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USE OF UNCONVENTIONAL FIBRES FOR THE MANUFACTURE OF FABRICS, SITRA COIMBATORE

DP/IND/86/038/11-04

INDIA

Technical report: Chemical modification of pineapple leaf fibres*

Prepared for the Government of India
by the United Nations Industrial Development Organization,
acting as executing agency for the United Nations Development Programme

Based on the work of Peter F. Greenwood
Expert on chemical modification of pineapple leaf fibres

Backstopping officer: J.P. Moll,
Agro-based Industries Branch

United Nations Industrial Development Organization

Vienna

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* Mention of company names and commercial products does not imply the endorsement of the United Nations Industrial Development Organization (UNIDO). This document has not been edited.

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EXPLANATORY NOTES

Local Currency: UNDP Exchange Rate $1 = 18.0 Rupees

Abbreviations Used:

PALF	:	Pineapple Leaf Fibre
% w/w	:	Percentage by weight (gms. per 100 gms.)
EDTA	:	Ethylene diamine tetra-acetic acid (disodium salt)
SEM	:	Scanning Electron Microscopy
o.w.f.	:	On weight of fibre

Units:

Micron	:	One millionth of a metre

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Table of Contents

ABSTRACT

Title and Number of Project : Use of Unconventional Fibres for the Manufacture of Fabrics

DP/IND/86/038/11

Objective and Duration of Activity:

To plan and conduct experiments on chemical modification and enzymatic degumming of PALF with a view to producing finer fibres, and to advise on dyeing and finishing of PALF and other leaf fibres.

(1 1/2 Months)

Conclusions and Recommendations:

Because of the variability of PALF, care must be taken to ensure thorough randomisation of the fibres in experimental studies.

Scouring with a dilute alkali and a sequestrant, followed by peroxide bleaching, may significantly improve fibre quality prior to spinning.

Mechanical separation of the fibres should not be neglected. Long fibre combing equipment similar to a flax "hackling" machine may be effective either before or after the chemical treatment.

Suitable industrial co-operation for enzyme studies has been established.

A programme of work on the dyeing of PALF has been drawn up.

INTRODUCTION

Previous work on this project has been summarised by Mr. Jarman and Mr. Terkelson (1,2,3) and later in the report of Dr. Cherian Iype, an expert in Machinery Design (4).

The defined programme of work for the present mission was set down as annex 6 to the report on the second mission (2), and is reproduced as annex I to this report.

Work at SITRA in connection with this programme began on January 10, 1991 and is scheduled for completion on or about February 14, 1991.

The original objectives, as set down in the report on the second mission, were to

- set up experiments on chemical degumming of PALF towards making them finer

- advise on dyeing and finishing of PALF.

Recommendation 3 of the Report on the second mission states that "the promising results obtained with chemical treatment should be measured after consultation with expert in fibre chemistry".

Unfortunately no detailed description of these studies appears in Jarman and Terkelson's report. A sub-section 4.4 chemical degumming, appears in the list of contents, but not in the body of the report. However, some SITRA internal reports have been made available, and the results are described in the present report.

I. CHEMICAL DEGUMMING

A. Previous Work at SITRA

Before the arrival of this expert, two series of experiments on the chemical degumming of PALF had been carried out at SITRA.

These are reproduced as Annexes II and III to this report.

In the first series of experiments, described in Annex II, a variety of chemical agents, including acids, alkalis, salts and enzymes, were applied in aqueous solution to raw PALF under varying conditions of temperature and time. The properties (fineness and tenacity) of the treated fibres were evaluated, and the surface characteristics studied by means of scanning electron microscopy.

The conclusion reached as a result of these trials was that sodium hydroxide could be used to remove gum from PALF without any severe loss in fibre strength. However, it would be necessary to optimise the process parameters.

Annex III describes a second series of experiments using sodium hydroxide, varying the parameters of concentration, time and temperature to achieve optimisation. The conclusion drawn from this study was that optimum conditions, giving some reduction in fibre thickness without producing excessive damage, would be a 4% concentration at room temperature (30° C) for four hours.

These were, in fact, the mildest conditions included in the study, and this conclusion begs the question that by reducing the severity of treatment even further, a better result may be obtained?

B. Work Done During the Mission

3.1 Examination of SEM Photographs

The SEM photographs taken during the work described under I.A were re-examined at the start of the mission.

The structure of the raw PALF fibres can be seen from these photographs to be composed of several multi-fibrillar bundles, perhaps a hundred or more fibrils in a bundle. The ultimate fibrils are between two and five microns in diameter, while the raw fibres are perhaps 40 - 100 microns in diameter, although this varies widely. This is two to four times the diameter of the more conventional natural cellulosic fibres, such as cotton (15 - 22 microns) and flax (5). An interesting feature to be seen in the raw fibre is a number of transverse markings across the fibrils, as though each fibrillar bundle has been tied. In most of the photographs of treated fibres these tie-marks have disappeared, and this seems to result in breakdown of the bundle structure, with consequent fibrillation and fibrillar entanglement. Sodium hydroxide solution at high temperature appeared to be particularly severe in this respect, indicating that such a treatment should be avoided at least prior to spinning.

Some cross-sections showed indications that a central lumen is present in the fibrils, but most were unclear. The preparation of good fibre cross-sections is difficult, and this point may not be clarified until the operators have obtained more experience.

B.2 Chemical Degumming (First Series)

Following lines suggested by the Scottish College of Textiles during the Fellowship training visit of Mr. S.Rajendran, it was decided to study the effects of concentrated sodium hydroxide (21.9% w/w). For processing cotton this would be regarded as "mercerising strength", and produces the maximum swelling effect. It was thought that the fibre swelling might break the adhesions between the fibrillar bundles.

Vegetable pectins and gums are often associated with alkaline earths, particularly calcium, which appear to assist in the structural bonding. It was thought that the presence of a sequestrant, EDTA, would help to remove the alkaline earths and weaken the bonding. A patent for such a process has been filed for the treatment of flax (GB 2, 186, 002) but the general principles have been known in the industry for some time, for example removal of cotton seed fragments.

The effect of dilute sodium hydroxide has been established in previous trials, but a further experiment was carried out to examine the influence of EDTA.

In the last experiment in this series, the effect of hydrochloric acid treatment was studied. This reagent also dissolves the alkaline earths. A 4N concentration of hydrochloric acid was used.

All these experiments were carried out at room temperature, for one hour, followed by rinsing and neutralising.

Part of the sample which had been treated with hydrochloric acid was re-treated with dilute (5% w/w) sodium hydroxide, to attempt to dissolve any precipitated pectic acids, for 15 minutes at room temperature, and neutralised. After drying, the samples were studied by SEM and evaluated for fibre strength and fineness. The results are described and discussed in sub-section B.3.

Chemical Degumming (Second Series)

Early on during the mission, a short series of spinning trials was carried out, to look at the effect of chemical softening on the spinnability of raw PALF (see Chapter II).

Carding was carried out on a cotton card, modified as described by Dr. Cherian Iype (4). The formation of sliver presented no problems, and this result indicated that bleached fibre might also process well through this card.

The decision to undertake trials on bleached fibre is supported by a report from the Jute Technological Research Laboratories, Calcutta (6), which indicates that PALF can be successfully bleached in fibre form. It is claimed that a mild bleach with hydrogen peroxide improves fibre fineness and yarn flexibility, although at some cost to yarn strength. It was further observed that separation of fibre at the carding stage was better for bleached fibre than for raw fibre.

Three bleaching treatments have been studied, with the objective of obtaining further breakdown of the fibres; not necessarily to obtain good whitening as this is better carried out after yarn formation.

1. Hydrogen Peroxide

$H_2 O_2$ (30%)	:	1 ml/l
Sodium Silicate	:	10 gm/l
Sodium Hydroxide	:	10 gm/l
Wetting agent (Ectalon) (non-ionic)	:	0.1 gm/l

Liquor ratio 1 : 50

28 - 30° C, 16 hours.

Neutralised with acetic acid, washed and dried.

2. Acid Hypochlorite

Sodium hypochlorite	:	1 gm. active chlorine/l.
Wetting agent	:	0.1 gm./l.

Adjust to pH 4 with acetic acid.

