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Chemical Modification and Processing of *Phormium tenax* Fibres

Part I: A Preliminary Report

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CHEMICAL MODIFICATION AND PROCESSING OF *PHORMIUM TENAX* FIBRES

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ABSTRACT

Phormium tenax *fibres have so far only been used for the manufacturing of grain bags and affiliated products. When these fibres are subjected to batchwise sodium hydroxide and sodium hypochlorite treatments in order to soften and split the fibres into finer fibres, an exhaustion of 90 per cent of both chemicals can be achieved. The resultant fibres are whiter and softer and although the wet tenacity is very poor the dry fibres do not show any significant loss in strength. When the treated* Phormium tenax *fibres are blended with acrylic fibres in a 70/30 ratio, yarns as fine as 225 tex can be spun from the blend and the yarn can be woven into attractive fabrics which become softer after repeated laundering.*

A preliminary *costing analysis shows that the fibre is still comparatively cheap after the chemical treatment.*

INTRODUCTION

Phormium tenax is a plant indigenous to New Zealand. It is also commercially grown in other countries such as St. Helena, the Azores, South Africa (Kwazulu), Transkei and some South American countries. The fibre which is obtained from the leaf is referred to as Phormium fibre or Phormium. It is commonly used for the manufacturing of grain bags and other coarse bagging materials.

A new aspect of the research programme at SAWTRI on Phormium fibre is to explore *new end-uses* for the fibre. The fibre, however, is considered as too hard and brittle for textile applications other than coarse bagging, and, therefore, it became necessary to examine the possibility of modifying the fibre. Forty years ago Brandt[1] indicated that the hope had often previously been expressed that Phormium fibre could be split up into finer fibres, softened and delignified to produce a material which would compete with the finer vegetable fibres such as linen. He also referred to a paper which as long ago as 1873 discussed aspects such as the softening of the fibre, chemical retting, etc. Brandt strongly recommended that research should be directed towards the softening of the fibre. As far as can be ascertained there is, to date, no commercial process in use for the softening and bleaching of Phormium fibres.

Jute, which is a related bast fibre, for many years also had only limited textile applications such as in packaging and in carpet backings. The Indian Jute Industries' Research Organisation realised that to maintain adequate markets for the fibre, there was a need to diversify its usage. They investigated means of softening and bleaching the fibre, and its possible use for decorative or furnishing fabrics and even apparel[2,3]. Their success in this field was marked by the patenting of the Huetex process[2,4] in 1968. It is claimed that this process gives a light fastness rating of 4 compared with a rating of 1 obtained with a conventional bleach using hydrogen peroxide. Similar softening and bleaching processes for other vegetable fibres such as sisal, hemp and ramie were also attempted[5-7].

Jute fibres have been blended with rayon, wool and other fibres[2,7-10]. The fibres are blended to impart softness, increase weaving efficiency, reduce imperfections and to increase dimensional stability of fabrics. In this paper some preliminary investigations on the softening, splitting and bleaching of Phormium as well as its blending with acrylic fibre for ultimate use in other textile applications, are described.

EXPERIMENTAL

Materials

The Phormium fibres were obtained after decortication with the Elgin decorticator[11] and contained from 5 to 18 *per cent* loose particles and dust as determined by Godawa's method[12].

Chemicals

Hypochlorite solution of technical grade was used throughout. All other chemicals were of laboratory grade during the initial, small-scale laboratory trials but of technical grade for subsequent, larger scale trials.

Apparatus

A Libbrecht loose stock Dyeing Apparatus was used for the large scale experiments.

