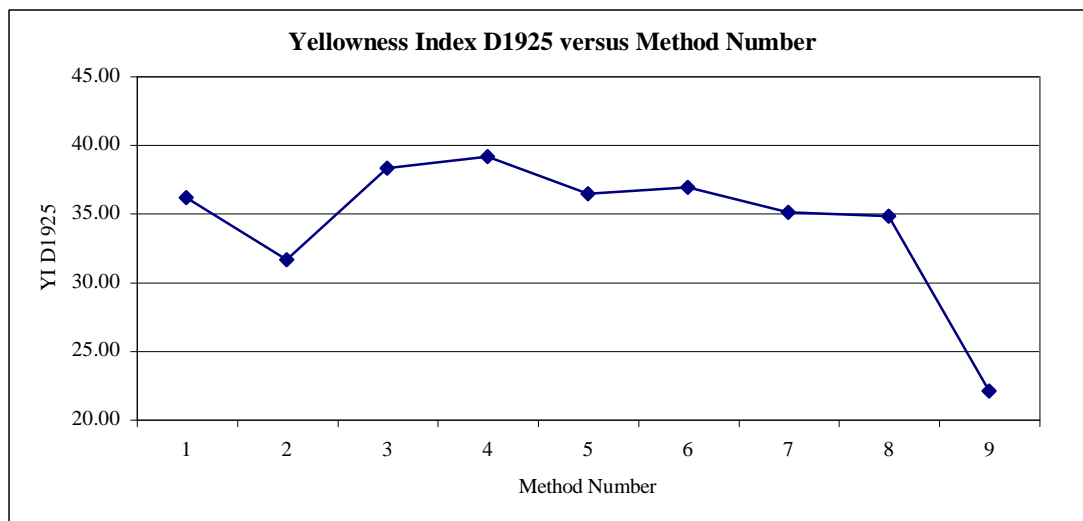


Figure 1 – D1925 Yellowness Index values

### Fibre Width and Coefficient of Variation of Fibre Width

The fibre width and the CV of fibre width are given in Table 4. Each value is the average of 20,000 fibre measurements. Figure 2 gives the width and CV of width of the fibre plotted against their trial number. Method 2 demonstrates the best values for both its width and its CV of width. These lower values for both width and CV are indicative for all of the methods (methods 2, 3 & 4) that involved retting with 7% NaOH. All but one of the other trials involved the use of 1% NaOH in the retting bath. All of these trials had similar width and CV of width values. Method 8 had the worst of the values recorded for width and CV of width. NaOH was used in the retting process of method 8 however the use of oxalic acid caused an acid base reaction which rendered the NaOH ineffective.

Table 4 – Fibre width and coefficient of variation of fibre width.

METHOD NO	FIBRE WIDTH (Micron)	%CV OF FIBRE WIDTH (%)
1	19.75	63.00
2	18.39	57.32
3	19.17	62.92
4	19.15	63.64
5	20.29	67.46
6	20.31	68.16
7	21.63	69.52
8	24.33	73.62

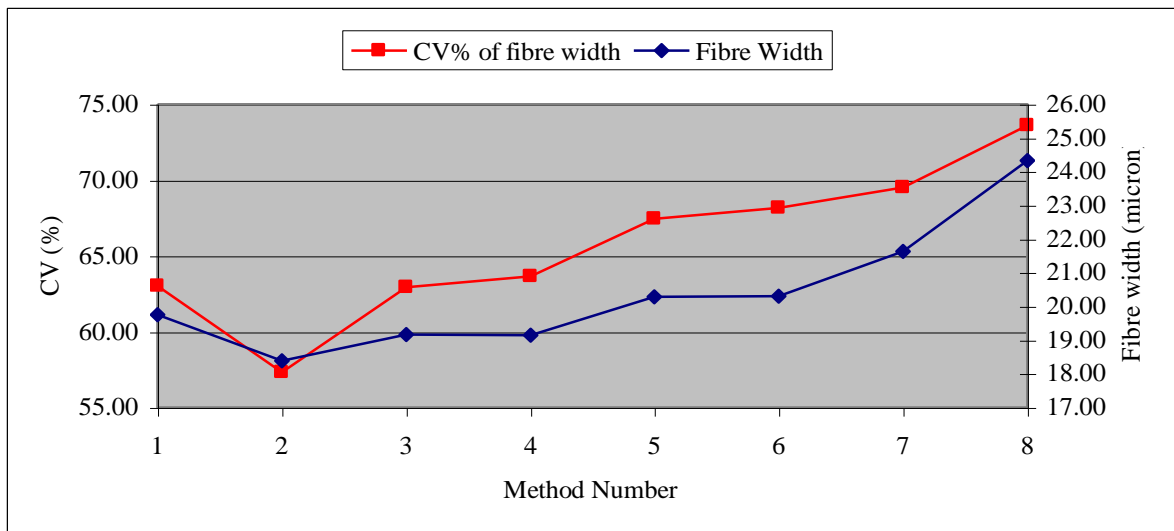


Figure 2 – Fibre width and coefficient of variation of fibre width.

### Handle Assessment

The handle assessment results are given in Table 5. The position column of the table shows that the handle achieved by the best sample of hemp was still harsher than that of cotton. The method that had the best handle standard was method 7. The handle of method 7 was very similar to that of methods 2, 3 and 4. This can be seen from the rank column of Table 5. The method with the worst handle was method 8. This poor handle was indicative of all the other results achieved for method 8.

Table 5 – Position and Rank values for handle assessment.

METHOD NO	POSITION	RANK
1	7	3/4
2	3	2/3
3	4	2/3
4	5	2/3
5	8	4
6	6	3
7	2	2/3
8	9	5
COTTON	1	1

## **CONCLUSION**

The processing trials have shown that the retting methods with 7% NaOH gave the best results for fibre width, coefficient of variation of fibre width and handle. The samples that were retted with 7% NaOH gave mixed colour results and a low ratio of skins to fibre conversion. The main problems with the methods that involved 7% NaOH was the cost of the chemicals that were needed for the retting process and the environmental effects that the effluent could cause. These negative factors caused methods 2, 3 and 4 to be ruled out as the best retting methods.

The fibre that was retted using method 1 gave good conversion of skins to fibre, good fibre width and coefficient of variation of fibre width, average handle and colour. As only 1% of NaOH was used the cost and the environmental problems associated with methods 2, 3 and 4 are not as prevalent with this process. This method was selected as it gave good overall results, a cost effective price and at a low effluent loading.

Method 8 showed the poorest results of all the trials. The chemicals used in the trial reacted together in an acid base reaction causing a reduction in the efficiency of the process.

Fibre width has been used as a simple measure of the effectiveness of the retting process however yarn processing is the only way that the retting process could be properly evaluated. Further work could be done in the analysis of the spin-ability of the fibre produced in each of the trials along with the properties of the yarn produced. This would allow for correlation of the effectiveness of fibre width measurement as a means of judging retting efficiency.

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