PAKISTAN SCIENCE FOUNDATION

PROJECT

ON

# UTILIZATION OF PINE NEEDLE FOR PAPER MANUFACTURE

BY

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# C O N T E N T S



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## 0- INTRODUCTION

Prior to the seperation of East Pakistan, Pakistan was self sufficient in paper. Most of the Paper Mills were located in former East Pakistan (Now Bangladesh). This was because the basic raw material i.e. bamboo and jute for paper industry were abundantly available there and the quality of the paper produced was very good. With the creation of Bangladesh, a grave situation arose and the Government imported huge quantity of paper to meet the requirements of the country. At that time paper industry was given top priority and some Paper Mills were installed using bagasse as a raw material. But still the production of paper within the country is not enough to meet the requirements of the country.

The covered area under forests in Pakistan, is limited i.e. about 4.0%. This covered area is very low as compared to the other paper producing countries of the world. Mostly wood is being used in most of the foreign countries for the production of pulp and paper. But in our country we cannot use wood pulp for paper production, as the forest wood is limited in quantity and very costly. At present, wood is being used in the construction of houses etc. Therefore, in order to meet the demand of our paper industry, we have to look for other non-woody raw materials.

In Pakistan, at present the major raw material available for making paper is sugar can bagasse. A few mills have been installed to use bagasse as a raw material for paper production and one more Mill is proposed to be installed at Faisalabad. But it may be mentioned that the season of sugar cane crushing is short and limited quantity of bagasse is produced in these Sugar Mills. For example, bagasse produced in Charsadda Sugar Mills is sufficient only for three months for the Charsadda Paper Mills.

In Pakistan, rice straw, wheat straw, cotton waste and grasses are the other common raw materials used for the manufacture of pulp and paper. Because of their extensive use as animal feed, wheat straw cannot be made available in large quantity for this purpose. The use of grasses and reeds as raw materials for the manufacture of paper is increasing throughout the world. It has been reported that the quality of end product and the economics of production of pulp and paper from grasses and reeds have been proved, beyond any doubt, and that they compare favourably with those manufactured from woody plants. In Pakistan also, some grasses are used in limited quantity and blended with imported pulp for the production of paper.

The study of "the utilization of pine needles for paper and board manufacture" was undertaken with a view to utilize this abundantly waste forest raw material for useful industrial purposes. Its profitable use, for paper industry will not only add to the economy of the country but at the same time it will create job opportunities for the down trodden people of these hilly areas. Moreover, the presence of these needles in the forest is itself a nuisance and is the cause of fire in these forests. Therefore, the removal of these needles will be of assistance to the forest industry.

In this report pine needles are used as a raw material for the production of pulp by the sulphate process. The report is composed of six chapters related to different aspects of production of paper from pine needles. It is hoped that the utilization of pine needles as a raw material for paper will contribute to the development of the country.

## 1. RAW MATERIAL

### 1.1. Raw material for pulp and paper:

Wood is generally used as a raw material for the manufacture of pulp and paper. At present, most of the common species of both soft and hard wood are used in some of the foreign countries for the production of pulp and paper. The so called soft woods or coniferous woods are generally preferred for the manufacture of strong papers, because of the greater fibre length of the cellulose isolated from these woods. The hard woods, however, are finding increased use because of their abundance and because of modifications of manufacturing methods which make them more desirable for certain grades of paper, either alone or mixed with pulp produced from coniferous wood.

Plants and other non-woody materials have been used to produce cellulosic pulp for more than a century. They were actually employed for paper manufacture long before wood. Modern industry, however, developed on wood because this material was available in large quantities from comparatively small areas. Moreover, wood supplies were plentiful and could be obtained cheaply from natural forests. Since most of the World's accessible forests have now been exploited, wood is not so cheap as it used to be. Non-woody materials are, therefore, being used increasingly all over the world to produce paper and pulp in order to meet the increasing demands. Countries short of wood are paying particular attention to their development and are trying to base their industries purely on these materials. Reeds, bamboo, bagasse, esparto, cereals, straw, flax, jute and hemp are being used for

the manufacture of paper, boards etc. in most of the countries of the world.

1.2. Position of raw material for pulp and paper in Pakistan:

The entire need of paper in the country was met by the paper produced in former East Pakistan. After the creation of Bangladesh, most of the paper has to be imported. At that time, there were a few Paper Board Mills in West Pakistan. The latest position of paper and Paper Board Mills in various parts of the country is given in table 1.1. These Mills are mostly using wheat straw, cotton linters, waste paper and bagasse as raw materials for paper production. The Mills are producing different types of paper as given in the Table. The use of bagasse as raw material for paper production is increasing in the recent past and Pakistan Paper Corporation at Charsadda is running exclusively on bagasse. The Government is planning to set up more Paper Mills which will use bagasse as raw material.

Table 1.2 shows a list of Board Mills in the country. These Mills are utilizing the same raw material as used in the Paper Mills. But mostly wheat straw is being used in the Mills for board production. Some of the Mills i.e. Packages limited and Adamjee Paper & Board Mills are manufacturing paper as well as board. Apart from these Mills, there are other small plants producing board of various types.

Pakistan is extremely poor in wood resources and most of the wood is being used in the construction of houses etc. The pulp industry will, therefore, have to depend on non-woody raw materials. The country is, however, fairly rich in non-woody fibrous raw materials like reeds, grasses and agricultural

TABLE NO. 1.1

PAPER MILLS OF PAKISTAN

| S.No. | Name of Industry | Location | Raw material | Products |
|---|---|---|---|---|
| 1. | Packages Limited | Lahore | Wheat Straw, Cotton Linters Waste Paper. | Brown Kraft Printing Paper, Envelope Tissue Paper etc. |
| 2. | Adamjee Paper & Board Mills. | Nowshera | Kahi grass, Cotton linters Bagasse Berweza grass. | Writting and Printing Air Mail Paper. Bond Paper, Cigaritte Paper. |
| 3. | Mandiali Paper Mills. | Sheikhupura | Wheat Straw | Printing Paper, Wrapping Paper. |
| 4. | Allied Paper Industries Ltd. | Gharo | Wheat Straw, Cotton Linters | Printing Paper, Writting Paper, Duplicating Paper. |
| 5. | Dadabhoy Paper Mills. | Karachi | Wheat Straw, Waste Paper. | Kraft Paper, Wrapping paper. |
| 6. | Pakistan Security Printing Corporation. | Karachi | Rags, Cotton Linters | Bank Note, Security Paper. |
| 7. | Pakistan Paper Corporation Ltd. | Charsadda | Sugar Cane bagasse. | Writting Paper, Printing Paper, Duplicating Paper. |

## TABLE NO-1.2

## BOARD MANUFACTURING MILLS OF PAKISTAN

| S.No. | Name of Industry | Location | Raw material | Products |
|---|---|---|---|---|
| 1. | Sethi Straw Board Mills. | Rahwali | Wheat Straw Rice Straw, Waste Paper. | Straw Board, Chip Board, Paper Board Grey Board. |
| 2. | Lasani Straw Board Mills. | Gujran-wala. | Wheat Straw, Waste Paper. | Straw Board. |
| 3. | Mehr Straw Board (Aziz Industrial Corporation). | Gujran-wala. | Wheat Straw | Grey Board. |
| 4. | Ghulam Qadir Straw Board Mills | Gujran-wala. | Wheat Straw | Straw Board. |
| 5. | Dawn Paper & Board Mills. | Hyderabad | Wheat Straw, Waste Paper. | Straw Board, Chip Board, Paper Board. |
| 6. | Central cord board Industries | Karachi | Wheat Straw, Waste Paper. | Card Board. |
| 7. | Packages Limited | Lahore | Wheat Straw, Cotton Linters, Waste Paper. | Duplex, Kraft Lined, Fly Board, Grey Board, Card Board. |
| 8. | Adamjee Paper & Board Mills. | Nowshera | Cotton linters, Wheat Straw, Bagasse, Kahi grass. | Duplex, Grey Board, File Board, Card Board. |

residus. All these materials can be used for the production of paper. Such materials are being utilized for the manufacture of a wide range of pulp and paper products in countries like Netherlands, Italy, France and Germany which are shorter in wood. As to the quality of the products and the economics of production, it has been proved that non-woody material compares favourably with wood.

In Pakistan three raw materials from agricultural residues i.e. sugar cane bagasse, wheat straw and rice straw are being used but are not so abundently available to meet the growing need of the paper industry. We will have to look for other raw materials to meet the future demand of our paper industry. The use of bagasse for paper industry has drawn special attention after the operation of our Eastern Wing. A few paper Mills have been installed which are using bagasse as a raw material and other Mills are being set up. Bagasse is a waste product of sugar industry and unfortunately most of it was burnt by the sugar Mills in their boilers. Sui gas is now being used in these boilers. However, most of the bagasse is utilized as a fuel by the Gur manufacturing units and small sugar units in the country. Paper made from bagasse is of good quality and is also blended with imported good quality pulp for producing quality papers.

Rice grows abundently in Pakistan and in recent years, it is a major exportable commodity. As straw obtained from rice is poor in nutrients, so it is very rarely used as a cattle feed. It is used to some extent as litter in cattle yards and as a packing material for glass-ware but most of it is burnt or just left unused in the fields. It has recently been used for manufac-

2.3. Pulping of Pine needles:

Sulfate process was used for making pulp of the pine needles. As the needles contain oils and resins, it was therefore, appropriate to use the sulfate method.

In the sulfate process, 1500 grams of the needles were taken and fed into the digester. The cooking liquor of total alkali percent of 22,20,18 were introduced into the various cooking trails. Then the digester was switched on and after about 30-37 minutes the temperature reached 105 $^{o}$C. At this temperature, the pressure was released and then it was allowed to continue raising. After about 90-98 minutes, the temperature reached 170 $^{o}$C and at this temperature the pressure was at 9.5 Kg/Cm$^{2}$. This temperature and pressure was kept constant for four hours. At the completion of the cooking time, the pressure was blown out of the digester. The black liquor was collected for alkali recovery and finding the residual alkali, at the base of the digester. The pulp was then taken out into a mesh tray where it was thoroughly washed to remove the alkali and the soluble lignin compounds. After thorough washing, the crude yield was calculated.

The results of various cooks have been recorded in Table 2.1. The sulphidity % of the various cooks was kept at 20. The total alkali concentration has been varied for each cook. The liquor concentration of cook I is 22% which is maximum of all the cooks. The liquor concentration was then gradually lowered for the preceding cooks i.e. for cook II it is 20% and for cook III, 18% respectively.

At the end of table the crude yield of pulp is recorded. It is clear that the higher the liquor concentration,

the lower is the pulp production. In cook I, where 22% alkali has been used, the percent yield is 25.6, whereas in cook II at 20% concentration, the pulp yield is 26% and the yield % for the cook No.III is 28 where 18% total alkali has been used.

## 3. EVALUATION OF PULP

The pulp from the digester is then subjected to the process of screening. After screening the pulp is treated in a valley beater. The pulp is beated for different timings and the freeness value is determined. The processes of screening and beating are discussed in detail below.

### 3.1. Screening:

The pulp from the digester contains incompletely cooked chips togather with other foreign matter that enter the system such as stone, sand etc. To produce a top quality pulp as well as to protect subsequent equipment, such materials must be separated from the pulp. Thus screening has two functions (i) To remove the dirt, non fibrous cells and fibre bundles with a minimum loss of good fibre (ii) To improve fibre dispersion. The first function, dirt removal is generally carried out on screen in the pulp mill. The screens just ahead of the paper machine are used to remove any dirt remaining in the stock and to improve fibre dispersion.

Screening is normally accomplished by using a perforated screen having 0.25mm slit to remove the over-sized material, the pulp being in the form of a dilute solution, so that it will pass through the preparations easily. In processes of screening and cleaning, the consistancy of pulp (percentage by weight of pulp in liquid) is kept on the lower side in order to separate the contamination from the pulp thoroughly.

Kraft pulp does not require as fine screening as do bleached sulfite and sulphate pulps. In some kraft Mills, only high frequency rotary screens are used but in most mills of this type

the pulp is further screened in centrifugal or flat screens. Kraft pulps have traditionally been screened in the pulp mill, but with the trend towards more bleaching and higher grades, more screening is being done in the paper mills. The screening data (Table No.3.1) shows that percentage loss on oven dry pulp is greater in cook No.I,II while that of cook No.III is least (6.8%). However, the rejects (%) is almost negligible and are the same in the three cooks i.e. 0.1 - 0.2%). The brightness of the paper sheet of cook No.I,II is almost the same i.e. 14% while that of cook No.III is less (11.5%).

The Kappa Number of pulp is useful in determining cooking degree, that is, whether the pulp has been cooked to a high degree of removal of non-cellulosic constituents of wood or whether a considerable amount of lignin remains in the pulp. For bleached pulp, it indicates the amount of chlorine that will be required. Thus kappa number is determined before bleaching so that the conditions for bleaching are set up. From table No.3.1 it is also evident that cook No.I will require less chemicals for bleaching as compared to cook No.III. Cook No.II occupies intermediate position.

3.2 Freeness of pulp:

The freeness tester is an instrument which is commonly used to determine the degree of beating. The freeness value is measured by the ease with which water passes through paper making fibres while they are being formed into a wet mat on the perforated plate of the freeness tester. Freeness is, therefore, a measure of the rate of drainage of the pulp. A "free" pulp drains readily, whereas a slow pulp drains its water slowly. There are

TABLE NO-3.1

Screening data of the various Cooks of pine needle pulp.

| Particulars | Unit | Cook No. | | |
|---|---|---|---|---|
| | | I | II | III |
| Crude Yield | % | 25.6 | 26.0 | 28.0 |
| Screened Yield | % | 22.0 | 22.5 | 26.0 |
| Actual loss on O.D. Raw material | % | 3.5 | 3.4 | 1.8 |
| Rejects | % | 0.11 | 0.20 | 0.10 |
| Loss of O.D. pulp | % | 14.0 | 13.2 | 6.8 |
| Brightness | % $G^{o}B$ | 13.5 | 14.0 | 11.5 |
| Kappa No | - | 30.7 | 33.5 | 35.3 |

several different freeness testers commonly used. The TAPPI Official Standard calls for the Canadian Standard Freeness Tester.