Chemical treatment of the fibres

A liquor-to-goods ratio of 20:1 was used throughout. The fibres were rinsed in boiling water, followed by treatments using combinations of the following reagents: sodium hydroxide, sodium hypochlorite, sodium chlorite[13], sodium bisulphite, sodium sulfide, sodium sulphite and sodium bisulphite. The concentrations varied from 0,2 to 20 *per cent* (m/v). Temperatures ranged from room temperature to boiling point and treatment times from 10 to 180 minutes. In order to evaluate the effect of certain chemical treatments on the light fastness of Phormium, grain bags were treated with sodium hydroxide and sodium hypochlorite, followed by a treatment with:

(a) 1 *per cent* sodium dithionite (room temperature, 15 minutes) or,
(b) 1 *per cent* sodium bisulphite (room temperature, 15 minutes) or,
(c) 1 volume hydrogen peroxide, 1 *per cent* sodium silicate and 0,25 *per cent* trisodium phosphate (70 — 75° C, 1 hour).

The fibres (or grain bags) were rinsed at least once in water after each chemical treatment.

Mechanical processing

Preliminary attempts to spin the softened Phormium on the available equipment at SAWTRI were carried out as follows:
The fibres were cut into bundles of length approximately 180 mm, then loosened by hand to avoid matted pieces entering the card, and at the same time lubricated with ®Bevaloid fibre lubricant 4027 (2 *per cent*, o.m.f.) and water (4 *per cent*, o.m.f.). The lubricated fibres were then fed by hand to a small Turner Atherton card, the rate being judged subjectively, and delivered in loose form. The carded fibres were again lubricated with the same lubricant (2 *per cent*, o.m.f.) and water (2 *per cent*, o.m.f.) and then hand-blended with 3,3 decitex acrylic fibres of staple length 100 mm in the proportion Phormium/acrylic 70/30. The blend was fed to the card by hand and delivered in sliver form. The carded slivers were gilled three times on a Schlumberger GNP gill box, then drawn once through the same machine and finally fed directly to a Dandy rover of a Bradford flyer spinning frame. Yarns having a linear density of 225 tex were spun.

Tests

1. Loose particles and gum content on untreated fibres: Reference 12.
2. Cellulose content: Reference 14 (p.358).
3. Lignin content: Method of Ritter *et al*, reference 14 (p. 373).
4. Amount of gum on fibres: Reference 13.
5. Amount of NaOH consumed (o.m.f.): Titration of the solution before and after treatment of the fibres with standard hydrochloric acid solution.
6. Amount of Cl_2 consumed (o.m.f.): Titration of the solution before and after treatment of the fibres using the sodium thiosulphate — potassium iodide method.

7. Yield.
8. Linear density of fibres: The samples were hand-combed and the length and mass determined.
9. Bundle tenacity (dry and wet): Stelometer No. 154 M (Spinlab) (3,2 mm gauge).
10. Yarn tenacity: Similar to BS 1932 Part I (1965), except that a specimen test length of 250 mm was used.
11. Brightness: A Zeiss Elrepho apparatus was used to determine the reflectance (Y filter) of treated Phormium grain bags.
12. Light fastness: The light fastness of woven Phormium grain bag fabric, treated with various chemicals, was determined in a Xenotest 450 apparatus and assessed against international blue standards.

RESULTS AND DISCUSSIONS

Laboratory experiments to soften and split Phormium

The following preliminary treatments were carried out:

(a) The method of Kulkarni[15] for bleaching jute with sodium chlorite was performed on Phormium. The fibres were white but very harsh.

(b) A fairly soft, and yellow fibre was obtained when the fibre was boiled in a mixture of 0,5 *per cent* (m/v) sodium hydroxide and 0,25 *per cent* (m/v) sodium sulphide solution for two hours.

(c) When the fibres were treated in sodium hypochlorite, 1 *per cent* (m/v) active chlorine, acidified to pH 2,6 and followed by boiling for 15 minutes in sodium hydroxide (1 *per cent*, m/v) solution, a pulp was obtained. This and other experiments indicated that an acid hypochlorite treatment resulted in the fibres being broken up into too short lengths.