Liquor Ratio 1 : 50

28 - 30° C for 1 Hour.

Anti-chlor, neutralise, wash and dry.

3. Alkaline Hypochlorite

Sodium hypochlorite	:	1 gm. active chlorine/l.
Wetting agent	:	0.1 gm./l.
Soda ash	:	1 - 2 gm./l.

to give a pH 11 - 12

Liquor Ratio 1 : 50

28 - 30° C for 2 Hours

Anti-chlor, neutralise, wash and dry.

Description of both series of treatments are given in Table 1, and test data on the treated fibres are shown in Table 2

Table 1 : Chemical Degumming Treatments

	Treatment
1st Series	
1	22% w/w NaOH, Cold, One Hour
2	22% w/w NaOH + 5 gm./l. EDTA, Cold, One hour
3	5% w/w NaOH, Cold, One hour
4	5% w/w NaOH + 5 gm./l. EDTA, Cold, One hour
5	4 N HCl, Cold, One hour
6	As 5, then 5% w/w NaOH, Cold, 15 Minutes
2nd Series	
7	H_2O_2 (recipe 1)
8	Acid Hypochlorite (recipe 2)
9	Alkaline Hypochlorite (recipe 3)

Table 2 : Test Data on Treated Fibre

Treatment No.	Fibre Fineness (tex)	Fibre Strength (gm.)	Tenacity gm./tex
Raw Fibre	2.99	88.2	29.5
1	2.99	74.3	24.8
2	2.97	61.0	20.5
3	3.30	106.9	32.4
4	2.38	98.4	41.3
5	3.25	78.6	24.2
6	3.49	78.8	22.6
7	3.07	108.5	35.3
8	2.31	84.7	36.7
9	3.57	83.7	23.4

B.3. Results and Discussion

Examination of SEM Photograph

The examination of earlier photographs (sub-section B.1) indicates that the important features to be considered are:

- transverse markings which may hold the fibrils in a bundle structure,
- separation of fibre bundles,
- separation of fibrils from the bundle structure and possible fibrillar entanglement
- surface deposits.

The "mercerising strength" NaOH treatments (treatments 1 and 2) show considerable fibrillar separation, which performance, because of fibrillar entanglement, and also in a reduction in strength due to the break-down of the bundle structure.

The treatments with dilute NaOH also show some fibrillar separation, although less severe. Some surface deposits are visible on the fibres treated with NaOH alone, but the addition of EDTA appears to have reduced or even eliminated these.

In the photographs of the treatment with hydrochloric acid the transverse markings are still visible, but there are considerable surface deposits, which the subsequent treatment with sodium hydroxide has not removed.

The photographs of both the peroxide-treated and alkaline hypochlorite-treated fibres show fairly smooth surfaces, but with some fibrillation, while the acid-hypochlorite treated fibres have retained the transverse markings, but show considerable surface deposits.

Test Data

The test results are given in Table 2.

The measurements of fibre fineness present a somewhat confused picture, with some of the treated fibres appearing to be coarser than the original untreated fibre. These figures must therefore be treated with some suspicion.

This is not an easy test to carry out on PALF fibre in the raw state. The fibres adhere together and the technician has to decide if he is dealing with a single fibre or several. Proper randomisation of sampling may be a problem, and it is possible that different parts of a leaf may be made up of fibres of differing fineness; for example fibres at the tip of the leaf may be finer than those at the base.

For this reason it is suggested that fineness data should be obtained after the fibre has been through a mechanical separating and randomising process. Tests carried out on carded sliver, for example, are likely to be much more reproducible and reliable.

Fibre strength data appear to be suffering from a similar problem, with some of the treated samples giving higher results than the original material. More of the treated samples were excessively weak, but the "mercerised" fibres were the lowest in strength, confirming the theory that fibrillar separation results in a weakening of the fibre structure.

B.4. Enzymatic Degumming

During the mission Dr. S.Sivakumaran, Head of the Textile Chemistry Department at SITRA, visited the Indian Office of Novo Industri A/S in Bangalore, and rade arrangements for the evaluation of a range of enzyme products as potential degumming agents for PALF.

Novo Industri A/S is a world-renowned manufacturer of commercial enzyme products, with first-class technical expertise, based in Denmark.

Unfortunately, at the end of the mission, these products had still not been received at SITRA.

II. CHEMICAL SOFTENING

Raw PALF fibres, because of their large diameter, are relatively stiff compared to cotton for example, and tend to produce hairy yarns.

Trials were therefore carried out to explore the possibility that an application of a chemical softener might reduce fibre stiffness sufficiently to produce an improvement in the hairiness.

Two types of softener were used in the study

- a polyethylene emulsion (non-ionic)

- a cationic softener, Katasoftener SP from Ahura Chemical Products, Bombay.

The polyethylene emulsion was applied at concentrations of 1 and 5 gms. per litre, with a pick-up of 150% o.w.f. and dried. These applications produced no discernible improvement in lubrication or softness, and were rejected.

The cationic softener was applied at concentrations of 2, 5 and 7 gms. per litre, immersed for 15 minutes, liquor ratio 1 : 50, squeezed to a pick-up of 150% and dried. All gave noticeable improvements in lubrication and softness, but the treatment at 7 gms. per litre produced a greasy handle.

In order to assess the spinning performance of the softened fibre, a larger sample (100 gms) of raw PALF, cut to 3 inch staple length, was treated with 5 gms. per litre of cationic softener, with a pick-up of 150% o.w.f., and dried.

III. SPINNING TRIALS

A. Chemically Softened Fibre

The chemically softened PALF, prepared as described in Section II, was carded through a Platt semi high production card with modifications suggested by Dr. Iype (4). A satisfactory sliver was obtained.

This was directly spun on a converted silk frame. A similar sample of untreated fibre was spun in the same way.

Test Data:

	Untreated	Chemically softened
Count Ne,	3.41	3.13
Turns/inch	9.0	8.8
Hairiness.	Above range of testing equipment.	
Black board	No apparent difference. Both samples irregular and very hairy.	

Conclusion:

Softening alone is unlikely to improve either the spinnability or the quality of the spun yarn. The fibre diameter must be reduced to produce a fibre which will accept twist more readily. Also there were relatively heavy particles of shive, straw or non-fibrous leaf which can cause breaks, especially if increased spinning speeds are attempted.

B. Bleached Fibre

Following encouraging results from the small-scale studies, a larger quantity (10 - 15 kg.) of the uncut fibre was bleached with hydrogen peroxide. It was considered that the severity of the bleach could be increased, compared with the earlier trial.

The chemical treatment was as follows:

$H_2 O_2$, 30%	:	2 ml./l.
Sodium silicate	:	10 gm./l.
Sodium hydroxide flake	:	8 gm./l.
Wetting agent	:	0.1 gm./l.

Overnight at ambient temperature (outdoors)
Neutralised, Washed and Air-dried.

The fibre so treated was cut into 1 1/2 inch (38 mm.) staple, and formed into a sliver on the Platt semi high production card.

Spinning was carried out on a Shirley mini-spinning range. Two trials were carried out, with two drawings in the first trial and three in the second. The extra drawing produced a noticeable improvement in fibre parallelisation and yarn regularity.

The fibres were still very stiff and straight, so that inter-fibre friction was very low. The chemical treatment may have contributed to the low friction, but it was impossible to be sure about this.

A similar quantity of fibre, pretreated with 4% NaOH room temperature using current SITRA recommendations (Annex III, section 4.1) was also spun under the same conditions with two drawings.

Test Data:

	NaOH Treated	H_2O_2 Treated
Fibre Fineness (tex)	2.75	2.64
Fibre Strength (gm.)	52.3	51.3
Tenacity (gm./tex)	19.0	19.5
Yarn Strength (gm.) (after two drawings).	998	997

Conclusion:

Good spinning is possible with bleached fibre, achieving standards which are at least equal to those of unbleached PALF. Further studies to optimise bleaching conditions are indicated, to establish whether significant improvements in fibre fineness can be obtained.

IV. PROGRAMME FOR DYEING STUDIES

One of the objectives of the present project is the introduction of PALF into yarns produced on the cotton spinning system.

Products which might be envisaged would include 100% PALF yarns, blends of PALF with cotton, and blends of PALF with man-made fibres.