The freeness value decreases with increased beating and consequently, the freeness test is used to measure and control the results of the beating process. With most pulp, the freeness decreases in a slightly S-shaped curve as the time of beating is increased. The cereal straw are exceptions, since they show almost a straight line decrease in freeness with beating time. In the case of pine needle pulp (Table 3.2), the freeness decreases with beating time. Freeness at 45 $SR^{o}$ (schopper - Rieglar) or 550 ml (Canadian system) are regarded as standards. At this freeness value, the physical properties of the hand made sheets are taken. For chemical pulp from softwood in the unbeaten state, have a high freeness of 700 ml or more. Hard wood and semi chemical pulp are considerably lower in freeness. Ground wood pulp have a lowest freeness of all being in the range of 50 to 200 ml.

One of the weaknesses of the freeness tester is the difficulty of interpreting and applying the results. A low freeness value may result either from splitting and fraying long fibres which still essentially retain their length or from cutting and formation of much fines and debries. Fibre fines and debries have a major effect on strength properties of papers. Therefore, it must be concluded that the freeness test is only useful for control purposes of the refining of a given type of pulp over a comparatively narrow range of beating or refining condition.

3.3. Beating:

Beating is probably the most fundamental important process in paper making. Paper made from unbeaten stock is low in strength,

fluffy, porous and unfit for most uses, whereas paper made from beater stock is high in strength, dense and hard in texture. Well beaten fibres can be readily formed into a uniform sheet of paper. The principal effects of beating are physical and among the most important are fracture and partial removal of the primary wall of the fibres, decrease in fibre length, increase in fibre flexibility, formation of fibres (fibrillation) and increase the external specific surface of the fibre.

In general, beating improves some properties and has a bad effect on others. Thus the paper maker must select the proper beating conditions to bring out certain properties without detracting too much from other properties. By changing the beating procedure, it is possible to produce papers from the same pulp possessing radically different properties.

In general, increased beating within the commercial range increases beating strength, tensile strength and folding endurance but gradually tends to decrease tearing resistance. Stretch increase by beating to the highest consistancy. Increased beating tends to increase smoothness, hardness and amount of fibre bonding of the fibres, but on the other hand, tend to decrease the opacity and lower the bulk and dimensional stability of the paper. Fries lists two important requirement of an easy beating pulp, it should reach its maximum bursting strength in a short time and it should only show a slight drop in tear and burst after it reaches its maximum.

Beating data of the various cooks of pine needles pulp is given in table 3.2. With beating, the freeness (Canadian System) decreases and the drainage time increases. The most important

TABLE NO-3.2

Beating Data of various cooks of Pine Needle pulp

| Cook No. | Beating Time (Min) | Volume (ml) | Drainage Time (Sec) | Freeness | |
|---|---|---|---|---|---|
| | | | | °(SR) | ml |
| I | 0 | 554 | 6 | 22 | 785 |
| | 10 | 554 | 10 | 34 | 670 |
| | 20 | 626 | 16 | 45 | 550 |
| | 30 | 600 | 24 | 54 | 460 |
| | 40 | 576 | 40 | 61 | 390 |
| | 50 | 600 | 60 | 68 | 320 |
| II | 0 | 576 | 7 | 27 | 730 |
| | 10 | 576 | 13 | 45 | 550 |
| | 20 | 576 | 25 | 56 | 440 |
| | 30 | 576 | 68 | 67 | 330 |
| III | 0 | 533 | 6 | 24 | 760 |
| | 10 | 533 | 10 | 43 | 570 |
| | 20 | 533 | 17 | 56 | 440 |
| | 30 | 533 | 38 | 65 | 350 |
| | 40 | 533 | 78 | 71 | 290 |

factor in the beating data is the beating time in which $^{o}_{SR}$ freeness of 45 Mohlgrade or 550 ml (Canadian System) is obtained. In cook No.I it takes 20 minutes to reach that freeness. Cook No.II,III take approximately the same time i.e. 10 and 11 minutes respectively. At 45 $^{o}_{SR}$ (Mohlgrade) or 550 ml (Canadian System), the properties of handmade sheets are measured and are considered to be standard. It is concluded that cook No.II,III are good as it takes less time to reach freeness of $^{o}_{SR}$ 45 or 550 ml.

A comparison of unscreened, screened and after beating sheets show that unscreened sheet is rough, uneven, very weak, unwanted material and long fibre visible on the sheet, whereas screened sheet is uniform to some extent, smoother and stronger than screened sheet. Sheet after beating is uniform, even and stronger. The properties of the sheet improve remarkably after beating.

## 4. FIBRE DIMENSION

Paper making fibre generally comprises a great variety of size and shape. Not only there is a wide range in fibre dimension among such different plant sources as confi"erous wood, deciduous wood, cotton, flax, hemp, straw etc. but also the fibre of any one plant varies considerably in size and shape. Fibre dimensions are important in paper making because they influence the density, strength, flexibility, smoothness, sizing and dyeing properties of the paper.

### 4.1. Fibre Length:

A long fibre is essential for making strong paper and no paper of high strength is made from short fibre. However, there is a limit to which longer fibre will contribute to paper strength because they result in sheets with "wild" and uneven formation. A poorely formed sheet with thick and then areas will have more strength. The limit for most paper making is in the order of 3 to 6 mm. This value varies considerably due to such factors as ratio of fibre length to diameter, head box consistency and pressure of colloidal agents.

Fibre length is not so important measure of fibre quality as it used to be in the past. In fact fibre length is an exceedingly important index of fibre quality but it is subordinate to the wetting or fibrillating equalities, since adequate sheet strength can be obtained only by means of high degree of fibre to fibre bonding, which prevents the fibre from slippage past one another. For example, North American pulp from spruce and pine usually produces paper with high bursting strength (but lower tear) than pulps from the southern and western areas of the United States,

although the latter has a greater fibre length. It has been shown that the bursting strength of straw pulps which have an average fibre length between 0.30 to 0.46 mm may approach under special conditions the bursting strength of spruce pulps having a comparable average fibre length between 1.2 to 1.8 mm. Furthermore, beating increases the bursting, tensile and folding strength even though it tends to decrease the length of the fibre. Table (4.1) shows the mean fibre length and fibre diameter of pine needle bleached pulp. It is clear that fibre length varies from 0.96 to 1.71 mm. The thickness of the fibre is in the range of 16 to 31 microns. Comparing this with Table No.4.2 where fibre length and fibre diameter of other sources of paper have been given for comparison, it is clear that pine needles pulp has approximately the same fibre length and thickness as other non wood raw material. Its fibre length is comparable with esparto, straw, corn, poplar and reeds.

It is necessary to know that a sheet of paper contains how much short and long fibres. This is best illustrated by the fibre length distribution of pine fibre pulp (Table No.4.3). It is clear from this table that the fibre length ranges from 0.25 mm to 1.82mm. Fifty percent of the fibres are in the range of 0.79 to 1.82 mm. It may be mentioned that bleaching and refining of paper stock result in cutting and bruising of the fibre and in creation of short fragments of "fines". With rare exceptions, therefore, paper consists of fibrous particles of a wide range of dimensions. It is generally accepted that many paper products benefit from being composed of both long and short fibres or fragments. The short fine fibres tend to fill the spaces among

TABLE NO-4.1

Mean fibre Length, Fibre Diameter and ratio of fibre length to diameter of pine needle bleached pulp.

| Sample No. | Fibre Length (mm) | Fibre Diameter (micron) | Ratio of fibre length to diameter |
|---|---|---|---|
| 1 | 1.23 | 20.2 | 61:1 |
| 2 | 1.20 | 20.2 | 59:1 |
| 3 | 1.24 | 20.6 | 60:1 |
| 4 | 1.15 | 19.8 | 58:1 |
| 5 | 1.15 | 18.8 | 61:1 |
| 6 | 1.26 | 20.8 | 60:1 |
| 7 | 1.27 | 20.2 | 63:1 |
| 8 | 1.09 | 16.0 | 68:1 |
| 9 | 1.71 | 21.0 | 81:1 |
| 10 | 0.96 | 16.4 | 58:1 |
| Mean | 1.23 | 19.4 | 63:1 |

TABLE NO-4.2

Comparison of various paper making material for fibre length, fibre diameter and ratio of fibre length to diameter.

| S.No. | Species | Fibre Length (mm) | Fibre Diameter (Micron) | Ratio of Fibre length to diameter |
|---|---|---|---|---|
| 1. | Esparto | 1.5 | 9.0 | 166:1 |
| 2. | Wheat straw | 1.5 | 13.3 | 113:1 |
| 3. | Linen (flax) | 25.0 | 16.0 | 156:1 |
| 4. | Cotton (Staple) | 18.0 | 20.0 | 90:1 |
| 5. | Bamboo | 2.7 | 14.0 | 200:1 |
| 6. | Jute | 2.0 | 20.0 | 100:1 |
| 7. | Ramie | 140.0 | 50.0 | 28:1 |
| 8. | Manila hemp | 7.0 | 18.0 | 39:1 |
| 9. | Bagasse | 1.7 | 20.0 | 85:1 |
| 10. | Corn | 1.5 | 18.0 | 83:1 |
| 11. | Hemp (True) | 20.0 | 22.0 | 91:1 |
| 12. | Coniferous wood | 2.7-3.6 | 32-43 | 75-90:1 |
| 13. | Deciduous wood | 1.0-1.6 | 38-50 | 50:1 |
| 14. | Poplar | 1.1 | 21.0 | 54:1 |
| 15. | Pinus longifolia | 3.9 | 36.3 | 107:1 |
| 16. | Rice straw | 1.5 | 8.5 | 170:1 |
| 17. | Reeds | 1.0-1.8 | 8-20 | 80-90 |
| 18. | Pine needle | 1.2 | 19.4 | 63:1 |

longer and coarser fibres. The resultant sheet shows a favourable combination of strength, uniform surface brought about by short and fine fibres.

One of the disadvantages of short fibre pulp is that it has lower tear resistance. Paper made of short fibre pulp has in general, a lower tear resistance than paper of the same specification made of long fibred pulp.

4.2. Coarseness of pulp fibre:

The coarseness or ratio of fibre length to diameter is an important fundamental property of paper making pulp. The coarseness is more important factor in paper making than fibre length, since it determines the felting characteristics of the fibre. In the past only fibre length was considered important but recently ratio of fibre length to diameter is considered more important than fibre length.

The ratio of fibre length to diameter is given in Table 4.2 for a number of paper making materials. In some raw material i.e. bamboo, coniferous wood, pinus longifolia and linen which have longer fibre length, the ratio of fibre length to diameter is higher. But in other cases i.e. esparto, straw, reeds, bagasse, even with less fibre length, the ratio of fibre length to diameter is higher . In the case of pine needles, this ratio is 63 which is comparable with other paper making materials.

As a general rule, pulp fibres tend to increase in width with increasing fibre length up to a certain point beyond which width remains fairly constant in any further increase in length. Other things remaining unchanged, pulp fibre with a high ratio of length to diameter will contribute more towards the strength

TABLE NO-4.3

Fibre length distribution of pine needle bleached pulp.

| Interval (mm) | No of fibres | Percentage of fibres |
|---|---|---|
| 0.25 - 0.39 | 130 | 20.33 |
| 0.40 - 0.50 | 88 | 13.66 |
| 0.51 - 0.65 | 62 | 9.65 |
| 0.66 - 0.78 | 56 | 8.73 |
| 0.79 - 0.90 | 63 | 9.82 |
| 0.91 - 1.04 | 82 | 12.74 |
| 1.05 - 1.16 | 78 | 12.23 |
| 1.17 - 1.30 | 32 | 4.94 |
| 1.31 - 1.45 | 22 | 3.94 |
| 1.44 - 1.55 | 22 | 3.94 |
| 1.56 - 1.69 | 8 | 0.01 |
| 1.70 - 1.82 | 6 | 0.01 |

ture of board and writing paper. Rice straw possesses very good chemical and physical properties and its chemical pulp is almost equal to coniferous sulphate pulp in strength properties except tearing strength. Its pulp can be used in the manufacture of paper board, wrapping paper, and writing paper, after blending with imported pulp.

Another material available in the country is the waste paper, which is being used widely in the production of paper board and is also used in the manufacture of many kinds of paper. There are many grades of waste paper, ranging from clean trimmings and cutting from converting plants to mixed street collected waste paper. The better grades or carefully sorted grades of waste paper are quite suitable for use in many furnishes and can be substituted for at least a portion of pulp that would have to be imported otherwise. The better grades can also be used for board production in the furnish for top and bottom liners. There are a number of advantages to use the waste paper. The price of waste paper is generally below than that of virgin pulp, whether the virgin pulp is produced locally or imported. In areas where no source of long pulp is available, the use of waste paper may reduce, to some extent, the quantity of long fibred pulp that has to be imported if the clean paper was originally of long fibre pulp.

1.3. Pine Needles:

Pine needles are found abundantly in north west region of Pakistan i.e. Dir, Swat, Kaghan, Hazara, Murree Hills and parts of Baluchistan. The pine needles referred in this report are the matured needles that have fallen on the ground. The needles become dry and are of brownish colour. At present, the needles are not

TABLE NO-1.3

Production of Pine Timber in Pakistan
(1974-75)

| Province/Territory | P.Wallichi (cbm) | P.Roxburgia (cbm) | Total (cbm) |
|---|---|---|---|
| N.W.F.P. | 33,706 | 16,781 | 50,487 |
| Tribal areas | 16,359 | - | 16,359 |
| Punjab | 1,298 | 13,808 | 15,106 |
| Northern areas | 1,982 | - | 1,982 |
| Azad Kashmir | 48,417 | 19,090 | 67,507 |
| Total | 101,762 | 49,679 | 151,441 |

put to any use and in fact the presence of needles in the forests itself is a big problem for the forest department due to the following reasons.

i) They hinder the growth of grasses and check the growth of new plants.

ii) They can easily catch fire. The Forest Department destroys these needles in order to avoid fire hazard.

iii) They check the growth of young plants and thus create hindrance in the expansion of these forests.