(d) The fibres were boiled for 3 hours in a sodium bisulphite solution, subsequently boiled for 10 minutes in a 10 *per cent* (m/v) sodium hydroxide solution and then bleached with a sodium hypochlorite solution (3 *per cent* active chlorine, m/v) for 10 minutes at room temperature and finally dechlorinated with 2 *per cent* (m/v) sodium bisulphite. This gave fairly soft and bleached fibres which had a pale yellow colour.

(e) The Huetex patent for improving the light fastness of jute fibres[4] was also applied to Phormium fibres. Although the fibres were fairly white after applying this method, which involves treatment with sodium hypochlorite followed by sodium hydroxide (or sodium sulphite followed by hydrogen peroxide) solutions, they were harsh.

After further experiments it was concluded that the most promising results were obtained with the following:

(a) Boil in sodium hydroxide solution and rinse.
(b) Bleach in sodium hypochlorite solution and rinse.

The fibres were fairly soft and partially bleached after this treatment. Repeating the treatments on the same sample reduced the fibre yield but produced softer and finer fibres. This treatment on the *leaf* fibre, Phormium, is virtually the same as that described in a patent[7] for the softening and bleaching of *bast* fibres (flax, hemp, ramie and jute), except that the recommended addition of *acid* during the hypochlorite treatment did not seem to be advantageous in the case of the Phormium fibre, since they were weaker, more fragmented and not as well-bleached at lower pH values, although they were found to be somewhat softer.

It was, furthermore, noticed that the tip and the butt fibres responded differently to the chemical treatments. For example, when the tip and butt fibres were treated separately in 2 *per cent* (m/v) sodium hydroxide solution for 45 minutes at the boil, rinsed, treated in a sodium hypochlorite solution (1 *per cent* active chlorine, m/v) and rinsed, it was found that the fibres from the tips were significantly softer than those from the butt end of the leaves.

The results of the linear density determination and the bundle tenacity test of the decorticated and *chemically untreated* fibres are given in Table I. Sample No. 1 was used for all the laboratory experiments and sample No. 2 for the larger lots (more than 1 kg). There was a large difference in linear density between the two samples but this could probably be attributed to factors such as the age of the leaves, decorticator settings, etc. There was also a considerable difference in the linear density between the butt and the tips of the fibres.

Table II shows the effect of some of the treatments on the fibre linear density and breaking strength. In one case the fibre linear density was reduced by more than 50 *per cent*. The dry fibre bundle tenacity was only reduced significantly in the case of those fibres which had been treated with sodium bisulphate. This reduction is probably due to an acidic degradation of the cellulose (the pH of a solution of sodium bisulphate is below 1,5). The wet tenacity (not given) was very poor and wet strength losses of as much as 80 *per cent* were obtained in some cases.

The brightness and light fastness of chemically treated Phormium

The brightness of the untreated Phormium was 32 and increased to 46 after treatment with the sodium hydroxide/sodium hypochlorite process and further treatment with sodium bisulphite. When hydrogen peroxide was substituted for the sodium bisulphite a brightness of 55 was obtained.

The light fastness of bleached jute is normally very poor (1 — 2)[4]. It is, however, possible to produce bleached jute fibres with a relatively high light

TABLE I

THE LINEAR DENSITY AND BUNDLE TENACITY OF UNTREATED PHORMIUM FIBRES

	Sample No. 1		Sample No. 2	
	Butt End	Tip End	Butt End	Tip End
Linear density of fibres (tex)	24	19	36	26
Bundle tenacity (cN/tex)	34	38	25	41