An experimental plan for dyeing studies on PALF should therefore allow comparisons to be made with cotton dyeing, comparing

- colour yield and build-up
- penetration and fastness to rubbing
- fastness to light
- fastness to water
- fastness to washing.

A useful starting point is provided by Canning and Jarman, who studied the behaviour of a wide range of dyestuffs on sisal and other plant fibres, although PALF was not included (8).

Canning and Jarman make the point that in addition to the dye classes which are normally used in the dyeing of cellulosic fibres such as cotton, namely directs and reactives (vats, azoics and sulphur dyes are not mentioned), other dye classes may produce acceptable results on ligno-cellulosic fibres such as sisal (and possibly PALF).

Some acid, basic and disperse dyes were found to give reasonable results in the colouration of sisal, although wet fastness properties were generally poor.

Based on the work of Canning and Jarman, it is suggested that an experimental programme on the dyeing of PALF should be in two parts:

1) A detailed study on the dyeing of PALF with commonly used cotton dyes, selected from the direct and reactive ranges, and including some vat and sulphur dyes.

2) A short investigation into the dyeing of PALF with acid, basic and disperse dyes.

In part 1, the objectives will be to compare PALF with cotton, with respect to the points mentioned above; colour yield and build-up, penetration and fastness properties.

The objective of part 2 will be to identify any problem areas, for example cross-staining of blends of PALF and man-made fibres, which are not encountered in the dyeing of cotton and man-made fibre blends.

Experimental

Material : Ideally, the material to be dyed should be in the form of yarn or fabric. This is how it is likely to be processed in commercial practice; stock dyeing is almost unknown in the cotton processing industry. Also, instrumental colour measurement is more easily carried out on yarn or fabric, and samples can be more easily mounted for visual comparison.

Preparation : The material to be dyed must be well scoured to ensure absorbency; and bleached, although bleaching may be omitted when very deep shades are being produced.

To assess absorbency, place a drop of water, containing a small quantity of wetting agent, on the prepared material. The drop should be immediately absorbed.

If the absorbency is not sufficient, the scouring temperature must be raised until a satisfactory result is obtained.

Dyeing : (Part 1) Dyeings should be carried out at three or four concentrations, so that colour yield and build-up can be examined. Concentrations of 0.25, 1.0 and 4.0% o.w.f. are suggested. If possible, a similar series of dyeings on bleached, unmercerised cotton should be carried out to provide rough comparisons.

A controllable laboratory dyeing machine, such as the Ahiba, should be used, and the dye manufacturer's recommendations for dyeing in long liquors should be followed.

Evaluation : (Part 1) Colour yield and build-up can be assessed visually, and also instrumentally by using computer colour matching equipment in transmission mode for the analysis of dye liquors, and in reflectance mode for the assessment of depth of shade on the fabric.

The method for carrying this out is as follows : Prepare a series of solutions of the dye at a suitable range of concentrations. Measure the % transmission of each solution, preferably at the wavelength at which it is at a minimum, and plot this against concentration. This graph can then be used to measure initial and final dye concentrations during experimental dyeing, so that the amount of dye absorbed by the fibre can be estimated (Reference : "A laboratory course in dyeing", by C.H.Giles, published by the Society of Dyers and Colourists, Bradford, England).

Penetration of dye into yarns or fabrics may be assessed by examination of cross-sections. In woven fabric a slight displacement of yarns may be enough to show undyed spots under the cross threads. Fastness to rubbing will also give an indication of the penetration.

Fastness to rubbing, light, water and washing should be carried out by the well-known standard methods.

Dyeing : (Part 2) The absorption by ligno-cellulosic fibres of acid, basic and disperse dyes often depends on the amounts of non-cellulosic impurities present. The sisal used in the experiments of Canning and Jarman was not bleached, and probably contained significant amounts of lignin. PALF, on the other hand, will usually be bleached before dyeing, and may therefore be less dyeable with these classes of dyestuff.

Nevertheless, some exploratory studies should be carried out on the dyeing of PALF with acid, basic and disperse dyes. One concentration is sufficient for each dye studied.

Evaluation : (Part 2) The dyeings should be evaluated in terms of the depth of shade and fastness-to washing. Of particular interest would be the amount of staining of the fibres in the washing test, and the use of a multi-fibre strip, available for example from the Society of Dyers and Colourists, is advised.

Dyestuff Selection

It is a good general rule in dyeing experiments to include a red, a yellow and a blue dye so that if suitable products can eventually be identified, a wide range of mixture shades will be possible.

Reactive Dyes : Canning and Jarman have selected certain Procion MX dyes as suitable for the dyeing of sisal. These dyes form a useful initial selection for PALF studies. Procion Brilliant Red MX-8B, Yellow MX-4R and either Blue MX-R or Blue MX-4GD should be included in the first series of dyeing trials.

For red Procion MX dyes ICI recommend an after-treatment with Matexil FC - PN.

Direct Dyes : Canning and Jarman studied a wide range of ICI direct dyes. Unfortunately, many of these have since been withdrawn.

A selection of locally available direct dyes should be made, to identify suitable dyes in the red, yellow and blue colour ranges. Note that several after-treatments for example copper sulphate, can be used to improve fastness properties.

Disperse dyes: Of the dyes tested by Canning and Jarman, Dispersol Red B-2B and Dispersol Blue B-R (ICI) are still available. Both of these were observed to give dyeings on sisal with some light and water fastness.

Other dye classes: Select on an ad-hoc basis, or obtain suggestions from suppliers or dyestuff users.

V. RECOMMENDATIONS

Experimental Design and Testing

Raw PALF is a very variable material. The coefficient of variation of fineness, between measurements of the combined weight of fifty fibres, was found to be as high as 18%. It is therefore very important

a) to ensure thorough randomisation of the raw material before starting experimental work

b) to carry out sufficient testing to ensure a reasonable level of accuracy in the result.

Carding is a quite effective operation for fibre randomisation.

RECOMMENDATION 1:

That all laboratory-scale studies, for example chemical degumming treatments, should be carried out on well-randomised carded sliver.

RECOMMENDATION 2:

In the case of larger-scale trials, which may be carried out on uncut fibres, that the treated fibre should be suitably cut and passed through a card, to ensure randomisation, prior to testing.

Mechanical Processing

The effectiveness of mechanical processing for the separation of the fibre burdles should not be neglected. Some evidence was gained during the mission which suggested that improvements in fibre fineness can be achieved simply by passing the material through a card.

Raw PALF is very similar in length, appearance and structure to decorticated flax. One of the first stages in flax fibre processing is the operation of "hackling", in which the fibre bundles, aligned with roots at one end and tips at the other, are combed with successively finer gauge combs, first in one direction and then in the other.

This type of machinery is manufactured, for example, by James Mackie of Belfast, Northern Ireland.

RECOMMENDATION 3:

That the operation of flax hackling should be studied as a possible means for improving the fineness and cleanliness of PALF.

Chemical Degumming

Work carried out at SITRA prior to the mission has shown that dilute sodium hydroxide can be effective as a treatment for improving fibre fineness. It was decided that this is best carried out at ambient temperature (30^{o} C), as fibre entanglement was found to occur at elevated temperatures. This view is confirmed by SEM

photographs which show that fibrillar separation and entanglement can occur when severe sodium hydroxide treatments are employed.

Even under the presently used conditions, of 4% NaoH at 30°C for four hours, some fibrillation can be observed. It is possible that the treatment can be even less severe and still be effective by employing a sequestrant to remove alkaline earth residues from the gums and pectins, and so assist in fibre separation.

RECOMMENDATION 4:

> That trials be carried out with sodium hydroxide solutions in the range 0 to 4 per cent, with the addition of a sequestrant such as EDTA.

Bleached PALF can be successfully carded, prepared and spun on cotton system machinery, as shown in trials on the Shirley mini-spinning range at SITRA.

RECOMMENDATION 5:

> That trials should be carried out on scoured fibre, processed as under Recommendation 4, to optimise a bleaching treatment using hydrogen peroxide, and to establish its effectiveness in improving the quality of PALF for spinning.

Although no experimental work was carried out during the mission on the use of enzymes, this remains a promising approach.

RECOMMENDATION 6:

That contact between SITRA and Novo Industri A/S should be maintained, and trial products evaluated at laboratory level for effectiveness in improving fineness and removing or preventing the formation of surface deposits.