Thus, it is clear that the removal of these needles is a great service to Forest Department and it will also help in the improvement of pastures.

The production of pine timber in Pakistan is given in Table 1.3. It gives an idea of the availability of pine needle in various parts of the country. Most of the pine trees are located in Azad Kashmir and North West Frontier Province. Therefore, it is suggested that a Paper Mill utilizing pine needle as raw material should be located near these areas. The suitable location for such a Paper Mill is Mansehra, as it is a central place. Recently, the Government has announced to establish a Paper Mill at Mansehra. The requirements of this Mill can partly be met by utilizing pine needles for the production of pulp and paper.

1.4 Analysis of pine needles:

With a view to utilize pine needle for paper/board, it is essential to determine its chemical composition. Therefore, the chemical constituents such as cellulose, lignin, pentosan, ash content, solvent soluble part and water soluble portion were determined, according to the following procedure:

Cellulose determination:

The cellulose contents of the needles were determined by the known Bevan chlorination method according to which the disintegration of lignin compounds take place and they are, thus rendered soluble in sodium hydroxide and water. During this process other non-cellulosic materials are also dissolved. The sample after chlorination and thorough washing, is treated with sodium sulphite followed by boiling with sodium hydroxide. It is then washed and immersed in .1% potassium permanganate solution and finally treated with oxalic acid.

Lignin determination:

The percentage of lignin of needles was determined according to the method of Ellis and co-workers. This consists of treating the accurately weighed sample first with Alc.Ben.(1:2) and then the sample is treated with water at 100 $^{o}$C for few hours. After drying, the sample is then treated with 72% cold sulphuric acid for two hours. The contents were diluted to 3% and allowed to stand for sometime. It is then filtered and the percentage lignin is calculated.

Pentosan content:

The pentosan contents of the needles were determined by the A.O.A.C method. It consists of converting the pentosans into furfural with strong hot hydrochloric acid. It is then distilled and phloroglucinal solution is added. Greenish black ppt of furfural phloroglucide is formed. After settling, it is filtered and weighed and finally the percentage of pentosan is calculated.

TABLE NO-1.4

Chemical composition of pine needle and comparison with other raw material presently used in Paper / Board Mills.

| Raw material | x-cellulose % | Alcohol Benzene % | Lignin % | Pentosan % | Moisture % | Ash % |
|---|---|---|---|---|---|---|
| Wheat straw | 43.2 | - | 16.9 | 19.2 | 10.0 | 7.5 |
| Rice straw | 48.8 | 7.2 | 13.0 | 20.0 | 7.8 | 9.5 |
| Sugar cane bagasse | 35.1 | 0.9 | 21.0 | 23.1 | 6.8 | 1.7 |
| Kahi | 35.4 | 8.9 | 17.0 | 24.4 | 14.2 | 2.5 |
| Pine needles | 31.5 | 11.5 | 25.6 | 10.2 | 10.3 | 3.3 |

Ash content:

One gram sample after initial ignition was heated at temperature between 600-700 °C for 3-4 hours. The residue so resulted was taken as a measure of ash content.

Alco-Ben soluble portion:

Accurately weighed sample of pine needles was extracted in a soxhlet apparatus for 3-4 hours. The ratio of alcohol to benzene was kept at 1:2.

Moisture contents:

Moisture contents were determined by placing the sample in an oven at 100-105 °C till constant weight is obtained.

The results of chemical analysis of pine needles are recorded in table No. 1.4 . The chemical constituents are compared with the raw materials which are commonly used for the manufacture of paper and paper board in various mills of the country.

From the table it is evident that x-cellulose content of the pine needles is generally of the order of those of sugar cane, bagasse and kahi. The lignin contents of the needles are higher than those of the non-wood materials shown in the table. The lignin contents corresponds to the softwood species. Comparing the pentosan contents of the needles with those of the other raw materials, it is obvious that it is much lower than the other raw materials. Actually, it is in the range of wood species. The ash contents of needles are almost similar to the kahi grass. This percentage is sufficiently higher than most of the species of hard and soft woods. Much higher ash percentage affects physical properties of pulp and its bleaching and cooking requirements.

The moisture percentage of the needles is within the reasonable limits, therefore, there are remote chances of the deterioration of pine needles on storage.

## 2. PROCESSES FOR PULP PRODUCTION

### 2.1. Brief discription of cooking processes:

Pulping is the starting chemical operation in the paper manufacturing. In this process the wood chips are converted into seperate fibres, by the chemical reaction between lignin and the active chemicals in the cooking liquor such as sodium hydroxide, sodium sulphide and sodium sulphite. The binding materials of fibres are mostly the carbohydrates and lignin. Besides, soluble mineral salts, resins, fats and tannins are also present in the wood chips. The presence of these materials, in various species, of wood or other agricultural products varies considerably. For example, the percentage lignin in hardwood varies between 17-24% whereas the soft wood contains 24-30%. The structure of lignin of hardwood species are also somewhat different than the lignin of softwood. In view of this difference in the structure, the kraft pulp require longer periods for lignin dissolution than sulfite pulp of comparable lignin content. Furthermore, kraft chlorolignin is more difficult to remove from the pulp by alkaline extraction than sulfite lignin. The reason for these differences generally is ascribed to the belief, that as compared to sulphite lignin, kraft lignin is more condensed i.e. it comprised of larger molecule aggregates and hence, requires a longer period to effect the size reduction necessary for water and alkali solubility. The processes which are used for pulp making are as follows:

i) Acid sulfite:

In the acid sulfite process, the primary reaction between the bisulfite ions and the lignin takes place to form

lignin sulphonic acids, and also in the presence of a base, the lignin sulphonic salts of that base. The rate of the reaction depends upon the temperature and sulfite ions concentration. The acidity (pH) is an aggressive factor causing deeper penetration into the lignin portion. The pH varies between 2 to 3 during the sulfite cooking and the temperature ranges between 135°C to 165°C.

The acid sulfite method of pulp is not suitable for all species of wood, in particular, the pinus and many hard woods. The presence of phenolic compounds in pinus prevents the dissolution of lignin as under acidic conditions and higher temperature resin like polymers are formed. Likewise, all woods containing large amounts of natural resinous and fatty acids are not suitable for pulp by the acid sulfite method.

ii) Neutral Sulfite Processes:

As evident from its name, this process of pulping is carried out at the neutral zone, i.e. between the pH range of 7 to 9. This process is not fully a chemical process but semichemical one. The chip, after the sulfite treatment, softens and the pulp is made in the following mechanical operation. This process is more suitable for agricultrual wastes and grasses because they contain less lignin than the wood species. As such, they require lesser quantity of digestive chemicals. The process is also applicable for other wood species mostly for converting of hard wood into pulp. In this process the chips are treated under high temperature and pressure. Because of the near neutral conditions in pulping, the temperature is relatively high to bring about a reasonably rapid reaction rate, being in the range of 160 °C to 185 °C. During the specified pulping time, most of the wood substance (lignin and carbohydrate) are dissolved. The time of

pulping depends on the concentration of chemicals.

The pulp yield of the neutral sulfite method is much higher (more than 60%) than the acid sulfite method which is 40 to 50%. The chip size in this method is less than the size of chips used in other pulping methods. The consumption of chemicals is relatively less in this process.

iii) Soda Process:

In this process, sodium hydroxide is used for pulping. In fact it is an old method. Since the discovery of sulfate process, most of the mills using soda process have now changed to the use of sulfate process, as the soda process is much expensive.

In cases where more brightness is desired, the soda process can be used as the pulp obtained from soda process can be easily bleached to more brightness than the pulp of the sulfate process. In this process higher chemical concentration is required.

2.2. Sulfate Process:

This is also called the kraft process. Kraft is a German word, the meaning of which is "Strong". The sulfate process is in fact a modification of the soda process. With a view to reduce the cost of chemicals, this process has been developed. The pulp obtained from this process is not only cheaper but also stronger. The process has some of the advantages over the other chemical processes used for pulp making, which are given below.

1. The pulp obtained has higher strength.
2. It is used for making pulp of great variety of wood species.
3. There is choice of using inexpensive chemicals in different combination for various raw materials.
4. There is multichoice of bleaching processes for type of paper product desired.

5. After cooking, the excess chemical in the liquor can efficiently be recovered.

6. Wood species containing resinous matters can be cooked without much hindrence.

7. Various grades of pulp can be obtained for use in different grade of paper or board.

This process has also some disadvantages such as:

i) The pulp obtained from the process is darker in colour.

ii) Cost of bleaching is comparatively high when white pulp is required.

iii) The yield percent is low in comparison to acid sulfite process.

The sulfate pulping process uses an alkaline solution of caustic soda (sodium hydroxide) and sodium sulfide, termed "white liquor," to remove the lignin binding the cellulose fibres together in the wood. The cooking liquor also converts the fats, oils and resins present in the wood to soaps which are soluble in the liquor. This is one of the principal advantages of the sulfate over the sulfite process.

The chief ingredients of the cooking liquor are: Sodium hydroxide, Sodium Sulfide, Sodium Carbonate, Sodium Sulfate and Sodium Thiosulfate. The last three chemicals are not active in the cooking process.

Active alkali present in the cooking liquor varies considerably from mill to mill depending upon the pulp being made. Usually, 18 to 20% of the alkali based on oven dry weight is used. The rate of pulping is highly temperature dependent. The temperature is kept at 170 $^{o}$C to 175 $^{o}$C and the pressure is usually between 8 to 10 Kg/ $Cm^{2}$.

TABLE NO-2.1

Making of Pulp of Pine needles by sulphate process under varying concentration of cooking liquor.

| Cooking condition | Units | Cook Number | | |
|---|---|---|---|---|
| | | I | II | III |
| Weight of sample taken | g | 1200 | 1500 | 1500 |
| Sulphidity | % | 20 | 20 | 20 |
| Total Alkali as $Na_2O$ | % | 22 | 20 | 18 |
| NaOH as $Na_2O$ | % | 17.6 | 16.0 | 14.4 |
| $Na_2S$ as $Na_2O$ | % | 4.4 | 4.0 | 3.5 |
| Liquor Ratio | Ratio | 1:6 | 1:6 | 1:6 |
| Time to reach 105° Temp. (release pressure) | Min. | 30 | 37 | 32 |
| Time to reach Maximum (temperature 170°C) | Min. | 90 | 99 | 99 |
| Time at Maximum Temp. | Hrs. | 4 | 4 | 4 |
| Pressure | $Kg/Cm^2$ | 9.5 | 9.5 | 9.5 |
| Temperature | °C | 170 | 170 | 170 |
| Crude yield | % | 25.6 | 26.0 | 28.0 |
| $Na_2$ consumed on $Na_2S$ addition | % | 100 | 100 | 100 |
| NaOH consumed on NaOH addition | % | 95 | 96.2 | 96.3 |

factor than broader fibres of similar length. For example, bagasse (depithed) which are fairly short but have a high ratio of length to diameter make good paper making material except for tearing strength.

4.3. Blending of Fibre Length:

In Pakistan, usually bagasse and other raw materials are being used for paper manufacture. These raw material have a lower fibre length. In order to get a strong paper, pulp of longer fibre length is imported and mixed with locally available short fibred pulp, with the result that the resulting paper is strong.

Experience of making special kind of paper shows that better results can be obtained by using more than one pulp in a finish than by using one pulp alone. Each fibre of the finish will contribute a characteristic property associated with it which could not be easily obtained from the other fibre.

Lathrop has indicated that long fibred pulp produced from fibres like sisal, abaca, ramie when added to the short fibre bagasse pulp will produce paper of very high tear resistance. Pulp from other long vegetable fibres like sunn fibre, true hemp, linseed or oil flax fibres of the agave species, rag, thread waste, cotton waste, bamboo pulp and soft wood chemical pulps can also be used to improve the quality of the paper.

## 5. PHYSICAL PROPERTIES OF HAND MADE SHEETS

Handmade sheets were made on the sheet machine, pressed and dried under controlled conditions i.e. 70 $F^{o}$ and 50% relative humidity. To secure the comparable data, the test sheets of paper must always be formed in the same way and under the same conditions, so that differences in the paper will be caused only by differences in the qualities of the pulp. The purpose of hand sheet testing is to derive information about the pulp as a paper making material. Ideally, test data derived on standard handsheets should provide some means of controlling or modifying the properties of paper by suggesting adjustments to the pulp treatments or fibre furnish. The sheets were tested for various physical properties such as basis weight, thickness, bulk, bursting strength, burst factor, tearing strength, tear factor, tensile strength, breaking length and folding endurance.

### 5.1. Dimension of Paper:

Since the basic dimensions of paper i.e. weight or substance, thickness, and bulk affect nearly all strength determination of paper, they must be determined in the first instance.

#### i) Grammage or basis weight:

The weight or grammage per unit area is obviously fundamental in all paper products. Paper is usually measured in ream which comprises 480 sheets, but now also used to signify the substance of a lot of paper. The paper weight shall be expressed in at least two of the following ways (a) The equivalent basis weight in pounds for a ream consisting of 500 sheets. (b) The weight in grams per square metre. The weight of paper affects all its physical properties and the ratio of strength to weight varies

with the weight. In general, highest strength in relation to weight is obtained on paper higher than 35 lbs /ream. The basis weight ($g/mm^2$) of the three cooks No.I,II,III at 45 $^{o}$SR is 65.1, 58.7 and 61.0 g/mm respectively.

ii) Thickness or Caliper:

Like weight, thickness also affects all physical properties and is measured in a micrometer as the distance between two circular plane surfaces under a pressure of 8-9 P.S.I. The thickness of sheets of cook No.I,II,III at 45 $^{o}$SR is 109, 102, 159 microns respectively.

iii) Bulk:

Bulk is the reciprocal of density of paper and is expressed as

$$\text{Bulk} = \frac{\text{Average thickness of paper in micron}}{\text{Substance in gms. per square meter}}$$

The bulk of sheet of cook No.I,II (Tables 5.1, 5.2) is the same (1.7) while that of cook No.III is highest (2.5).