TABLE II

LINEAR DENSITY AND BUNDLE TENACITY OF TREATED FIBRES

Treatment	Range of Fibre Linear Densities (Tex)	Range of Dry Bundle Tenacities (cN/Tex)
No treatment	19—24	34—38
Boiled in water for 1 hour. boiled in NaOH 2% (m/v) for 1 hour and treated with NaOCℓ 1% (m/v) for 10 minutes at room temperature	11—18	38—44
Boiled in water for 1 hour, boiled in NaOH 2% (m/v) for 1 hour and boiled in $NaHSO_4$ 2% (m/v) for 1 hour.	7—11	25—28
Boiled in Na_2SO_3 2% (m/v) for 2 hours and treated with NaOCℓ 1% (m/v) for 10 minutes at room temperature.	14	42

fastness. For example, when jute fibres are treated according to the Huetex process, which includes a further bleaching step, a light fastness rating of 4 — 5 is claimed. Little is known, however, about the light fastness of Phormium and the effect of bleaching treatments on the light fastness. Consequently grain bags made from Phormium fibres were treated and tested for light fastness. A Phormium grain bag, first treated with sodium hydroxide followed by sodium hypochlorite and subsequently treated with sodium bisulphite (or sodium dithionite), had a light fastness of 5 to 6. Another sample bleached with hydrogen peroxide instead of the sodium bisulphite also had a light fastness of 5 to 6. It is obvious, therefore, that the chemical softening process, followed by a bleaching treatment, produced Phormium fibre of an acceptable light fastness.

The chemical composition and yield of fibres

The chemical composition of the fibres before and after treatment with sodium hydroxide and sodium hypochlorite is given in Table III. The cellulose content in Phormium has been reported to be 72,7 *per cent* [1] or 72,0 *per cent* [16]. In this investigation a value of 64,5 *per cent* was found for the untreated fibres and, depending on the conditions of treatment, values of 80,8 to 88,5 *per cent* cellulose were also found. The lignin content of Phormium, which was reported to be 11,1 *per cent* [16] and 12,6 *per cent* [1] decreased in the present study from 14 *per cent* to 6,4 *per cent* as the cellulose content increased. The yield of fibres varied with the different treatments, and yields for a sodium hydroxide/sodium hypochlorite treatment applied once only varied from 60 to 71 *per cent*. The yield after treatment with sodium hydroxide only was found to be about 80 *per cent*. The yield of the fibre was also correlated with the cellulose content, i.e. the higher the cellulose content, the lower the yield of fibres. In the case where the cellulose content was found to be 88,5 *per cent* the fibres were overtreated, causing excessive fibre breakage during carding.

Gums and loose particle content

The gum content of the fibres was determined according to the method recommended by Gupta *et al* [13] for ramie, but very high values (about 30 *per cent*) were obtained. It seems that the method, which involves the use of sodium hydroxide and hydrochloric acid, also extract other constituents in the case of Phormium. The method used by Godawa[12] seemed to give a more realistic value in the case of untreated Phormium.

Consumption of chemicals

The amount of chemical consumed during the reaction was found to be 6

TABLE III

THE EFFECT OF VARIOUS TREATMENTS ON THE CHEMICAL COMPOSITION OF PHORMIUM

Treatment	Chemical Composition (%)					Fibre Yield %	Chemicals Absorbed (%)	
	Cellulose	Lignin	Loose Particles	Gum	Total		NaOH (o.m.f.)	Active $C\ell_2$ (o.m.f.)
Untreated	64,5	14,0	4,5	5,5	88,5	—	—	—
Boiled in water for 60 minutes	71,4	14,5	—	—	85,9	95	—	—
Boiled in 1,4% NaOH for 60 minutes	80,8	12,8	—	—	93,6	79,7	6,9	—
Boiled successively in water for 60 minutes and in 1,9% NaOH for 60 minutes, followed by treatment in 2,1% $C\ell_2$ solution for 20 minutes at room temperature.	84,0	10,3	—	—	94,3	67,0	8,0	7,7
A. Boiled successively in water for 60 minutes and in 2,1% NaOH for 60 minutes, followed by treatment in 2% $C\ell_2$ solution at 27°C for 30 minutes	—	—	—	—	—	—	5,7	8,8
B. Treatment A followed by boiling in 1,6% NaOH for 30 minutes and finally treated with 1,3% $C\ell_2$ solution at 29°C for 15 minutes	88,5	6,4	—	—	94,9	57,5	7,6	15,6