These trials may not reach a satisfactory conclusion within the time scale of the current project, but efforts should be made by SITRA to enlist the co-operation of local industry. For example, the hank processing unit seen at the Lakshmi Mills Company, Coimbatore could be adapted for chemical degumming trials.

VI. ACKNOWLEDGEMENTS

Sincere thanks are offered to the Director and Staff at SITRA for making this visit particularly pleasant and memorable; especially to Miss Indra Doraiswamy, Deputy Director, and to Dr. S.Sivakumaran and Mr. S.Rajendran, who have provided not only welcome assistance in completing the technical programme, but also some fascinating and entertaining glimpses of Indian customs and traditions.

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ANNEX I

Job Description (11.04 Revised)

Post Title	:	Chemical Modification of PALF
Duration	:	1 1/2 months
Date Required.	:	Any time after June, 1990
Duty Station	:	Coimbatore, South India
Purpose	:	The Project on the "Use of Unconventional Fibres for the Manufacture of Fabrics" is aimed at developing suitable techniques/ machinery for the production of yarns and fabrics from leaf and bast fibres like pineapple, and banana. Unconventional fibres are generally very coarse due to the presence of high level of Lignin and pentosans. In order to make finer yarns and thereby finer fabrics, the unconventional fibres are to be made finer by chemical treatments.

Contd. ....

ANNEX I (Contd.)

Duties	:	The expert will work jointly with the Director, SITRA and National Project Director. He is expected to - set up experiments on chemical degumming of PALF towards making them finer - advise on dyeing and finishing of PALF.
Qualifications	:	A Post Graduate Degree (preferably doctorate degree) in Textile Chemistry.
Experience	:	Extensive experience in enzymic degumming/chemical modifications of textile fibres, particularly natural fibres.
Language	:	English
Background Information	:	SITRA is engaged in "the manufacture of fabrics from unconventional fibres" PALF extracted from pineapple leaves using decorticating machines is very coarse (25 - 35 denier) due to the presence of various gummy matters like Lignin, Pentosan etc. In order to produce finer yarns, PALF is to be made much finer. With a view to make them finer, chemical modifications/ enzymic degumming are to be tried.

ANNEX II

USE OF UNCONVENTIONAL FIBRES FOR THE MANUFACTURE OF FABRICS

Part II - Chemical Degumming of Pineapple Leaf Fibres

Miss Indra Doraiswamy
The South India Textile Research Association, Coimbatore - 641 014

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1. Introduction

Pineapple leaf fibres (PALF) is a multi-cellular lignocellulosic fibre obtained from the leaves of the plant. The fibre is extracted[1] from the leaves either by mechanical means or by retting the leaves in water. Fresh leaves yield about 2 to 2.5% of fibre. The acreage under pineapple cultivation in India, is estimated to be around 87,200 hectares[2].

The distribution of pineapple plantation in different States in India is given in Appendix I.

The cultivation of pineapple fruit is increasing : Consequently an increase in the production of the fibre is also envisaged. It is estimated[3] that, approximately 6 lakh metric tonnes of fibre may be available in the country if it is properly extracted and collected. The identification of suitable end use applications for pineappleleaf fibres could be expected to open up new avenues for substantial employment generation. Hence, a project has been undertaken at SITRA (The South India Textile Research Association), Coimbatore with UNDP assistance towards assessing the potentialities of PALF.

2. Material

Pineapple leaves were obtained from Basel Mission Farm, Moodibidri, Belvai, DK, Karnataka State.

Fibres are extracted from leaves using power operated decorticating machine.

3. Experiments

3.1 Chemical Constituents

The chemical constituents of the PALF sample were estimated by standard methods[4] and are given in Table 1. PALF contains more than 30% of gummy matter, and the gummy matter mainly consists of Lignin, Pentosan and Pectin. This makes the fibre stiff in nature. Some pretreatment, it is thought, may help to remove the gummy matter and render the fibre soft and fine.

Table 1 : Chemical Constituents of PALF

Chemical Constituent	Range in percent
Alpha Cellulose	61.3 - 69.6
Beta Cellulose	4.4 - 12.5
Gamma Cellulose	3.3 - 11.1
Residual Gum	27.2 - 36.0
Fat and Wax	0 . 98
Ash	0 . 74

3.2 Chemical Degumming

In the case of Ramie, opening and cocking with reducing agents is reported[5] to reduce the gum content significantly. In another publication[6] treating the fibres with Sulphuric Acid or Sodium Hydroxide solutions are recommended for the removal of lignin and similar gummy materials. However, only scanty references[7] are available with regard to degumming of pineapple leaf fibres. Therefore, a series of degumming trials on PALF have been carried out at SITRA using the commonly available degumming chemicals, so as to assess the extent to which PALF could be made finer. During degumming, the surface nature and the rheological and mechanical properties of the fibres could be expected to undergo degradation. It is therefore, essential to study the changes in the topographical and mechanical properties of these fibres critically so that the ultimate fibres modified for spinning would have certain minimum values. In connection with this UNDP sponsored project, SITRA has acquired a Scanning Electron Microscope (SEM - of high resolving power) Model - JEOL T330 A and an Instron Tensile Tester Model 6021. These instruments were utilised for fibre quality requirements.

The degumming trials carried out at SITRA on PALF are given in Table 2. After each degumming trial, the corresponding fibres were tested for denier (Gravimetric Method), Tenacity (Instron Model 6021) and Topography (SEM - Model JEOL T330 A).

Table 2 : Degumming Experiments on PALF

Experiment No. 1	Details of Experiments
1	PALF treated with 5% Sulphuric acid at room temperature for a duration of 5 hours (Material : Liquor Ratio = 1 : 20).
2	PALF is treated with 5% Sodium sulphate at the boil for 2 hours.
3	Fibre treated with 5% Sulphuric acid (Material Liquor Ratio = 1:20) at 60°C for 3 hours. Then the fibre is treated with 5% Sodium sulphate at boil for 2 hours.
4	PALF treated with 1% solution of Potassium alum (Material : Liquor Ratio = 1:20) at room temperature for 2 hours.
5	PALF treated with 5% Sodium hydroxide at 60°C for 5 hours (Material: Liquor Ratio = 1:20).
6	PALF treated with 1% Zymex (enzyme) for 30 minutes at room temperature. Then the heated material is again treated with 1% Hydrochloric acid at 60°C for 30 minutes.
7	PALF treated with 1% Zymex overnight at room temperature. Then the material is again treated with 0.5% Sodium hydroxide at the boil for 1 hour.

Table 2 (Contd.)

Experiment No. 1	Details of Experiments
8	PALF is treated with 5% Sulphuric acid at room temperature for 4 hours. Then the material is treated with 1% Potassium alum at room temperature for 5 hours. Then the material is again treated with 0.5% Sodium hydroxide at the boil for 1 hour.
9	PALF is treated with 0.3% Sulphuric acid at room temperature 2 hours. Then the material is treated with concentrated Acetic Acid at room temperature for 5 hours.
10	PALF is extracted in Soxhlet apparatus for 4 hours at the rate of 6 cycles/hour. Then the material is treated with 4% Sodium hydroxide at 80°C for 1 hour.

The denier and tenacity values of the various chemically degummed fibres are given in Table 3.

Table 3 : Denier & Tenacity Values of Chemically Degummed PALF

Fibre Code	Fibre Properties	
	Tex	Tenacity (g/tex)
1) Fibres from exp. no. 1	3.17	25.3
2) Fibres from exp. no. 2	3.09	22.1
3) Fibres from exp. no. 3	2.57	6.23
4) Fibres from exp. no. 4	3.25	32.9
5) Fibres from exp. no. 5	2.70	29.2
6) Fibres from exp. no. 6	2.96	20.7
7) Fibres from exp. no. 7	2.92	21.8
8) Fibres from exp. no. 8	3.12	18.3
9) Fibres from exp. no. 9	3.37	17.1
10) Fibres from exp. no.10	3.72	24.2

Of all the treatments, that with Potassium alu yields the strongest fibre. The finest fibre is offered by treatment with Sodium hydroxide. However, the fineness improvement (due to removal of gummy matter in the fibre) cannot be considered in isolation, since the fibres must be sufficiently strong to withstand tensions imposed during the spinning process. Acid treatment as well as treating the fibre with Sodium Sulphate gives moderate improvement in fibre fineness, but treating the PALF with Sulphuric Acid and Sodium Sulphate together (experiment no. 3) renders the fibres very weak and makes them unsuitable for spinning, even though the linear density of the fibres obtained from this trial is the lowest.