5.2 Tensile Strength:

Tensile strength of paper is determined from the load required to pull apart a strip of paper of known dimensions. The tensile strength of paper is determined by the combined effects of the following factors.

a) The strength of the individual fibres of the stock furnish.

b) The average length of the fibre.

c) The inherent bonding ability of the fibre surface both in terms of bonded area and strength per unit of bonded area.

d) A factor determined by the formation or fibre orientation within the sheet.

The great importance of the interfibre bonding in paper has led to the belief that this is the predominant factor in tensile

strength, fibre strength playing a secondary role. The inherent bonding ability of the fibres of a paper stock is of outstanding importance in paper strength. It is well recognised that some pulps have a strong tendency to form interfibre bonds while others have only a slight tendency to do so. The beating and refining action has the effect of swelling the fibre by increasing the imbibition of water, of rupturing and fibrillating the fibre surface and of rendering the fibre more flexible and better able to mat and contact neighbouring fibres. All these effects enhance the interfibre bonding ability of the stock. Pressure exerted upon the wet sheet is also an important factor in promoting bonding.

The tensile strength of pine pulp is increasing with beating time for all the three cooks (Tables 5.1, 5.2, 5.3). The tensile strength for cook No.I,II, is the same (3.1 Kg/mm) at $^{o}$SR 45. However, the tensile strength for cook No.III is less i.e. 2.7 Kg/mm.

Breaking length is calculated from tensile strength. Breaking length is the length of a strip of paper which if hanging from one end will just support its own weight.

$$\text{Breaking Length} = \frac{1,00,000 \times \text{Tensile strength (Kg) percent width}}{\text{Substance in gm per square metre.}}$$

For pine needle pulp, the breaking length increases with beating time. The breaking length of cook No.II at 45 $^{o}$SR is highest (3543 metre). Therefore, it is concluded that as regard breaking length, cook No.II is the best.

5.3 Tearing Strength:

Tearing strength is one of the most important properties of the paper. The force required to tear a sheet of paper is an

TABLE NO-5.1

Physical properties of the standard Hand Made Sheets of unbleached pine needle pulp ( Cook No.I ).

| Particulars | Units | | | | | | |
|---|---|---|---|---|---|---|---|
| Beating Time | min | 0 | 10 | 20 | 30 | 40 | 50 |
| Freeness | $^{o}$SR | 22 | 34 | 45 | 54 | 61 | 68 |
| Freeness (Canadian). | ml | 780 | 660 | 550 | 460 | 410 | 280 |
| Basic weight | $g/m^2$ | 63.0 | 60.7 | 65.1 | 63.4 | 60.9 | 62.0 |
| Thickness | micron | 144 | 114 | 109 | 99 | 83 | 81 |
| Bulk | - | 2.2 | 1.8 | 1.6 | 1.5 | 1.3 | 1.3 |
| Bursting strength | lbs/ sq.inch | 11.5 | 14.4 | 20.4 | 21.3 | 21.4 | 22.6 |
| Bursting strength | $Kg/cm^2$ | 0.802 | 1.008 | 1.425 | 1.492 | 1.498 | 1.583 |
| Burst factor | - | 12.7 | 16.6 | 21.8 | 22.1 | 24.5 | 25.5 |
| Tearing strength | g | 30 | 29 | 27 | 23 | 21 | 19 |
| Tear factor | - | 47.0 | 47.0 | 41.0 | 36.0 | 34.0 | 30.0 |
| Tensile strength | Kg/15 mm | 2.43 | 2.80 | 3.19 | 3.44 | 3.74 | 3.88 |
| Breaking Length | metre | 2570 | 3075 | 3266 | 3579 | 4094 | 4172 |
| Folding Endurance | D/F | 5 | 8 | 8 | 10 | 14 | 15 |

indication of its performance in use. Heavier papers will have more tearing resistance than light paper. It is often important to determine the inherent tearing quality of fibre independent of the factor of sheet weight. For this tear factor is used.

$$\text{Tear Factor} = \frac{100 \times \text{tearing strength (g)}}{\text{Basis weight}}$$

The most important contribution to tearing strength appears to be the effect of fibre length and interfibre bonding. The tearing strength of pulp fractions, of blend of long and short fibres and of pulps which have been subjected to severe cutting all demonstrate that any reduction in fibre length will cause a significent reduction in tearing strength. This is because the longer fibres tend to distribute the stress over a greater area, over more fibres and more bonds, while short fibres allow the stress to be concentrated in a smaller area.

The tearing strength and tear factor of pine needle pulp is decreasing with beating time. (Tables 5.1, 5.2, 5.3) for all cooks. The tear factor of cook No.I at zero beating time is 47 and is decreasing with beating time. However, at $\overset{o}{SR}$ 45 its value is 41. In case of cook No.II, the tearing factor is constant i.e. 30 up to beating time of 20 minutes (Table 5.2). In cook No.III initially (at zero beating), the tear factor is the same as in cook No.I but at $\overset{o}{SR}$ 45, it is the highest i.e. 46. It is concluded that as far as tear factor is concerned, it is highest in cook No.III.

5.4 Bursting Strength:

One of the oldest and most widely used of the strength tests for paper and paper board is the bursting test. Bursting strength according to TAPPI is the hydrostatic pressure required

TABLE NO. 5.2

Physical properties of standard Hand Made Sheets of unbleached pine needle pulp (Cook No.II).

| Particulars | Units | | | | |
|---|---|---|---|---|---|
| Beating Time | Min | 0 | 10 | 20 | 30 |
| Freeness | $^{o}$SR | 27 | 45 | 56 | 67 |
| Freeness(Canadian) | ml | 670 | 550 | 560 | 330 |
| Basis weight | $g/m^2$ | 58.7 | 58.7 | 58.3 | 57.7 |
| Thickness | micron | 123 | 102 | 92 | 83 |
| Bulk | - | 2.0 | 1.7 | 1.5 | 1.4 |
| Bursting strength | lbs/sq.inch | 12.0 | 16.6 | 17.4 | 18.4 |
| Bursting strength | $Kg/cm^2$ | 0.840 | 1.162 | 1.218 | 1.284 |
| Burst factor | - | 14.3 | 19.7 | 20.5 | 22.2 |
| Tearing strength | g | 23 | 20 | 19 | 16 |
| Tear factor | - | 30 | 30 | 30 | 20 |
| Tensile strength | Kg/15mm | 2.71 | 3.12 | 3.48 | 3.72 |
| Breaking Length | metre | 3043 | 3543 | 3979 | 4298 |
| Folding Endurance | D/F | 3 | 5 | 8 | 8 |

to rupture paper when deformed in an approximate sphere of 1.20 inches in diameter at a controlled rate of loading. The primary function of the bursting test is to indicate the resistance of a paper product to rupture in use. It is quick and easy and one test is sufficient for both directions of machine made paper. For this reason the bursting test has found almost universal use throughout the paper industry.

Burst factor is often used for comparing two papers with regard to their bursting strength and is determined as follows.

$$\text{Burst factor} = \frac{1000 \times \text{Bursting strength } (\text{Kg/cm}^2)}{\text{Basis weight}}$$

The bursting strength and the bust factor are increasing with beating time in all the three cooks of pine needle pulp (Tables 5.1, 5.2, 5.3). Initially at zero beating time, the burst factor of cook No.I is 21.8 and that of cook No.III is 17.0. The burst factor of cook No.II at 45 $^{o}$SR is 19.7. Thus cook No.II occupies intermediate position as regards bursting strength.

5.5. Folding Endurance:

Folding endurance is an important test relating to the durability of paper. This test is a performance test and gives the best idea of serviceability of paper. Folding endurance measures the number of times a strip of paper can be bent around a very small cylinderical surface and back again in the opposite direction under standardized tension till it finally breaks at the crease point.

The great limitation of the folding endurance determination is the fact that each test covers a very small area and slight imperfection in this small area gives entirely a wrong indication. A large number of tests should be made in each direction and the

TABLE NO.5.3

Physical properties of standard Hand Made Sheets of unbleached pine needle pulp (Cook No.III).

| Particulars | Units | | | | | |
|---|---|---|---|---|---|---|
| Beating time | Min | 0 | 10 | 40 | 30 | 40 |
| Freeness | $^{o}SR$ | 24 | 43 | 56 | 65 | 71 |
| Freeness (Canadian) | ml. | 760 | 570 | 440 | 350 | 290 |
| Basis weight | $g/m^2$ | 60.0 | 61.0 | 59.5 | 59.5 | 59.5 |
| Thickness | micron | 194 | 159 | 133 | 118 | 111 |
| Bulk | - | 3.2 | 2.6 | 2.2 | 1.9 | 1.8 |
| Bursting strength | lbs/sq.inch | 10.6 | 14.5 | 16.6 | 18.5 | 21.8 |
| Bursting strength | $Kg/cm^2$ | 0.742 | 1.015 | 1.142 | 1.295 | 1.526 |
| Burst factor | - | 12.3 | 16.6 | 19.2 | 21.7 | 25.6 |
| Tearing strength | g | 29 | 29 | 27 | 26 | 23 |
| Tear factor | - | 48 | 47 | 43 | 43 | 38 |
| Tensile strength | Kg/15mm | 1.74 | 2.73 | 2.99 | 2.06 | 3.48 |
| Breaking Length | metre | 1933 | 2983 | 3350 | 3428 | 3899 |
| Folding Endurance | D/F | 2 | 2 | 4 | 4 | 7 |

TABLE NO. 5.4

Strength properties interpolated values at 45 SR (Degree of freeness).

| Particulars | Units | Cook Numbers | | |
|---|---|---|---|---|
| | | I | II | III |
| Total Alkali as $Na_2O$ | % | 22 | 20 | 18 |
| Beating time | Min | 20 | 10 | 11 |
| Drainage time | Sec. | 16 | 13 | 11 |
| Bulk | - | 1.6 | 1.7 | 2.5 |
| Burst factor | - | 21.8 | 19.7 | 17.0 |
| Tear factor | - | 41 | 30 | 46 |
| Tensile strength | Kg/15mm | 3.19 | 3.12 | 2.73 |
| Breaking length | metre | 3266 | 3543 | 3000 |
| Folding Endurance | D/F | 8 | 5 | 2 |

average taken.

In the case of pine needle pulp, folding endurance increases with beating time of all the three cooks. At 45 $^{o}$SR , the folding endurance of cook No.I is highest i.e. 8 and lowest (2) in cook No.II, cook No.III has a folding endurance of 5. It is concluded that the folding endurance of the three cooks is fairly low.

5.6. Comparison of the physical properties:

It is important to know the properties of other raw materials being used in Pakistan for the production of paper. It is also of interest to know the work already done on some of the possible future raw materials produced in the country. We will first discuss the later portion.

Table 5.4 gives the interpolated values at 45 $^{o}$SR freeness of the three cooks. Comparing the physical properties of some of the raw materials not used in the country with that of pine needle (Table 5.5), it is evident that the properties of Euclyptus, banana and coconut are superior to that of pine needle.

Table 5.6 shows a comparison of the pine needle handmade sheet at 45 $^{o}$SR freeness of the already used raw material found in the country. The various raw materials that are being used are lickerin (cotton waste), barweza grass, Kahi grass, rice straw, wheat straw and bagasse. The physical properties of lickerin, barweza grass, Kahi grass and wheat straw are superior to those of pine needle sheets. However, the properties of rice straw are comparable with those of pine needles. The tear factor of rice straw is less than that of pine needle. The burst factor is almost equal to that of pine needle. Breaking length was greater in the case of rice straw and the folding endurance of rice straw was

TABLE NO.5.5

Physical properties of various raw material not used in the country.

| Particulars | Banana | Bucalyptus camaldulensis | Coconut | Pine needle Cook No.I | Pine needle Cook No.II. |
|---|---|---|---|---|---|
| Na OH% | - | 18.0 | 15 | 22.0 | 20.0 |
| $Na_2 SO_3$ 3% | - | - | 5 | - | - |
| Cooking time(hours) | - | - | 1½ | 4 | 4 |
| Cooking Temp. $C^o$ | - | - | 170 | 170 | 170 |
| Beating time(min) | - | 47 | - | 20 | 10 |
| Dranage time (Sec.) | - | 17 | - | 16 | 13 |
| Bulk | 2.1 | 1.2 | - | 1.6 | 1.7 |
| Burst factor | 29.0 | 53.6 | 16.9 | 21.8 | 19.7 |
| Tear factor | 61.6 | 48.0 | 80.5 | 41.0 | 30.0 |
| Breaking length metre | 4980 | 8600 | 3360 | 3266 | 3545 |
| Folding Endurance | - | 275 | 12 | 8 | 5 |

TABLE NO.5.6

Physical properties of the various raw material used in paper Mills.

| Particulars | Lickerin | Barweza | Bagasse Depith | Bagasse Raw | Kahioat 39 SR | Rice straw | Wheat straw at 16 SR |
|---|---|---|---|---|---|---|---|
| Na OH% | 10.0 | 3.2 | 3.5 | 3.5 | - | 5.0 | $Na_2 Co_3$ 3% |
| $Na_2 So_3$% | - | 12.5 | 14.0 | 14.0 | - | 12.5 | 10.0 |
| Cooking time(hours) | 6 | 3.5 | 3.5 | 3.5 | - | - | 3.5 |
| Cooking Temp C° | 170 | 170 | 170 | 170 | - | - | - |
| Beating time (min.) | 50 | 31 | 17 | 21 | 50 | 30 | - |
| Drainage time (Sec.) | 10 | 31 | 12 | 11 | 12 | 25 | - |
| Bulk | 1.85 | 1.75 | 1.85 | 1.8 | 1.4 | 1.8 | 2.3 |
| Burst factor | 36.0 | 46.0 | 15.4 | 11.9 | 30.6 | 24.2 | 31.2 |
| Tear factor | 250 | 74.0 | 31 | 33 | 34.5 | 27.0 | 41.9 |
| Braking length (metre) | 4900 | - | 2890 | 2000 | 4934 | 4620 | 6100 |
| Folding Endurance (D/F | 210 | 68 | 2 | 2 | 8 | 3 | - |

less than that of pine needle. It is concluded that the properties of pine needle hand made sheets are comparable with those of rice straw.