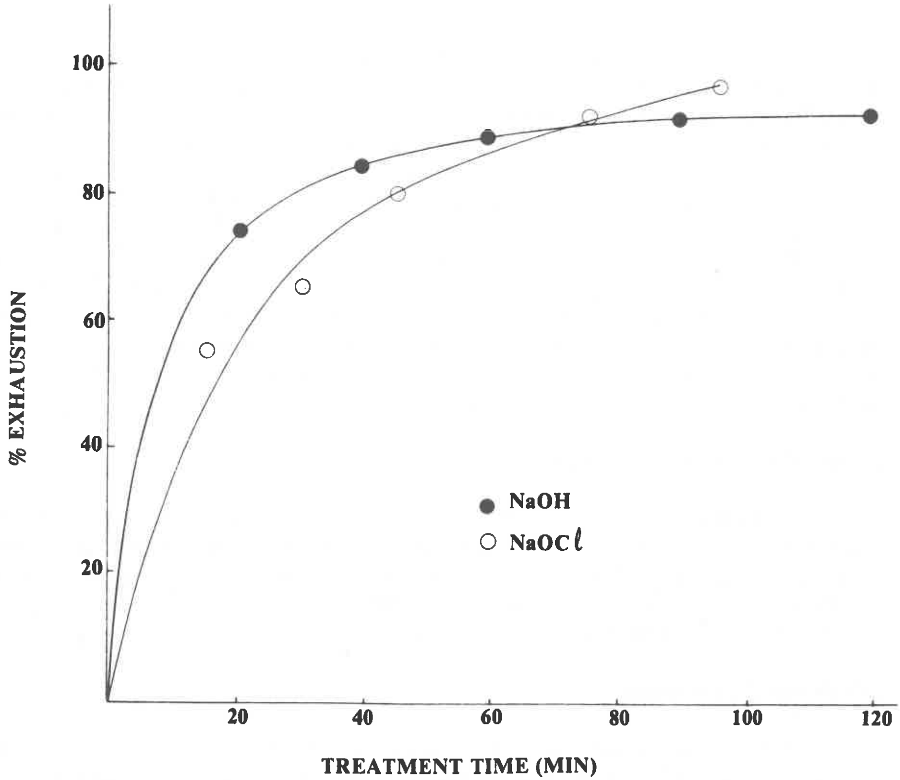

Fig. 1 The rate of exhaustion of 0,4% (m/v) NaOH solution at the boil and 0,4% (m/v) NaOCℓ solution at 25° C onto Phormium tenax fibres

to 8 *per cent* (o.m.f.) sodium hydroxide and 3,5 to 8 *per cent* (o.m.f.) active chlorine.

Preliminary experiments showed that in the case of fibres which had been treated with 2 *per cent* (m/v) sodium hydroxide and subsequently rinsed in water, the concentration of the alkali in the first rinse bowl, after the fourth sample was rinsed in the same bowl, increased to 0,7 *per cent* (m/v) and after the tenth sample to 0,8 *per cent* (m/v). The concentration of the alkali in the second rinse bowl increased to 0,35 *per cent* (m/v) after rinsing five samples and reached 0,5 *per cent* after ten samples. The concentration of sodium hydroxide in the treatment bowl was re-adjusted to 2 *per cent* (m/v) prior to each treatment. It was obvious that too much alkali was lost in the rinsing water and consequently the possibility of an exhaustion process was investigated in order to minimise wastage of chemicals. It was found (see Figure 1) that a 90 *per cent* exhaustion

(7,2 *per cent* NaOH, o.m.f) was achieved after boiling the Phormium for one hour in the caustic soda solution and a 92,5 *per cent* exhaustion after two hours (see Figure 1). The concentration of the alkali in the rinse bowl was approximately 40 p.p.m., which is below the level required by most local authorities. Furthermore, it was found that the chlorine could also be exhausted and 97 *per cent* chlorine was consumed by the fibres within 1,5 hours at room temperature.