Methanol extraction of PALF (experiment no. 10) or treatment with 0.3% Sulphuric acid/concentrated Acetic Acid do not seem to improve the linear density of the fibres very much.

Treating the fibre first with 5% Sulphuric acid then with 1% Potassium alum and finally with 0.5% Sodium hydroxide even though results in moderate improvement in fibre fineness, but reduces fibre tenacity markedly and therefore cannot be considered as an appropriate degumming agent. Of the remaining three treatments, the one subjected with 5% Sodium hydroxide produced yielded the finest fibre. The tenacity of the resultant fibre was also, incidentally, found to be the highest. Treatment of PALF with Enzyme followed by acid (experiment no.6) and Enzyme followed by alkali (experiment no. 7) both resulted in a more or less similar fibre in terms of linear density and strength.

On the whole, the above set of trials indicate that Sodium hydroxide could be advantageously used for the removal of gummy material from pineapple leaf fibres without very much affecting the fibre strength. However, the various process parameters to be adopted during alkali treatment need optimisation so as to achieve the finest possible fibre with minimum reduction in tenacity. This forms the subject matter of another paper (III - Optimisation of Degumming of PALF using Sodium Hydroxide).

3.2.1 Topography of PALF after Chemical Degumming

In order to find out whether any surface damage has occurred due to treating the PALF with chemicals, observations were made using Scanning Electron Microscope. Since the PALF is fairly long (due to its multi cellular nature), sample preparation for SEM was fairly simple. Fibres, both untreated and treated (at various stages of degumming) were mounted on a standard specimen holder, about 1 cm. in diameter. The specimen was then coated in vacuom with a thin gold layer and finally examined in the JEOL T 330A Scanning Electron Microscope (SEM) at an accelerating potential of 10 KV.

Figures 1 and 2 show the morphology of untreated PALF. These figures indicate scaly, cellular structure with vegetable matter intact. The micrograph of transverse section (figure 3) reveal that there is no trace of lumen. This may be due to the high gum content of the fibres. Only when the gum content is substantially reduced (by treating the fibres) could the lumen be seen clearly in the cross sections (fig. 4). It appears that, apart from the possibility of a certain amount of gum remaining in the lumen, the whole cross-section might have smeared by gum during the reactions of the fibres with gum content as high as 30%.

The surface Morphology of fibres treated with Sulphuric Acid is given in Fig. 5. Some fibres appear without any damage whilst some others show moderate rupture. Sulphuric acid treatment imparts an yellowish colour to the fibre and the resultant fibre is soft and lustrous.

Fibres treated with Sulphuric acid and Acetic Acid (successively one after the other) are shown in figs 6 & 7. High level of fibre matting was observed together with substantial peeling-off of fibrous layers. This could be the reason for the low strength values noted for fibres treated in this fashion).

Figs. 8 & 9 represent the longitudinal view of the PALF treated with Sodium Sulphate and Fig. 10, the cross section of the same. Moderate to severe surface damage have appeared to have occurred during the above treatments (Fig. 8 & 9). Also, the traverse section become matted after treatment (fig. 10). This could be probably due to the swelling of the individual filaments in the fibrous (multi-cellular) strand for testing Sulphuric Acid treatment followed by Sodium Sulphite causes very extensive damage to the fibres (fig. 11 & 12) and the individual filaments become highly matted over.

This is quite evident from the values of fibre tenacity reported in table 3. The tenacity was the lowest (6.23 g/tex) for fibres from this treatment. However, due to more effective removal of gummy matter, which is evident from the lowest value of fibre tex obtained, (Table 3) - experiment no. 3, the lumens are visible in the transverse section (fig. 13). The filaments in PALF appear to have separated and formed into coils while treating with Potassium alum (fig.14). However, not much improvement in the linear density of the fibres was found due to this alkali treatment.

Enzymes are known to be very good desizing agents. With a view to estimating their effectiveness towards gum removal, PALF was treated with 1% Zymex (enzyme) for 30 minutes at room temperature. Then the treated fibres are again treated with 1% Hydrochloric Acid at 60°C for 30 minutes and the cross section of the resultant fibres is shown in fig. 15. Substantial gum removal during this treatment is quite evident from the re-appearance of lumen in the fibre cross section. However, due to swelling of the individual filaments in the multicellular fibrous structure, joining together of strand is also observed. Vegetable matter can be seen in some places (fig. 16). In fibres treated with Sodium Hydroxide, the strands appear to be well separated (fig. 17). This may be due to very good gum removal (which cement together the individual filaments in the fibrous structure). This is also confirmed by marked reduction in the tex value of the fibres. Finally, the fibres were treated with Sulphuric Acid followed by Potassium Alum followed by Sodium hydroxide. The exact procedure of treatment is explained in Table 2 (treatment no. 8). Very severe surface damage and fibrillation have been noticed (fig. 18) on the fibres that has undergone this particular type of treatment.

4. Conclusion

Chemical degumming of PALF helps to improve the fineness by way of reducing the residual gum present in these fibres. However, improvement in fibre fineness, in most of the cases is accompanied by fibre damage. Treating the PALF with NaoH appears to be a promising degumming method. However, the process conditions need careful optimisation towards achieving the best out of this treatment.

5. Acknowledgement

This project has been undertaken with the financial support of The United Nations Industrial Development Organisation, (UNIDO)Vienna. The author is thankful to UNIDO, Vienna. The author is also thankful to Mr. T.V. Ratnam, Director, SITRA for his keen interest in this investigation. All the SEM photograpns are taken by Mrs. Ananthi of Physics Division. Thanks are due to her also.

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APPENDIX I

Distribution of Pine Apple Plantation in India

	State	Area of Pine Apple Plantation (x 1000 hectares)
1)	Bihar	1.79
2)	Karnataka	3.77
3)	Kerala	9.42
4)	Assam	41.94
5)	Meghalaya	7.90
6)	West Bengal	20.92
7)	Tamil Nadu	0.85
8)	Orissa	0.21
	All India =	87.20

ANNEX III

USE OF UNCONVENTIONAL FIBRES FOR THE MANUFACTURE OF FABRICS

Part III - Optimisation of Degumming Trials on Pineapple Leaf Fibres Using Sodium Hydroxide

Miss Indra Doraiswamy
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1. Introduction

Pineapple leaf fibres (PALF) are coarser with an average fineness of 3.0 tex and somewhat rigid. Therefore, it would be difficult to spin such fibres either as a single component or in blends with other fibres. In order to make them spinnable, it is necessary to make the fibres finer and reduce the rigidity. Degumming is the process that can be used to accomplish this objectives.

The various degumming trials carried out at SITRA towards making, PALF finer are reported elsewhere[1]. Sodium hydroxide proved to be a promising reagent for the removal of gummy material from PALF without unduly affecting the fibre strength. However, the various process parameters to be adopted during alkali treatment needs careful

optimisation for achieving the finest possible fibre with minimum reduction in tenacity. This forms the subject matter of the present study.

2. Objectives

- Optimisation of degumming trials on PALF using Sodium hydroxide towards making the fibres finer.

3. Material

Pineapple leaves were obtained from Basel Mission Farm, Moodibdri, Belvai, Karnataka State.

Fibres are extracted from the leaves using power operated decorticating machine.

4. Experiments

Raw pine apple fibres were treated with Sodium hydroxide with concentration ranges between 4 and 12%. The time of alkali treatment was varied between 4 and 8 hours and the temperature of the degumming bath between 30°C and 90°C. Therefore, the process variables considered in this study are

i) X_1 - Concentration of Sodium Hydroxide (4 - 12%)
ii) X_2 - Time of treatment (4 - 8 hours)
iii) X_3 - Temperature of Degumming bath (30°C - 90°C)

4.1 Methodology

In order to optimise the process variables, a factorial design approach was adopted so that each one of the parameters was taken at 3 levels which are coded -1, 0 and +1. The details of experiment are as under:

Variable	Code		
	- 1	0	1
X_1 (%)	4	8	12
X_2 (hrs)	4	6	8
X_3 (^oC)	30	60	90

The effectiveness of degumming was assessed in terms of weight loss, fineness and bundle strength improvements. For each one of these properties the response surface equation was worked out using the set of data given in Table 1.