Comparing the properties with bagasse (depith) and as a whole, it is evident that pine needle handmade sheets are superior to those made from bagasse. The tear factor of pine needle (cook No.I) is superior, while that of Cook No.II is the same as that of bagasse. The burst factor of pine needle is far greater than that of bagasse. Similarly the breaking length of pine needle sheet is greater than that of bagasse. The folding endurance of bagasse is low as compared to that of pine needle. This shows that the properties of sheets of pine needle are superior to those of bagasse.

The crude yield of bagasse depith is 34 to 37% while that of bagasse as a whole is 43 to 48%. It shows that little difference occurs in the yield of pine needle which is 35.6%. Owing to the good properties of pine needle sheets and low cost of raw material, the low yield of pine needle can be supplemented.

From the above comparison, it is concluded that to improve further the properties of pine needles handmade sheet and to utilize pine needle for writing and printing purposes, it will have to be blended with long fibred wood pulp.

## 6. BLEACHING

Bleaching is an important step in the pulp and paper manufacturing. This follows after the cooking process i.e. the pulp obtained from cooking and its subsequent screening and beating is then subjected to the process of bleaching. Actually bleaching is a continuation of the fibre purification beginning from the pulping process. During the cooking, the adhering hemicelluloses are dissolved to a considerable extent because of their low resistance to hydrolyses. An appreciable portion of the lignin is also dissolved during cooking. The residual lignin is modified slightly, and is largly removed by the bleaching process.

The main object of the bleaching process is to remove residual lignin material and the colouring material in the fibres and to obtain white pulp having satisfactory physical and chemical properties. In soda and sulfate pulps, the lignin and hemicellulose residues are modified to a greater extent. These pulps are darker and, therefore, require more bleaching to produce the desired brightness. The water used in the bleaching operation, must be free of hardness, organic extracts, becteria and undissolved solvents as these impurities adversly affect the bleaching and colour of pulp.

The careful selection of the type of bleaching system and the operating conditions, such as time, temperature, pulp consistancy, concentration of chemical, pH etc. are essential for obtaining the desired properties for the bleached pulp.

Chlorine and its compounds such as hypochlorous acid sodium hypochlorite, calcium hypochlorite, chlorine dioxide and sodium chlorite containing "available chlorine" are commonly used

for bleaching wood pulp fibres, chiefly because of their low cost. The "available chlorine" content of such a bleaching agent is a measure of its oxidizing capacity to react with the residual lignin and colouring materials in the unbleached fibre. Bleaching of pulp is usually made by either Single-stage Hypochlorite Bleaching or Multistage Bleaching.

6.1. Single-stage Hypochlorite Bleaching:

Hypochlorites react readily with unbleached pulp to whiten or bleach them. The simplest bleaching process comprises a single stage operation, using either calcium or sodium hypochlorite on a chemical pulp, sulfite, soda, or sulfate. A great number of pulps, especially those produced by the sulfite or soda process, can be bleached to a brightness of 80% with a single stage hypochlorite treatment but with serious loss of fibre strength. Single hypochlorite stage is usually not applicable to sulphate pulps unless a low brightness is desired.

The pH is maintained between 9-11. This is maintained by using buffered hypochlorite containing an excess of alkali to neutralize the acidic substances formed during the operation. At a pH range of 8-6 undesirable reactions, resulting in the weakening of fibres, occur. This may be caused due to the great activity of hypochlorous acid in this pH range. The consistancy varies between 4% and 15% and the temperature is kept between 30 $C^{o}$ to 38 $^{o}C$. Higher temperature is avoided as oxidation of cellulose may occur which will result in the loss of fibre strength.

6.2. Multistage Bleaching:

1. Chlorination stage:

In the 1st stage, the pulp after screening and beating,

is treated with chlorine water. The concentration of chlorine is usually kept at 7% of the weight of pulp. Higher percentage of chlorine can be used keping in view the nature of pulp being used. The reaction proceeds rapidly and requires ½ to 1 hour for completion. The pH varies from 2-3 while the temperature is maintained at about 20 °C. The consistancy at this stage is kept at 3 to 4%. The control of pH is essential in this stage as chlorine solution varies in composition under different pH (degree of acidity or alkalinity). At the pH range of 2-3 it is present in the form of elemental chlorine and hypochorous acid. With the rising pH it changes to more hypochlorous acid and hypochlorite. Thus, the pH of the system is an important factor because it controls the proportion of chlorine, hypochlorous acid and hypochlorite ions in the bleaching solution.

2. Alkali Extraction Stage:

The soluble chlorinated compounds formed in the chlorination stage are removed by washing the pulp. Then the pulp is subjected to hot sodium hydroxide extraction. The concentration is maintained at 1 to 2% or more and the temperature is kept between 60 °C to 70 °C. The reaction time is usually from 1 to 1½ hour and the pulp consistancy is kept at 10 to 16%.

In this stage, most of the constituents of fibres which are alkali soluble are removed. The lignin removal depends on the chemical nature of the lignin, the physical nature of the chlorinated lignin and the chemico-physical nature of the fibres. The extraction conditions such as pH, temperature, time and concentration play vital role in the alkaline extraction.

3. Hypochlorite Bleaching:

The final brightness or whitening of pulp takes place in the hypochlorite stage. In the 1st and 2nd stages actually the fibres purification takes place but no bleaching. To minimize chemical and physical degradation of the fibres, the pH range of this stage should be preferably between 9 and 11. Pulp strength decreases as the pH drops below 9. The stability of the colour of pulp is also affected below this pH. The initial pH is by far the most important variable in the hypochlorite bleaching stage. Other variables fall in the following order, temperature, percentage of available chlorine and time. The pulp consistancy is kept at 5% whereas, the temperature varies between 40-45 °C and the time period is two hours. The hypochlorite concentration can be altered for obtaining the desired whitening of the pulp.

6.3. Bleaching of pine needles pulp:

The bleaching of pulp, obtained from pine needles, was carried out in the multistage bleaching operations. As the pulp obtained by sulfate process is darker, so they need thorough bleaching.

The results of bleaching of pine needles pulp are recorded in Tables 6.1, 6.2. In table 6.1 one sample each of cook I, II and III has been bleached. The bleaching conditions are similar for all the three samples in all the bleaching stages but the concentration of chemicals (Total alkali) varies in the three samples. It is evident from the results that, actually, brightness results in the hypochlorite stage bleaching i.e. in the 1st sample the brightness in the 1st stage is 24%, in the 2nd stage it is 28% while final brightness of 61% is obtained in the last stage of bleaching. Similar is the case with the samples of II and III cooks. The

TABLE NO.6.1

Multi-stage bleaching of pulp of Pine needles obtained from different cooking.

| Particulars | Units | Cook No. | | |
|---|---|---|---|---|
| | | I | II | III |
| Sulfidity | % | 20 | 20 | 20 |
| Total Alkali as $Na_2O$ | % | 22 | 20 | 18 |
| **Chlorination 1st stage** | | | | |
| $Cl_2$ added on O.D. Pulp | % | 7 | 7 | 7 |
| Consistancy | % | 3.0 | 3.0 | 3.0 |
| pH | | (pH was not maintained during chlorination due to short time). | | |
| Bleaching temp. | °C | 25-30 | 25-30 | 25-30 |
| Bleaching time | Hrs. | 1.0 | 1.0 | 1.0 |
| Residual $Cl_2$ | % | - | - | - |
| Losses during chlorination | % | 3.5 | 4.3 | 4.5 |
| Brightness | GB° | 24.0 | 19.0 | 13.5 |
| **Extraction 2nd stage** | | | | |
| NaOH added on OD Pulp | % | 2.0 | 2.0 | 2.0 |
| Consistancy | % | 10.0 | 10.0 | 10.0 |
| pH | - | Over 10 | Over 10 | Over 10 |
| Temperature | °C | 60-70 | 60-70 | 60-70 |
| Time | Hrs. | 1.0 | 1.0 | 1.0 |
| Losses | % | 2.6 | 5.5 | 6.2 |
| Residual NaOH | % | - | - | - |
| Brightness | GB° | 28 | 22.0 | 14.5 |
| **Ca-hypochlorite 3rd stage** | | | | |
| Hypochlorite added on OD Pulp | % | 3.0 | 3.0 | 3.0 |
| Consistancy | % | 5 | 5 | 5 |
| pH | - | 7.6 | 7.6 | 7.6 |
| Temperature | °C | 40-45 | 40-45 | 40-45 |
| Time | Hrs. | 2.0 | 2.0 | 2.0 |
| Losses | % | 2.0 | 3.2 | 3.1 |
| Total Losses | % | 8.6 | 13.0 | 13.8 |
| Bleached yield of pulp | % | 91.4 | 87.0 | 86.2 |
| Bleached yield on R.M. | % | 18.5 | 19.4 | 22.4 |
| Brightness | G°B | 61.0 | 49.0 | 21.0 |

results of the table also reveal, that maximum of brightness has been attained in the case of I cook i.e. at the total alkali concentration of 23%, the brightness is 61%. As the total alkali concentration is lowered i.e. 20% and 18% in the II and III cooks respectively, the corresponding brightness of 49% and 21% was obtained. It is, therefore, evident that pulp obtained at higher chemical concentration are liable for more brightness than the pulp produced at comparatively low liquor concentration under similar bleaching conditions.

It is further evident that the higher the brightness, the lower is the bleached yield on raw material, or in other words the higher the chemical concentration, the lower is the pulp yield on the raw material. In cook III, the pulp yield has improved considerably but its brightness has become much lowered. The results of the table indicate that pulp of cook III has not been completely purified i.e. greater portion of lignin remained undissolved. The residual non-cellulosic portion in the pulp of cook I has been less whereas, the pulp of cook II has contained slightly more residual lignin than cook No.I. The bleaching conditions shown under Cook I and II in the table are, therefore, suitable for obtaining white pulp of pine needles. When higher whiteness is desired, the conditions of cook I may be followed.

The percentage losses in the weight of pulp in each stage of bleaching have been given in the table. It can be distinctly seen that the loss percent in each stage of bleaching is less in the case of sample of cook I than the other two samples of cook II and III. The loss % of cook II sample is also less than the sample of cook III. The minimum loss percent is indicated in the different stages of cook III sample. This also reveales the fact that the more

TABLE NO.6.2

Multi-stage bleaching of the pulp of Pine needles of Cook I and II.

| Particulars | Units | Cook No | |
|---|---|---|---|
| | | I | II |
| Sulfidity | % | 20 | 20 |
| Total alkali as $Na_2O$ | % | 22 | 20 |
| Chlorination: 1st stage | | | |
| $Cl_2$ added on O.D. Pulp | % | 10 | 10 |
| Consistancy | % | 3.0 | 3.0 |
| pH | (pH was not maintained during chlorination due to short time). | | |
| Bleaching temp. | $^oC$ | 25-30 | 25-30 |
| Bleaching time | Hrs | 1.0 | 1.0 |
| Residual $Cl_2$ | % | - | - |
| Losses during chlorination | % | 4.0 | 4.7 |
| Brightness | $GB^o$ | 26.0 | 17.0 |
| Extraction: 2nd stage | | | |
| NaOH added on O.D.Pulp | % | 2.0 | 2.0 |
| Consistancy | % | 10 | 10 |
| pH | - | Over 10 | Over 10 |
| Temperature | $^oC$ | 60-70 | 60-70 |
| Time | Hrs. | 1.0 | 1.0 |
| Residual NaOH. | % | - | - |
| Losses | % | 4.3 | 5.5 |
| Brightness | $GB^o$ | 36.0 | 22.5 |
| Ca-hypochlorite: 3rd stage | | | |
| hypochlorite added on O.D.pulp | % | | |
| Consistancy | % | 5.0 | 5.0 |
| pH | - | 7.6 | 7.6 |
| Temperature | $^oC$ | 40-45 | 40-45 |
| Time | Hrs. | 2.0 | 2.0 |
| Losses | % | 3.2 | 3.5 |
| Total Losses | % | 11.5 | 13.7 |
| Bleached Yield of pulp | % | 88.5 | 86.3 |
| Bleached yield on R.M. | % | 17.9 | 19.4 |
| Brightness | $GB^o$ | 70.0 | 54.0 |

properly the pulp are formed, the less are the lesses in the bleaching. As in the cases of sample II and III the remaining lignin are higher, hence more loss results in the bleaching. Whereas, in the case of the pulp of sample I, they have been almost fully purified, hence little losses result in the bleaching.

In table 6.2 the pulp of pine needles has been bleached by altering the concentration of the bleaching agent. Here, sample of two cook I,II have been treated. Pulp of cook III has not been included as the cooking in this case has not been completed and no brightness is obtainable. As distinct from the table in the chlorination stage, the percentage of chlorine is 10 instead of 7 in the normal case. Similarly the hypochlorite concentration in the last stage is 5% instead of 3%. The brightness after the 3rd stage bleaching is 70% and 54% for the samples of cook I and II respectively. It is clear that by increasing the concentration of bleaching chemicals, improved brightness can be obtained. The increase in concentration of bleaching agents, decreases the pulp yield as evident, the bleached yield of cook No I in the table 6.1 is 18.5% and under increased bleaching agent in Table 6.2 it is 17.9%. Moreover, the degree of brightness is greater in the case of cook I than in the II. It is also evident from the table that bleaching affect of the pulp in the I and II stage, is faster for cook I than the II, whereas the bleaching in the last stage is faster for the pulp of II cook then the I.

Percent losses in different stages of bleaching have been recorded in the table 6.1, 6.2. Higher loss percentage is evident in the case of cook II sample. This further reveals the fact that at comparitively lower alkali concentration the extraction of the lignin material is less and as a result of this, higher losses

occur in the bleaching. Moreover, it is clearly indicated that maximum losses occur in the 2nd stage of bleaching. In fact, in the chlorination stage the non-cellulosic constituents of the fibres are converted into alkali soluble compounds which are removed in the preceding stage of bleaching. In the 3rd stage minimum of the losses are indicated as most of the fibre binding materials are removed in the 1st and 2nd stage of bleaching. In the last stage, whitening of pulp takes place. Comparing the results of table 6.1 with the results of table 6.2, it is evident that at higher concentration of bleaching agents, higher bleaching losses results, which subsequently improves the brightness.