Phormium fibre should be relatively clean prior to the chemical treatment. For example, it was noticed that one lot of Phormium fibre, which had a loose particle content of 18 *per cent*, was more glued together after the treatment than another lot which contained only 4,1 *per cent* of loose particles. Fibres glued together caused excessive fibre breakage during the carding operation and produced an irregular yarn.

Cost of the chemical treatment

A *preliminary* costing analysis has shown that the treated fibre can be marketed at about 80c/kg . The calculations are based on a 10 *per cent* profit after tax, a capital outlay of R70 000, a yield of 65 *per cent*, a purchase price of Phormium of 32 cents/kg, a quantity of 600 tons processed per year, and on current chemical, water, electricity and labour costs.

Mechanical processing

Some tenacity results of the yarns spun from the Phormium/acrylic blend are given in Table IV. The reduction in the wet tenacity of the fibres is not

TABLE IV

THE YARN TENACITY OF A 225 tex PHORMIUM/ACRYLIC (70/30) YARN MADE FROM UNTREATED OR CHEMICALLY TREATED PHORMIUM FIBRES

Treatment of Phormium	Tenacity	
	Dry	Wet
	cN/tex	cN/tex
Untreated	4,86	5,44
7,8% NaOH + 7,4% Cl_2	4,79	4,74
6,6% NaOH + 4,1% Cl_2 followed by 4,2% NaOH + 6,6% Cl_2	5,01	4,26

considered serious, as the yarn tenacity results show that the low tenacity of the *treated fibres* is not reflected in the yarn tenacity results. This is probably due to the presence of the acrylic fibres.

Subsequently some weft faced fabrics were produced from these yarns using a polyester/cotton warp. These fabrics, which could possibly be used for table mats or wall decorations, etc., were washed in a domestic washing machine and it was found that the fabrics became softer after repeated washing. The possibility of using fabrics, made from blends of these chemically treated Phormium fibres and synthetic fibres for apparel, is to be investigated.

SUMMARY AND CONCLUSIONS

Phormium fibres can be softened, split into finer fibres and partially bleached by treatment with sodium hydroxide and sodium hypochlorite solutions. It was shown that the chemicals could be exhausted onto the fibres, therefore saving chemicals and reducing pollution problems.

The treated fibres were blended with long staple acrylic fibres. The blend consisted of 70 *per cent* Phormium and 30 *per cent* acrylic fibres. The fibres were processed on the best available equipment, and it is possible that even finer fibres can be produced by investigating the mechanical processing in more detail. A yarn (225 tex) was spun and it is possible that finer yarns could be spun.

The dry tenacity of the fibres was not affected by the treatment. The wet tenacity of the treated fibres is very poor, but the wet tenacity of the yarn is not much different from the dry tenacity of the yarn since the fibres were blended with acrylic.

The fibres were whiter after the treatment and the brightness increased from 32 to 46 and increased to 56 when the fibres received an additional hydrogen peroxide bleach. A relatively high light fastness (5 to 6) was obtained after the softening process and the same light fastness was obtained after further bleaching with hydrogen peroxide.

After the sodium hydroxide and sodium hypochlorite treatments the cellulose content was found to be higher and the lignin content lower than in the case of untreated Phormium. The yield of fibre decreased when the treatment was repeated on the same sample.

A preliminary costing has shown that the price of the treated fibre is still relatively low when compared with that of other natural and synthetic fibres.

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THE USE OF PROPRIETARY NAMES

The fact that products with proprietary names (®denotes registered trade marks) have been used in this report does not in any way imply that SAWTRI recommends them or that there are not substitutes which may be of equal value or even better.

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