Table 1

S.No.	X_1	X_2	X_3	Weight Loss	Denier	Bundle Strength (g/tex)
1.	- 1	- 1	0	17.2	20.1	33.23
2.	- 1	1	0	19.2	20.9	35.40
3.	1	- 1	0	24.5	15.7	43.35
4.	1	1	0	25.1	17.9	42.98
5.	0	- 1	- 1	17.8	16.8	39.53
6.	0	- 1	1	26.0	12.6	28.35
7.	0	1	- 1	18.6	15.0	42.53
8.	0	1	1	29.6	13.0	29.55
9.	- 1	0	- 1	12.1	17.6	42.00
10.	- 1	0	1	20.9	20.2	23.93
11.	1	0	- 1	19.5	18.1	45.60
12.	1	0	1	30.0	13.8	36.83
13.	0	0	0	26.6	17.28	35.85

$$\text{Bundle Strength} = 35.6 + 4.27X_1 + 0.75X_2 + 6.38X_3 + 2.5X_1^2 - 1.25X_3^2 - 0.64X_1X_2 + 2.33\,X_1X_3$$

$$\text{Denier} = 17.28 - 1.66X_1 - 0.99X_3 + 2.22X_1^2 - 2.08X_3^2 - 1.73X_1X_3$$

$$\text{Weight Loss} = 26.6 + 3.71X_1 + 0.88X_2 + 4.81X_3 - 3.74X_1^2 - 1.36X_2^2 - 2.24X_3^2 + 0.7X_2X_3$$

The response surface equations are plotted in the form of contours (figs. 1 - 4). It could be seen that the bundle strength does not have any optimum point. As the temperature of the degumming bath increases, the bundle strength decreases. Maximum bundle strength occurs for the highest concentrations, irrespective of the time of treatment.

However, in the case of denier, there is a saddle point (fig. 4). The lowest denier is obtained at the maximum concentration of NaoH and at the highest temperature. But the fibres are seen to be entangled and also rigid. This makes further processing difficult considering the suitability of fibres for further spinning trials, a concentration of 4% Sodium hydroxide (on the weight of material) and a treatment time of 4 hours at room temperature was selected.

Variability of Denier

The raw fibre denier is observed to vary from 22 to 32 depending upon the lot. Further, it varies even for fibres extracted from a single leaf. The assessment of denier also poses problems while counting the number of fibres. Occasionally 2 or 3 fibres are glued together thus making it appear as a single fibre. This points out that considerable care has to be taken while assessing and interpreting the denier.

4.2 Preparation of Fibres for Spinning

The fineness of PALF ontained after degumming at optimum process conclusions (Sodium hydroxide concentration - 4%, Time of treatment-4 hours - Temperature of degumming bath - 30^{o}C) varies between 1.9 to 2.2 tex: The actual procedure adopted during this treatment is given below:

A 4% concentration Sodium hydroxide solution (on the weight of the material) is prepared with water. (Fibre Water = 1:20) Pineapple leaf fibres are dipped into the solution and kept in the solution for 4 hours (at atmosphere temperature).

Then these fibres are taken out of the NaoH solution washed with hot water and then with cold water. The excess NaoH left over the fibres is neutralised by treating the fibres with 0.1% concentration Hydro chloric acid. The acid treated fibres are again washed with cold water.

Then a 0.5% solution of wetting agent (on the weight of the material) is prepared. The acid treated and washed fibres are dipped into the soap solution and kept for 20 minutes. Then these fibres are taken out, washed and dried.

Now the pineapple leaf fibres are ready for further mechanical processing treatments.

5. Conclusion

The degumming trials using Sodium Hydroxide at different temperatures and time of treatment reveal the following conclusions.

The bundle strength of the pine apple leaf fibre is maximum at the highest concentration of Sodium Hydroxide (12%) irrespective of the time of treatment.

The lowest denier of the pine apple leaf fibre (124.2) is obtained at the maximum concentration (12%) of Sodium Hydroxide and at the highest temperature (90^{o}).

6. Acknowledgement

This project has been undertaken with the financial support of the United Nations Industrial Development Organisation (UNIDO), Vienna. The autnor is thankful to UNIDO. The author is also thankful to Mr. T.V. Ratnam, Director, SITRA for his keen interest in this investigation. Part of the work reported in this paper has been carried out by Dr. P. Balasubramanian who since left SITRA. Thanks are also due to him.

7. Referer-ces

(1) "Use of Unconventional Fibres for the Manufacture of Fabrics"

Part - II Chemical Degumming of Pine apple leaf fibres (PALF)

ANNEX IV

COMPANY VISITS

Four companies were visited in the course of the mission. Brief notes on these, together with the main contacts made, are given below:

1. HANTEX PROCESS HOUSE,
   BALARAMAPURAM-695 501,
   TRIVANDRUM DISTRICT,
   KERALA STATE.

   Mr. Sauleana Moragana Pillai, Director.

   Mr. V.Ravindranatha Panicker, Project Officer.

   Visited on January 28, 1991.

This is a company set-up by the handloom weavers' co-operative for the state of Kerala. The co-operative has about 250 local associations in affiliation, each with about 100 members, so that about 25,000 weavers are represented. About 80% of these can produce only plain weave fabrics, which are often difficult to sell.

Hantex offers dyeing and printing facilities to convert these fabrics into more attractive and marketable products, and can also provide design services and technical advice on machinery improvements to increase the versatility and quality of handloom production.

Mr. Panicker is interested in the design potential of PALF and intends to visit SITRA in the near future.

Note: An application for funds to expand the fabric design services was recently rejected by UNDP.

2. UNITED BLEACHERS LIMITED.
P.B.NO.12,
METTUPALAYAM-641 301.

Mr. S.Jayachandran, Factory Manager.

Visited on February 1, 1991.

This dyeing, printing and fabric finishing unit is part of the Lakshmi group of companies. Production capacity is 1,00,000 metres per day. About 60% of production is exported, mainly to the U.S.A.

J-box peroxide bleaching is used, with some open-width (pad-roll) bleaching for delicate fabrics. Dyeing is either in open jigs or continuous; printing by flat-screen and rotary screens.

An ACS computer colour matching system is in operation, and it is planned to introduce full computer control into the dyehouse.

The effluent treatment plant is quite impressive. Much of the treated water is re-cycled for re-use.

They have some experience in processing imported linen fabrics which might be useful for application to PALF fabrics in any future commercial developments.

3. THE LAKSHMI MILLS COMPANY LIMITED,
348 AVANASHI ROAD,
COIMBATORE-641 037.

Mr. T.L.Viswanathan, Mill Manager.

Visited on February 2, 1991.

Lakshmi Mills is one of the largest cotton spinning companies in Coimbatore. There are four mills in the group, employing over 5,000 people. Count range is from 10s cotton count (open-end) to 2 fold 200s (ring spun). Much of the machinery is manufactured in

their own engineering works, in co-operation with Rieter of Switzerland.

There is a yarn processing plant which includes singeing, mercerising and hank bleaching operations. These would be interesting for the PALF project; singeing could be examined to reduce the yarn hairiness, and the bleaching process could be adapted to the treatment of fibre bundles. Mr. Viswanathan thought that both of these could be carried out without too much difficulty.