## 7. CONCLUSIONS

1. For any raw material used for paper manufacture it is important that the material should be cheap and abundantly available. Pine needle is a waste product of the forests and, in fact, the very removal of these needles is a great service to the Forest Department as it is the cause of fibre hazard in the forests. The Forest Department collects these needles annually and then these are burnt. Therefore, the collection of pine needles will not be a problem. The importance of this work will further enhance as the Government proposes to set up a Paper Mill at Mansehra. As Mansehra is a central place and almost all the forests are lying in that region, the transportation charges of the needles will be less. The Mill will utilize pine needle with other raw material. With the usage of the pine needles in the proposed paper Mill, the people of the area will be benefited and also the forests will be free of pine needles which will create a good pasture condition for the animals. Therefore, the utilization of pine needle will result in the developments of the forests.

2. The various trials of the cooking conditions show that as the quantity of total alkali is increased, the crude yield of pulp decreases. By using 18% total alkali, the crude yield of pulp is 28%. However, when the concentration of total alkali is increased to 22%, the crude yield of pulp decreases to 25.6%. In the former case, the screened yield is 26.0% while in the latter case, the yield is 23.0%. Although this yield is less as compared with other paper producing raw material, yet the pine needles will not be a costly raw material. As the pine needle is a useless product

of forests and, moreover, its removal will be in the interest of the development of forest in the country.

3. Chemical analysis shows (Table 1.4) that pine needle contains 31.5% of cellulose, 25.6% lignin and 10.2% pentosan. It is comparable with sugarcane bagasse and Kahi grass, which contain about 35% cellulose. The percentage of lignin is higher than that of bagasse and kahi grass, while pentosan percentage is lower. The ash percentage of pine needle is 3.3% which is lower than wheat straw and rice straw and almost equal to Kahi grass. Much higher ash percentage affect bleaching and cooking properties of pulp. It is, therefore, concluded from the chemical analysis that pine needle is suitable for the manufacture of paper board.

4. An important aspect of the bleached pine needle pulp is that its fibre length is 1.23mm and diameter is 19.4 micron. This fibre length is comparable with other materials being used for paper production. The ratio of fibre length to diameter is 63 which is also greater than most of the raw material for paper production. Bagasse has short length fibre just like those of pine needles pulp, generally short fibred pulp is mixed with long fibred pulp in order to increase the strength of the resulting paper. It is better to blend as each fibre will contribute a characteristics property associated with it which could not be easily obtained from the other fibre.

5. Bleaching is an important property of the pulp. The pulp which can be bleached to a high percentage of brightness is considered to be a good pulp. Results of pine needle pulp show that by applying multi-stage bleaching, 10% chlorine applied can produce a brightness of 70% for cook No.I (22% total alkali). When the

total alkali is decreased, the brightness also decrease with the result that by applying 18% total alkali, a brightness of 21% is obtained. In cook No.II by using total alkali of 20%, a brightness of 49% is obtained. The yield of the bleached pulp varies between 18 to 20%. Usually when the paper is of low brightness, it is better to colour the paper, as now-a-days demand of coloured paper is increasing. Pine needle pulp with less brightness can be used for coloured paper.

6. The physical properties of the hand made sheets show that at 45 $^{\circ}$SR freeness, the pine needle properties are comparable with other non-woody raw material and gr-asses. The physical properties of the three cooks differ (Table 5.4). For example, the burst factor of cook No.I (22% total alkali) is highest, while tear factor in cook No.III (18% total alkali) is highest. The tensile strength of cook No.I,II are the same i.e. (3.2 Kg/mm). The breaking length of cook No.II (20% total alkali) is highest (3543 metre). The choice of selection of any one cook depends upon the purpose for which the paper is to be used. If high brightness is required, then cook No.I is best. If high tearing strength is required then cook No.III is good. For paper board and wrapping paper, the pine needle may be used as such (without bleaching). For this cook No.III is best as at this cook, highest yield (28%) is obtained.

7. A comparison of the hand made sheets at 45 $^{\circ}$SR with other paper making materials used in the country shows (Table 5.7) that the properties of pine needles pulp are superior to those of bagasse pulp. The tear factor, bursting strength, folding endurance and breaking length of pine needle are superior to those of bagasse. Bagasse is at present widely used in the country and is mixed with

imported pulp for the production of paper. At present 25% of the imported pulp is mixed with 75% bagasse. As the physical properties of sheets made from pine needles are superior to those of bagasse, so it means that less percentage of imported pulp may be used with pine needle to get a strong fibre. It is, therefore, concluded that pine needle may be used as an alternative to begasse for the production of paper at places where bagasse is not available. Pine needle may also be used as such (without bleaching) for wrapping paper and paper board.

8. Most of the raw materials, which are used, at present, for paper and board making in the various mills, are also used for other useful purposes i.e. rice straw and wheat straw are being used as animal feed, bagasse as fuel and Kahi grass in the construction of houses, but the pine needles are not used for any useful purpose. Beside this, there is another aspect which imparts it unique value for use in the paper mills. It concerns with the abundant and constant availability of these needles, which distinguishes it from the other raw materials, mentioned above, as their availability depends on the crop condition i.e. poor crop would yield less raw material. Moreover, pine trees do not require cultivated land. The cost of this raw material is comparatively very low than the other paper producing raw materials. It is, therefore, concluded that the use of pine needles would be more beneficial than the raw material used at present for paper/board making.

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Project No.
PSF/RES/F-CSIR/UTZ(28)

Project Title

# Utilization of Pine Needle for Textile and Paper Manufacture

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CONTENTS

## 1- INTRODUCTION

After the creation of Bangladesh, Pakistan faced a grave situation in packaging industry, as earlier, the entire need of the country was met by the jute produced in former East Pakistan, but after 1971, jute became an import liability. At present large quantities of jute are being imported to meet the requirements of the jute mills in Pakistan. Efforts are being made to cultivate jute on large areas of Punjab and Sind, but so far only a limited quantity of jute is being produced which is not sufficient for the home consumption. Therefore, the need for other raw materials to partially substitute jute is evident.

The Natural Fibres Technology Division of PCSIR Laboratories, Peshawar, has started investigations on utilizing the natural fibres of the country for the purposes of substitution of jute. A number of fibres such as Sisal, Bindi, Sunn, Elephant grass, Kenaf and Ak were selected and a detailed study was made on these fibres. Some work on Khip fibre was reported by Agricultural University Faisalabad and on Banana and Mazri fibre by PCSIR Karachi. But most of these raw materials are largely scattered and supply is irregular and limited in quantity. Therefore, an industry based on these raw materials may not be economical.

Pine needle is a waste product of the forests and is abundantly available in North Western areas of Pakistan. The disposal of pine needle is a big problem for the Forest Department, and also the material is the cause of fire in the forests. The Forest Department collects these needles annually and burns them. Therefore, the collection of pine needle will not be a problem for concerns intrested in its utilization. One of the advantage

of pine needle is that no extra land is required for its cultivation as the pine trees shed the needles regularly.

Usually, retting is employed for the extraction of vegetable fibre, but the pine needle is hard and the chemical methods only are suitable for fibre extraction. The chemical method employed for the extraction of pine fibre is cheap and simple. The fibre obtained is harsh and, therefore, methods have been devised to make the fibre soft. The fibre obtained can be bleached and dyed.

It is important to know the physical and chemical characteristics of a fibre to evaluate the fibre for specific purposes. The physical properties such as filament length, linear density and strength characteristics of the pine fibre have been determined. The composition of the fibre such as cellulose content, lignin and ash etc. have also been found out to evaluate the suitability of pine fibre for various uses.

The report consists of four chapters covering all aspects of the problem. Apart from pine fibre, other vegetable fibres found in the country have also been included in the report for comparison. Likewise, the retting processes have been discussed in detail, apart from the chemical process for the extraction of pine fibre. Physical and chemical properties of some of the vegetable fibres have been included to make the report interesting and informative. It is hoped that utilization of pine fibre as a jute substitute will provide job opportunities for the poor people of the hilly areas. It will also contribute to the further development of the jute industry.

## 2 - RAW MATERIAL

### 2.1. Types of vegetable fibre as available in different countries:

Fibres of plant origin are used in a variety of textile and industrial products such as hessian cloth, bags, carpet yarn etc. Of all the vegetable fibres, cotton is used extensively. Flax and jute are also best known for their extensive use. Of the estimated 30 billion lbs. of vegetable fibres produced annually on commercial scale[1], it is estimated that one quarter of the said quantity are other than the vegetable fibres mentioned above. In this report all vegetable fibres are discussed except cotton.

Vegetable fibres are generally classified in four groups of fibre (Table No.2.1). The most important is the bast fibre group. The fibres occur in the portion of the fibrovascular area, generally termed the phloem, located around the woody, central portion with the fibres under the outer bark or cuticle of the stalk. The so-called "true" bast fibre or ultimate fibre occur in bundles, with the end overlapping so as to produce continuous filaments throughout the length of the stalk. The bast bundles of fibres are held in place by the cellular tissue of the phloem and by gummy and waxy substances that also hold the fibres to each other within the bundles. The function of the bast bundles is to give strength to the stalk of plant. With the exception of flax and ramie, all the other bast fibres are utilized commercially in the form of full length bast bundles. In the case of flax and ramie, the individual fibres are seperated during the spinning process for

making fibre yarn. Bast fibres are also termed "soft fibres" and the principal textile fibres in this group are jute, flax, sunnhemp ramie and kenaf.

The second group of fibres is the leaf fibre. Here also the fibres occur in bundles i.e. aggregates of individual cells, with the end overlapping so as to produce continous filament throughout the length of the leaf. The fibres are held in place by the cellular tissue of the leaf and by gummy and waxy substances. These substances also serve to hold the fibres to each other within the bundles. The function of the leaf fibre is to give strength and rigidity to the leaf and to give support to the water-conducting vessels. Leaf fibres are often termed "hard" fibres, because they are generally harder, stiffer and coarser in texture than those of the bast fibre group, which were utilized for textile purposes on a large scale before the leaf fibres were discovered. Agave, abaca and pine apple fibre are the most important leaf fibres.

The third group of vegetable fibre is the palm brush fibre, which is usually harder and mostly used for brush making and matting. Coir or coconut and para piassaba are the important fibres of this group. The fourth group is the seed fibres, which is attached to the seed-pods. Kapok and Akund floss are the fibres of this group. The fibre of this group is especially used for packing, insulation and life jackets.

If we look at the Table 1.1, it is evident that most of the vegetable fibre are produced in Asia, Brazil and Indonesia i.e. in less developed countries of the world. In fact in some countries, like Bangladesh, the whole economy of the country depends on jute. Thailand, Philippines and China

TABLE NO. 2.1

Type s of vegetable fibre and their availability

| Type of fibre. | Name of family | Main fibre | Countries(availability) |
|---|---|---|---|
| Bast fibre | Lime (Tiliaceae) | Jute | Bangladesh, India, Burma, Thailand, Brazil. |
| " | Flax (Linaceae) | Flax | Russia, Belgium, Japan, Luxumburg. |
| " | Pae (Leguminosae) | Sunn or Sunn-hemp | India |
| " | Mulberry (Moraceae) | Hemp | Yougoslavia, China, Chile, Philippines, Brazil, China. |
| | Mallow (Malvaceae) | Kenaf | India, Argentina, China, Egypt. |
| " | Sterculia (Sterculaceae) | Abroma augusta | Philippines, Congo. |
| Leaf fibre | Pine apple (Bromeliaceae) | Pine apple | Philippines, China. |
| " | Agave (Agavaceae) | Agave | Brazil, Haiti, Eastern Africa. |
| " | Banana (Musaceae) | Abaca or Manila hemp | Philppines, Singapoure. |
| Palm brush fibre | Palm (Palmae) | Coir or Co-conut | Ceylon, India, Mexico, Jamaica |
| " | Palm | Para Paissaba | Brazil |
| Miscellenous fibre | Bombax (Bambacaceae) | Kapok | Indonesia, India, Thailand Combodia. |
| " | Milkineed (Asclepiadaceae) | Akund floss | India |

R.H. Kirly, vegetable Fibres, Leonord Hill Books Limited London 1963.

have also developed vegetable fibre industry. However, in developed countries large quantities of synthetic fibre are used as a substitute for vegetable fibre.

World production of textile fibres:

Table 2.2 shows the world production of textile fibres, which shows that cotton constitutes 52%, vegetable fibre 23%, man made fibre 17% and wool (scoured) 8%. The production of cotton declimed slightly, while that of synthetic fibre have increased considerably. The production of bast fibre has remain almost constant. For developing countries , like Pakistan, the exploitation of vegetable fibre is more economical than synthetic fibre. Moreover, the exploitation of vegetable fibre requires the use of indigenous raw material which are abundently available in the country. But in the case of synthetic fibre, we have to rely on imported raw material which is not in accordance with the present Govt's policy.

Jute dominates the group of bast fibre, followed by hard fibre, flax and hemp. Data with regard to the production of these materials in 1960, 1965 and 1970 are presented in the table 2.2. The volume of production of jute and related fibre rose from 2.6 million tons in 1965 and has reached to 3.5 million tons in 1970, showing an increase from the 1960 level by 36%. The production of other fibres i.e. flax, hemp and hard fibre remained almost constant in 1960, 1965 and 1970.

Practically all jute comes from Bangladesh and India, each of these countries producing about 45% of the world supplies of the material. The remaining 10% is grown in Brazil, Burma, China and the Soviet Union.

TABLE NO.2.2

World production of textile fibres in 1960, 1965 and 1970.

| Type of fibre | 1960 | | 1965 | | 1970 | |
|---|---|---|---|---|---|---|
| | 1000 Tons | Percent | 1000 Tons | Percent | 1000 Tons | Percent |
| Flax | 630 | 3 | 650 | 3 | 700 | 3 |
| Hemp | 380 | 2 | 400 | 2 | 400 | 1 |
| Jute | 2,580 | 13 | 3,300 | 14 | 3,500 | 12 |
| Hard Fibres | 940 | 5 | 1,000 | 4 | 1,000 | 4 |
| Cotton | 10,150 | 52 | 11,400 | 48 | 12,900 | 46 |
| Wool (Scoured) | 1,450 | 8 | 1,500 | 6 | 1,500 | 5 |
| Silk | 30 | - | 30 | - | 30 | - |
| Viscose | 2,360 | 12 | 2,965 | 12 | 3,600 | 13 |
| Acetate | 252 | 1 | 273 | 2 | 400 | 1 |
| Synthetic | 704 | 4 | 2,035 | 8 | 4,000 | 14 |
| Glass fibre | 100 | - | 175 | 1 | 300 | 1 |
| Total | 19,626 | 100 | 23,728 | 100 | 28,330 | 100 |

The LODZ Textile Seminars, Textile Fibres, United Nations, New York, 1970.

The second group of vegetable fibres that is of some importance comprises the "hard fibres" and includes sisal, manila and the like. The volume of their production has not changed over the period discussed here and amounts to about one million tons. It is very unlikely that the production of these fibres will expand further. The reason for this is the increasing competition by synthetic fibres.

Position of vegetable fibre industry in Pakistan:

Prior to the seperation of east Pakistan, Pakistan jute industry was on firm footing. Apart from meeting the demand of the country, considerable foreign exchange was earned by exporting jute. Withe the creation of Bangladesh in Dec. 1971, Pakistan faced a grave situation, as at that time only a few jute mills existed in West Pakistan. Jute products were imported in large quantities to meet the demand of the country. Priority was given to the establishment of jute industry in Pakistan with the result that in a period of 7 years, three jute Mills were added to the already existing four mills. (Table 2.3). With the establishment of more jute mills in the country, the import of jute products is decreasing, but the import of raw jute is increasing as evident from Table 2.4.

The jute industry in Pakistan depends largely on imported jute. Jute is imported especially from Bangladesh, India, Burma, Thailand and China. The quality of jute from Bangladesh is superior as compared to the jute imported from other countries. In order to place the jute industry on firm footing, it will be necessary to cultivate more jute on more area and to exploit other vegetable fibres of the country in order to use it as such or blend it with jute in order to minimize the import of raw jute.

2.2. Vegetable Fibres already investigated in Pakistan:

Pakistan is rich in various types of vegetable fibres. But

TABLE NO. 2.3

List of Jute Mills in Pakistan

| S.No. | Name of Jute Mill | Locat-ion | Raw material used |
|---|---|---|---|
| 1. | Crescent Jute Products Ltd. | Jaranwala Faizalabad | About 1-5% local jute Imported jute. |
| 2. | Thal Jute Mills | Muzaffar Garh | About 1-5% local jute Imported jute. |
| 3. | Amin Fabric | Kotri | About 1-2% local jute Imported jute. |
| 4. | Indus Jute Mills | Dhabeji-Thatta | Imported jute. |
| 5. | Pakistan Jute & synthetic Mills. | Korangi Karachi. | Imported jute. |
| 6. | Mehran Jute Mills | Korangi Karachi. | Imported jute. |
| 7. | Latif Jute Mills | Bela Baluchistan | Under installation. |

TABLE NO. 2.4

Import of jute and jute products for 1975-76 and 1976 to 1977.

| S.No. | Jute and products | July 1975 * M.T. | June 1976 Rs.(million | July 1976 M.T. | June 1977 Rs. (million) |
|---|---|---|---|---|---|
| 1. | Hessian Cloth | 4,675 | 18.911 | 2,378 | 6.127 |
| 2. | Ropes | 449 | 1.837 | 622 | 2.274 |
| 3. | Twine | 1,076 | 4,262 | 2,456 | 8.637 |
| 4. | Bags, heavy Socks | 1,259 | 7,523 | 639 | 2.199 |
| 5. | Gunny bags | 12,186 | 32.413 | 9,587 | 34.461 |
| 6. | Hessian bags | 775 | 3.337 | 539 | 1.637 |
| 7. | Old bags | 214 | 0.573 | 28 | 0.634 |
| 8. | Jute yarn | 1,728 | 6.197 | 1,835 | 7.006 |
| 9. | Jute cutting | - | 6.197 | - | 7.006 |
| 10. | Others | 254 | 1.006 | 578 | 2.561 |
| | Total | | 69.862 | | 58.530 |

* M.T= Metric Ton.

Monthly statistical Bulletin (1975-77) Statistic Division, Ministry of Finance, Planning & Economic Affairs, Govt. of Pakistan.

unfortunately very litte effort has been made to exploit the natural vegetable f-ibre wealth of the country. This need was felt especially after the seperation of East Pakistan. The Govt. also gave top priority to the cultivation of jute in the Punjab and Sind, but so far with little success. The Wool Research Division (Now Natural Fibres Technology Division) of PCSIR Laboratories Peshawar started an elaborate programme to investigate the natural fibres of the country as a substitute for jute. Some work on Mazri and Banana fibre has also been done in PCSIR Laboratories Karachi and on Khip fibre at the Agriculture University, Faisalabad. The following is the list of fibre which have already been done investigated.

1. Typha Elephantina Roxb (Elephant grass):

Typha Elephantina is a leaf fibre abundantly and cheaply available in marshy places such as the indus Delta, Jhelum and Gujrat Districts. In a study of PCSIR Labs., Peshawar[5] the fibre was obtained by retting. The plant was soaked in water for about a week, when the plant became soft and the fibre could be removed easily. The fibre yield was about 40%.

As regard properties of Typha Elephantina, the mean diameter is 40.1 u and breaking strength is 83.4 gm. wt. It was also found that the strength of root end is greater than tip end, thus confirming Berkley[6] et al results for abaca leaf fibres. The strength of the wet fibre is greater than that of fibres at 65% R.H, which makes the fibre suitable for marine ropes. The stress and tensile strength are inversely related to diameter. This is in general agreement with Stout and Jenkins[7] who showed that the breaking strength of bast fibres increases as the area of cross section decreases.

The results of chemical analysis show that the fibres have approximately the same proportion of cellulose and lignin as compared to leaf fibres. The ash % is, however, high. The percentage of hemicellulose is less than that in other leaf fibres. In view of the above properties, the fibres may be used for coarse textiles, marine ropes and fishing nets.

2. H ibiscus Esculentus Okra (Bindi) :

Okra (bindi) is a popular vegetable of the summer season in Pakistan and is grown in almost every part of the country. After fruiting, the plant is usually burnt. If the stock is collected in the green condition and subjected to retting without drying, useful fibres can be extracted. The fibre is white, light cream or yellow, silky, strong in nature and of fine to coarse qualities depending on the type of retting and maturity of the plant.

The characteristics of okra fibre, show[8] that the mean length of the fibre is 5 inches, which is less than the bast fibre. The mean diameter (26.8 u) is higher than that of jute and flax fibres. The tensile strength is 37.2 $Kg/mm^2$ is smaller than most of the bast fibres, but it is close to that of jute fibres.

The cellulose content of Okra fibre is higher than those of jute, flax and ramie fibres, but lower than hemp fibres. The wax content is higher than that of hemp fibres but lower than that of flax and ramie fibres, and is close to that of jute fibres. The ash content is lesser than that of all bast fibres, but it is equal to that of jute fibres. The above study reveals that okra fibres are close to jute fibres with the exception of filament length and cellulose content, all the other characteristics resemble the characteristics of jute fibre. Okra fibre can be used for ropes etc.

3. Lilaceae Yucca, Glauca (Bear Grass):

From the point of view of fibre production, there are two types of Yucca, the low growing stemless types and the long stemmed Yucca which are tree like in growth. The yucca also differ in flexibility of their leaves. Some have rigid leaves while other have flexible leaves. Yucca Glauca (S.R) lily family (Liliaceae), comes under the later group of fibres. The plant grows wild in abundance in Hazara District, but in some cases, it is cultivated as ornamental plant, irrespective of the other types of the plant of the same botanical family such as Sisal. The leaves are softer in handle and the fibres could be extracted from the leaves very easily giving a 90% yield[9]. Even after two or three days retting, the skin of the leaf and the fibres could be seperated very easily. The fibres are obtained in bundle form and the strands of the fibres are brown to creamy colour like jute.

Filament length of yucca fibres is 11.6 inches and the diameter 40.1 u. This indicates that yucca fibre's maximum length is greater than the minimum length of flax and ramie but smaller than hemp and jute. The fibre diameter is higher than all the four types of bast fibres. It is less elastic than all the bast fibres. The cellulose content of yucca fibres in the present investigation is higher than jute, flax and ramie but lower than the hemp fibres. The wax content is higher than jute and hemp but lower than flax and ramie. The ash content is greater than jute but less than flax, hemp and ramie. Due to its higher degree of flexibility, with more stiffness and less brittleness, it can be easily used in the cordage and brush fibre industries.

4. Ak (Calotropis Procera) Fibres:

Ak (calotropis procera) is a common wild plant of our plains. It has fibre in the seed floss as well as in the stem. The former is short but the latter is long and strong. The fibre when bleached can be spun by itself or mixed with cotton.

Direct extraction without steaming, retting etc., yields the best fibre in terms of strength and colour but the production is the lowest, reed length is low and the produce is entangled[10]. Light beating of the stems in the beginning with a mallet as well as steaming assist in extraction. Retting in water, in general, reduces fibre strength considerably. This results in fibre breakage and consequent shorter reed length. A further disadvantage of the use of water is that, the bark sticks more securely with the pith and when removed results in much shorter reed length. However, mild retting followed by partial drying for a few hours facilitates extraction. Stripping off the bark, followed by mild retting is considered to be the best approach as retting does not then lead to consequent breakage of strips and the reed length is high.

The reed length varies between 2.7 to 40.0 cm. The colour also varied from white to dull and the feel from soft to harsh. The mean strength at "standard" atmosphere is 50.36 $\pm$ 2.9 g/tex. Under these conditions, the strength varies from 44.4 to 56.4 g/tex. These results are expected in view of the differences in origin of the samples. Strength increases slightly with maturity. At 65% R.H, the specific stress increased from about 42.5 g/tex. to 46.0 g/tex. from the pre-flowering to the late flowering stage. The values for diameter were 19.2 to 22.8 u with a mean of 21.6 u and for length were 18.3 to 21.8 mm with a mean of 19.71 mm. Diameter increases slightly with maturity. This increase

is nominal up to the flowering stage. Length also increases slightly with maturity. The ratio of length to diameter (L/B) virtually remains constant averaging 927. There exists a positive correlation between L/B values and strength i.e. the higher the L/B value, the higher the strength. A comparison of strength of twine of equivalent spyndle made from Ak and jute reveals that Ak not only compares favourably with jute but is slightly stronger, indicating the suitablity of the fibre for the range of end uses dependent largely on such level of strength.

5. Sunn-hemp(Crotalaria Juncea) Fibres

Sunn-hemp (Crotalaria Juncea) is a cultivaible plant moustly, grown in India, Pakistan, Uganda, the U.S. and several other countries. In the sub-continent two varieties of sunnhemp are known i.e. Rabi and Kharif varieties.

The fibres are extracted either by retting in stagnant water or in slowly flowing water. Beside this, stripping and retting techniques with mild alkaline solution also lead to fibre extraction. The colour of the fibre varies from white to dirty yellow,with the extraction methods, soil and climatic conditions. The reed length ranges from 4 to 5 feet[11]. The mean fibre strength is 32.06 g/tex. The strength of fibre increases with the increase in relative humidity. At 100% r.h. the average fibre strength is 35.36. g/tex. The mean diameter of sunn-hemp ultimate fibre is 26.71 u and its mean length is 5.93mm. The length to diameter ratio i.e. L/B is 221.4.

The sunn-hemp fibres are used in ropes, twine, cordage, fishing nets and canvas. Besides, the fibres are also used for paper making particularly for cigarette and high quality tissue paper.

6. Cannabis Sativa (Bhang):

Cannabis sativa is abundantly available throughout Pakistan, especially in the tribal belt. It is a wild plant and grows ever-y where up to 9000 feet and even higher. It is some time called "true hemp". Three products are obtained from the plant, i.e. fibre, oil and narcotics. In Pakistan, cannabis sativa is a neglected plant but in some other countries of the world, the plant is cultivated commercially for fibre.

The fibre is e-xtracted either by water retting or dew retting. The latter method is common in Europe, while in Russia and India, water retting is the common practice. The stalks are tied in bundles and immersed in ponds or slow running streams until the bark including the fibre seperates out from the woody inner portion. The duration of retting varies in different localities depending upon the temperature of water. In hot and damp weather, 3-4 days may be sufficient. In cool and dry weather 1-2 weeks are required.

Cannabis fibre is white and lustrous. Fibre Length varies from 11.7 to 17.4 inches[12]. The length can be increased if the method of retting is improved. The fibre is of 22 u thickness. The bundle strength of the fibre shows that breaking strength varies between 1006 to 1773 gm.wt. The linear density is also comparable with other vegetable fibres.

Cannabis fibre may be used as a substitute for jute as its fibre resembles that of jute in softness, length and fineness. The fibre may be used as a packing material, for the manufacture of fine cordage, twine and carpet back yarn.

7. Khip (leptadenia pyrotechnia):

Khip is obtained from wild plants grown over 30,000 square miles of desert area in the Divisions of Bahawalpur,

Khairpur, Hyderabad, Quetta and Kalat. The length of Khip fibre is about 1.5" and fibre finness 5.1 to 5.8 micrograms per inch.[13] The average strength is 104.7 thousand lbs. per square inch. Khip fibre has, however, been found quite suitable for hand spinning in blends with wool. Good quality rugs and carpets can be prepared from wool-khip mixture and wool-khip industries on a large scale can be established in the cholistan area of Bahawalpur Division.

8. Banana fibre:

Banana cultivation has been increasing for the last 10 years. The best species found is Musa cavendishii. Its local names are Basari and Harichal. Banana tree constitutes a huge agricultural waste and continues to pose disposal problem. PCSIR Labs. Karachi have therefore extracted fibre from the leaf of banana tree. The leaf ribs of banana tree are used for cordage. The fibre content in the banana stem is 4-4.5%. The leaf stem fibre is of superior quality, being higher in cellulose content. Fibres in the outer sheaths are strong, while the fibre in the inner core are soft, tender and thin. In view of smaller percentage of fibre, banana fibre is unsuitable for commercial exploitation from economic point of view[14].