4. GODWELL ENGINEERING PRODUCTS,
   AVANASHI ROAD,
   COIMBATORE.

   Mr. S.K.Devarajan

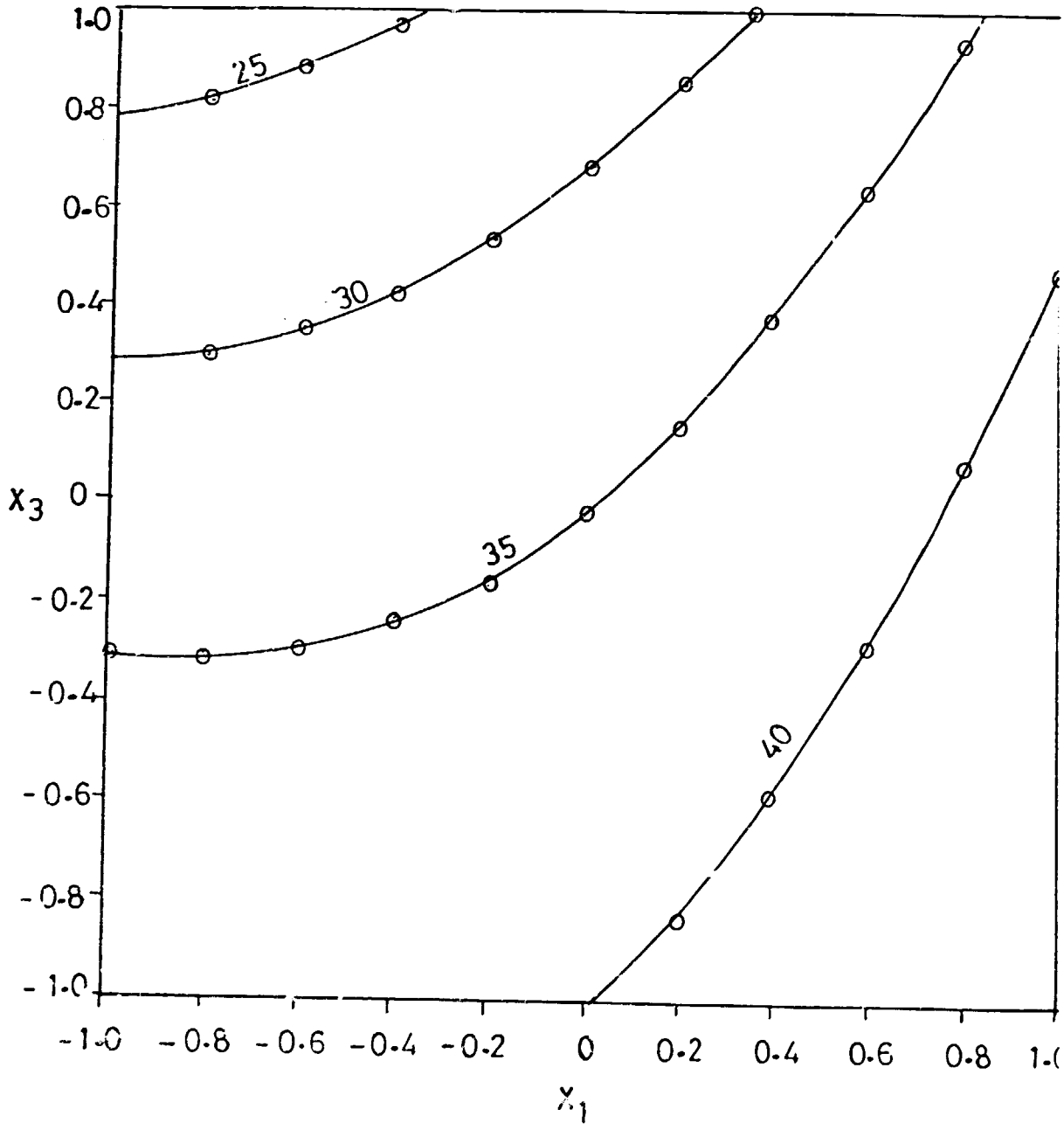
   Visited on February 8, 1991.

This company manufactures machinery for extracting coir fibre from the coconut husks. Following Dr. Cherian Iype's visit in December, they are working on a modified machine designed for the extraction of PALF.

Mr. Devarajan indicated that he was familiar with the type of equipment used for hackling flax, and might be a useful co-operator in developing machinery for the mechanical cleaning and combing of PALF by similar means.

CONTOURS FOR BUNDLE STRENGTH

(1)

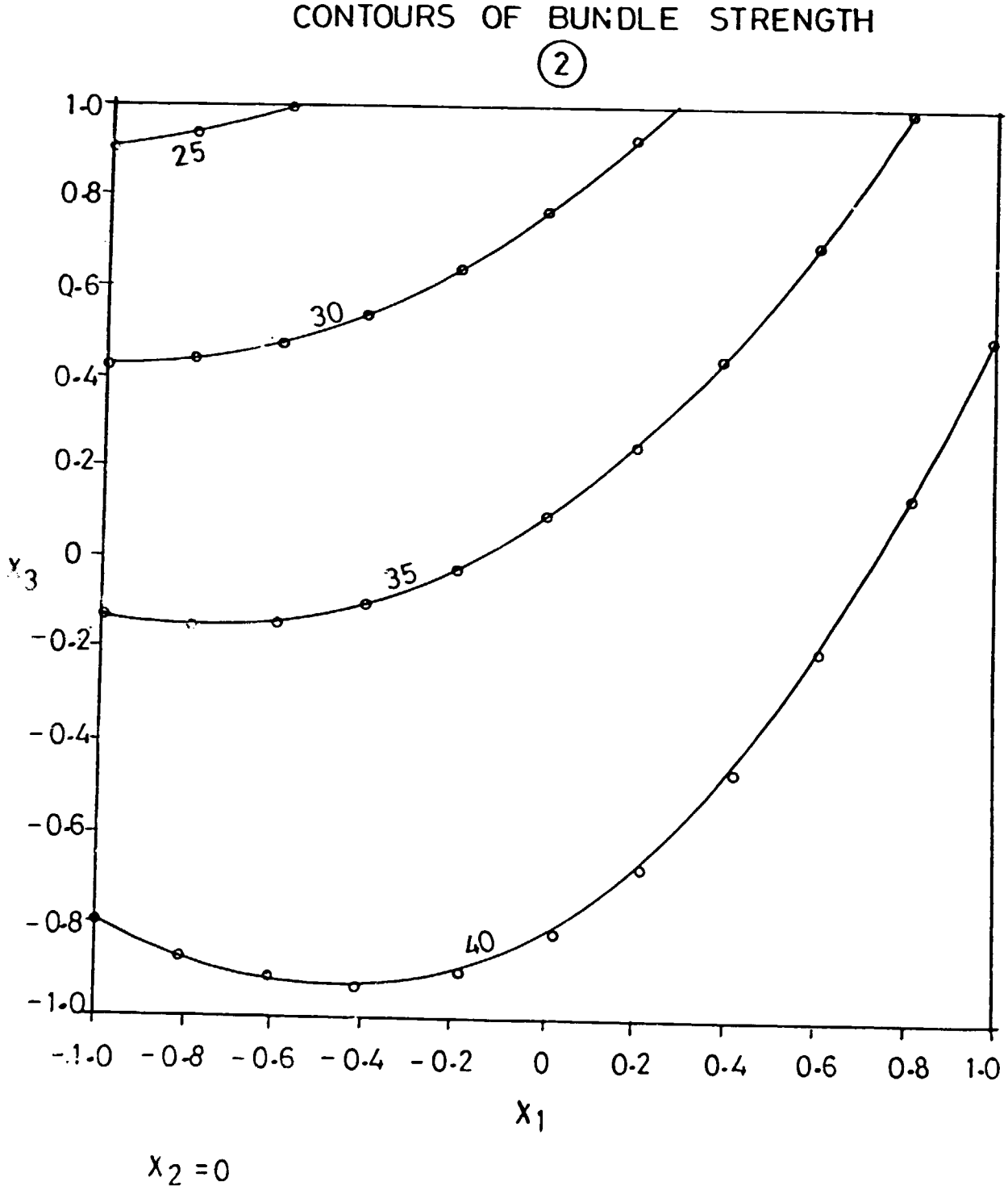


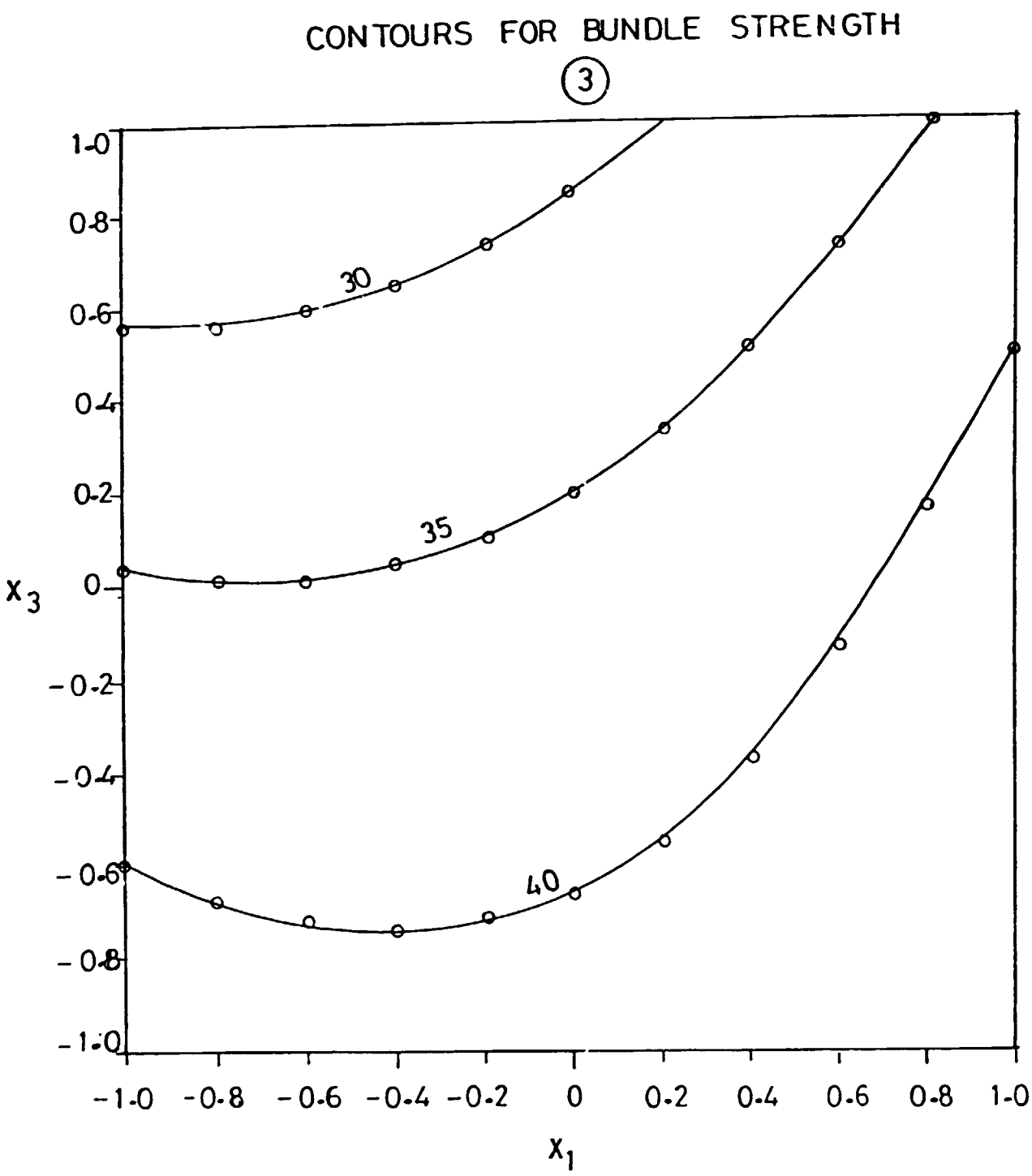
$X_2 = -1$

X_1 = Conc. of NaOH

X_2 = Time of treatment

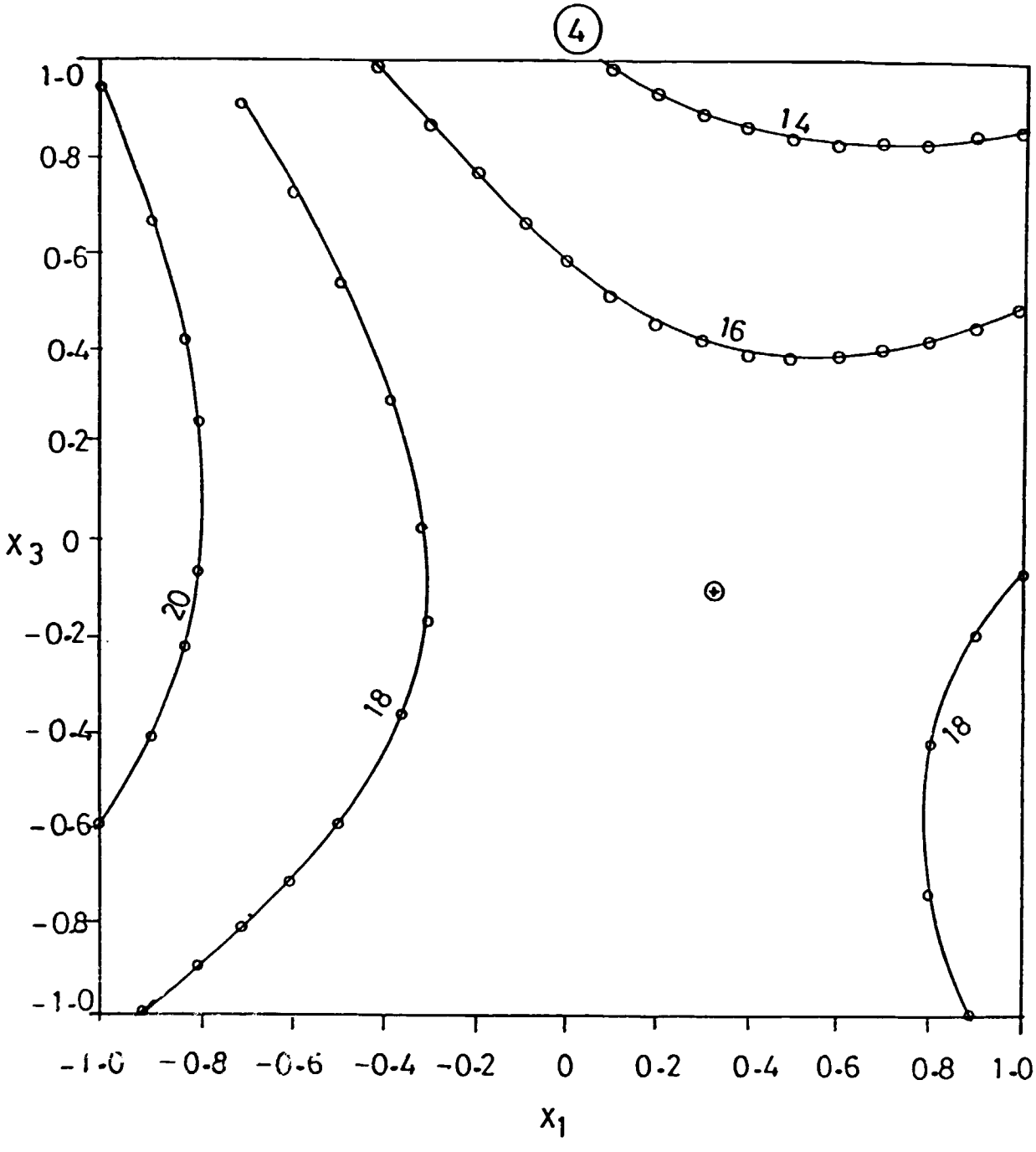
X_3 = Temp.


CONTOURS OF BUNDLE STRENGTH
(2)
25
30
35
40
1.0
0.8
0.6
0.4
0.2
0
-0.2
-0.4
-0.6
-0.8
-1.0
X_3
-1.0 -0.8 -0.6 -0.4 -0.2 0 0.2 0.4 0.6 0.8 1.0
X_1
$X_2 = 0$


CONTOURS FOR BUNDLE STRENGTH
3
30
35
40
1.0
0.8
0.6
0.4
0.2
0
-0.2
-0.4
-0.6
-0.8
-1.0
X_3
-1.0 -0.8 -0.6 -0.4 -0.2 0 0.2 0.4 0.6 0.8 1.0
X_1

$X_2 = 1$

CONTOURS FOR DENIER



X_1 = NaOH conc.

X_3 = Temp.

Saddle point using differen-tiation function $\left\{\begin{array}{l} X_1 = 0.34 \\ X_3 = -0.1 \end{array}\right.$