9. Mazri fibre:

PCSIR Labs., Karachi is working on the extraction of fibre from mazri plant. Mazri plant is found abundantly in Baluchistan and NWFP provinces. In the raw state, it is extensively used in matting, ropes and a variety of handicraft. Due to high price of mazri, it would not seem economical to extract fibre from mazri. However, the fibre extracted may be blended with jute for package industry.

10. Sisal:

Sisal is obtained from the leaves of the plant. Agave Sisalana, which is available in Hazara and other areas of Pakistan. The leaves are fleshy and require mechanical decortication for seperation of the fibres. The strength of the fibre is higher than jute. It is mainly used in the manufacture of twines, ropes and cordages. Sisal fibre is especially useful for marine ropes due to its moderate resistance to deterioration in sea water.

11. Other vegetable fibres:

Work on Kenaf, Jantar, Kapok and some other fibres is being carried out in the Natural Fibres Division of PCSIR Labs., Peshawar. But so far no data has not been published.

From the above, it is evident that a number of vegetable fibres are available in Pakistan, but so far only Laboratory work has been done on these fibres. Actually no fibre is being used on commercial scale at present in the country. The major reason for this would seem to be that no pilot plant facilities are available for conducting such trials in the country. Moreover, data on economics of the fibre, the estimated quantity etc. is not available. The main criteria for any fibre to be suitable for package industry is that at the factory gate the raw material should be available at a cheap price and it should be available abundantly and regularly. At the moment, the only major use of these fibres is in the cordage industry. In order to utilize these vegetable fibres for commercial use, it is suggested that feasibility reports of individual fibres be prepared and actual trials of individual fibre and in blend with others be carried out to know which types of end product can be made from them.

2.3 Production of Pine Needles:

Pine needles are the product of species of "Chir" pine (Pinus longifolia) which are abundantly available in North Western areas of Pakistan. Pinus longifolia is abundantly available in Hazara, Murree Hills, Azad Kashmir and to some extent in Swat and Dir areas. These needles are longer than other species of pine and in the range of 6-10 inches. Generally pinus longifolia is found in the forest up to 6000 feet altitude. The other type of pine i.e. " blue pine". (Pinus wallichiana) is found abundantly in Gallis, Kaghan and to a lesser extent in Murree Hills, Azad Kashmir and Baluchistan. The needle of this type is shorter in length than chir pine. Blue pine is usually found between 6000 to 12000 feet in altitude[15].

The mature dried needles from pine tree that fall on the ground are dark brown in colour. They lie under the trees of these forests in the entire bed of the forests area. These needles are not put to any useful purpose. In fact, the presence of these needles is a big problem for the Forest Department due to the following reasons.

1. They check the growth of new plants and thus create hinderance in the expansion of these forests.
2. The needles stop the growth of grasses which are essential for the animal life.
3. The most stricking disadvantage of these needles is that sometimes, they prove to be the cause of fire in the forests.
4. When the needles are moistened, they are slippery and occasionally result in incidents causing servere injuries or loss to human life.

TABLE NO. 2.5

Area of Forests and Rangelands under the control of Forest Departments (thousand hectores) 1976-77

| Provinces | Coniferous | Irrigated | Scrub | Rangelands |
|---|---|---|---|---|
| N.W.F.P. | 854.7 | - | 269.5 | 1.7 |
| Punjab | 67.2 | 115.3 | 328.2 | 2,702.1 |
| Sind | - | 69.6 | 13.4 | 454.4 |
| Baluchistan | 115.7 | 0.8 | 596.9 | 371.6 |
| Northern Areas | 284.9 | 0.8 | 658.0 | 2,104.4 |
| Azad Kashmir | 367.5 | - | 12.9 | 202.4 |
| Total | 1,690.0 | 186.5 | 1878.9 | 5,836.6 |

General Directorate Bulletin No.9 (Forest Economics Branch) Pakistan Forest Institute, Peshawar.

The Forest Department collects these needles annually and burns it. Therefore, the collection of these needle is not a problem. Moreover, the very removal of these needles will result in the development of the forests.

Table No.2.5 shows the area under forests of coniferous, irrigated, scrub and rangelands. It is evident that most of the areas of pine trees are located in NWFP. Northern areas and Azad Kashmir. The total area of coniferous forests is 1,690 thousand hectors[16]. The pine tree sheds the needles throughout the year. Therefore, the supply of these needles will be constant throughout the year. The best and economical way is to establish small plants near these hilly areas like Mansehra, Murree hills, Kaghan, Swat and Azad Kashmir for the extraction of pine fibre. The pine fibre can be transported to a nearby jute mills, thus reducing the transportation cost. In this way the people of hilly areas will benefit as they will get employment and the waste pine needle will become a useful industrial raw material.

## 3. EXTRACTION OF FIBRES

Generally the bast fibre is extracted by retting process and leaf fibre is extracted by crushing and beating. For this, special decortication machines have been manufactured for extraction of the fibre. Pine needle is very hard and the fibre cannot be extracted by retting. Beating or crushing will not be of much use. The only suffessful method of extraction is the chemical process. Various processes for the extraction of vegetable fibres are discussed below.

### 3.1 Retting processes:

Retting is the most important process in the production of fibre. If retting is not carried out properly, the fibre may be ruined or the quality lowered. Retting cannot improve the fibre which is in the plant, but proper retting can ensure that the original properties of the fibre are maintained and not lost.

To understand the retting process, it is necessary to consider the nature of the bast fibre and the position in which it is situated in the stem. If a stem be examined in cross section, certain definite layers can be observed. The outside layer consists of a waxy covering called cuticle which envelopes the next layer, the epidermis. The epidermis has stomata or pores and it is through these that the bacteria enter the stem at the beginning of the retting process. Beneath this epidermis is the 'cortex' and inside the cortex are the bast fibres. These fibres occur in bundles in the pericycle, each bundle containing individual fibres or ultimate fibre cells and one bundle represents one strand or filament of fibre.

The process used for this isolation is retting or rotting, by which the stems are submitted to the action of water, fungi and bectaria, which decompose the material surrounding the woody part of the stem but leave the fibre bundles intact. The soaking in water softens and seperates out the straw and at the same time expells the air and extracts the water soluble materials. The various bacteria and other micro-organisms which are present on the stems, or in the small pieces of soil that are attached to the stem, then develop rapidly if conditions are favourable and break down the straw. The organisms which are commonly responsible for the retting are sporeforming bacteria which are regarded as variants of a single species. During retting the bacteria enter the stem through the stomata. If, however, the retting process is allowed to go too far, the cementing material binding the fibre together in the bundle will also break down. The great art in the retting is to know exactly when to stop the process so that the natural characteristics of the fibre are unimpaired. This art can only be acquired by experience and although the retting process itself is a very complex bio-chemical and biological process, there are no scientific yards-ticks which can be used on their own to indicate when the retting should be stopped.

The time taken for retting depends on a number of factors e.g. the temperature of the water, the nature of the water used and the crop itself which varies, of course, from season to season and from batch to batch. One of the most striking facts about the retting of fibre is that is a highly complex scientific process which is carried out by persons who generally have no scientific knowledge whatsoever. Good judgement

and considerable experience on the part of the retter are required if best results are to be obtained. The two main retting techniques are dew retting and water retting.

1) Dew retting:

Dew retting is best done in the spring, as in late autumn or winter the temperature is often too low and difficulties are experienced in drying the retted fibre. Moderate humidity (which can be provided either from dew or from rain), warmth and freedom from wind, provide the ideal conditions for dew retting. A shower of rain just after the plant (flax or jute) has been spread on the ground not only helps the retting but also helps to weigh the plant down, preventing it from being blown about by wind.

The time taken for dew retting depends on the prevailing weather conditions and the presence in the soil of the necessary bacteria or fungi to start the retting. Moulds and other fungi are the principal retting agents in dew retting. The average time taken seems to be from about three to seven weeks, but under really favourable conditions retting may take only two weeks, whereas under very unfavourable conditions it may take as long as three months. To test the progress of the ret, stems are taken out periodically, dried, and broken at short intervals along the stem and moved backward and forwards. If retting is completed, the wood or shive seperate out easily, although the best method of testing is to dry the straw and scutch it.

Dew retting is cheaper than other methods of retting, as it requires no capital expenditure. However, it has many disadvantages. It may take long time, depends almost entirely on weather conditions and requires the use of fairly large areas of

ground which cannot be used for any other purpose entil retting is finished. Where the cost of labour is high spreading and turning can also be expensive. From the commercial point of view, the main disadvantage is that no control can be exercised over the process and this is why it is used for poor quality straw which is not considered worth retting in water.

2) Water retting:

The better quality grades are retted in water, either in rivers or ditches or in retting tanks or vats which have been especially installed in factories or in water retting. However, whichever medium is used, the principles involved in the retting process are much the same. Before retting, the straw should be classified into different qualities or grades and any bad straw should be removed from the beets or bundles. Sometimes the beets are put into the water singly or sometimes two beets are tied togather with two or three bands. Retting in rivers or ponds may take between two to three weeks or longer, but much depends on the speed of flow of the river and its temperature. The temperature of the water is important and water at low temperature (around 60F$^{o}$) is normally considered unsuitable.

3) Warm water retting:

The modern method of retting, however, is with warm water and in this method the retting process is made to start and proceed more quickly. From the commercial aspect, the method is more efficient, as a proper control can be kept over the retting process, so that the best results can be obtained from the point of view of fibre quality. However, the capital cost is high compared with that for cold water retting in rivers, ponds etc. One of the chief causes of faulty or difficult retting is the

use of two little water for the amount of straw which is being retted. Unless sufficient water is used to dilute the acids produced during retting, the bacteria will be destroyed or reduced in number and retting will take much longer time. The temperature should be never above 76 $F^{O}$.

4. Double retting:

This process is used for good quality flax or other vegetable fibres. The main retting process is almost completed in the first ret, while the second ret is only complementary to it and is carried out by the offspring of the surviving spores of the bacteria from the first ret. Where the straw is being retted in rivers or ponds, it is retted for between five and seven days and is then taken out and allowed to dry in the fields. It is then commonly put in the water again for the second ret, alternatively it may be stacked and kept until the next season for the second ret.

The second ret will normally only last for about one to two days. If warm water retting is being carried out, the first ret will last about three days and the second ret about one or two days at the most. The variation of the double retting method is merely to leave the straw in the tank after the first ret, empty the tank and refill it with fresh water in whic h the straw is left standing for about two hours. The tank is then emptied again, the straw being left standing exposed to the air. The second ret is then carried out in the usual way. This method does not give such good result but does, of course, save the labour and cost of unloading and re-loading the tanks and drying the straw before the second ret.

The advantages of the double ret are that most of the obnoxious materials are removed in the first ret in which the soft tissues have been broken down. In the second ret, although some fermentation does go on, it is not so rapid and consequently there is far less danger of over retting than with the single ret. One great advantage of double retting is that it gives the retter an opportunity to examine the straw thoroughly after the first ret, so that he can estimate with fair accuracy the time that is likely to be required for the second ret.

3.2 Extraction from leaf fibres:

Usually retting process is applied in the case of bast fibre. The fibre is extracted from the leaf fibre by the following methods.

(i) Hand stripping method:

The hand method consists of drawing the strips or "tuxies" while the leaves are still fresh, between the edge of a knife and a hard smooth wooden block attached to a light frame. The knife usually has serrations in its edge and the more numerous the serrations are and the greater the pressure which is put on the tuxie, the finer will be the fibre produced but the lower will be the yield. Owing to the harder work and the higher waste in producing the finest fibre, when prices are low, there is a tendency to produce the coarser fibre which requires less work and involves less waste. After stripping, the fibre is hung on bamboo poles or wire lines and allowed to dry in the sun as quickly as possible. It is then tied up in bundle and graded.

(ii) Mechanized Hand Stripping:

Hand stripping is laborious work and by this method only small quantity of fibre can be produced per man per hour. Therefore, to obviate some of the hard work which is entailed in hand

stripping, a machine known as the "Hagotan" is used. In principle, this consists of a slightly tapered, cylindrical block attached to a spindle in front of the knife. The tuxies are inserted under the knife in the usual way and the other end of the tuxie is wound around the block on the spindle, but the extreme end is still held in the hand by the s-tripper. The spindle is driven by a small engine and as it revolves, the stripper gives the tuxie a steady pull outwards, away from the knife. The revolving spindle helps to pull the strips under the knife, thus saving the labourer the hard work of pulling the "tuxie". The block and knife used are similar to those used in hand stripping. When one half of the tuxie has been cleaned, the tuxie is reversed and the other portion is cleaned. Despite the obvious advantages of the "Hagotan" machine, however, a considerable portion of the crop of the fibre in the Philppines is still produced by the ordinary hand-stripping methods. This is rather costly for growers, who only produce small quantity of the fibre.

(iii) Decortication machines:

Decortication machines have been built in Philippines and Indonesia for decortication of Sisal and abaca leaves. There are minor differences in the machines but the main principle is the same. The fibre from such machines is known in the trade as "Deco" fibre or decorticated fibre. The machine can deal with 35 to 40 tons of stems per hour and the whole stem is put through it, no attempt being made to seperate the outer and inner sheaths.

For the extraction of abaca fibre, a Japanese firm has made a machine known as "Kawahara machine". In this machine the tuxie is fed in by hand. Decortication is done in two continous stages, one half of the tuxie being cleaned on one decorticating

drum and the other half on a second drum, following which the fibre is delivered at the end of the machine on a conveyor belt.

Table No.3.1 shows a list of fibres of the different families and their extraction methods. It is evident that all the fibres are not retted. Some of the fibres like ramie, sisal, pine apple , abaca are not retted at all and special machines have been manufactured to extract the fibres. Only two fibres i.e. kapok and Akund floss are used as such and their collection is the main problem. However, the majority of fibres are obtained through retting process.

In Pakistan vegetable fibres are not extracted on commercial scale. The only vegetable fibre used in the mills i.e. jute is produced in limited quantity in the Punjab and Sind. The limited jute produced in the country would result in low quality, because the farmers do not know the retting methods and moreover sufficient water is not available for retting purposes. In other jute producing countries like Bangladesh, Thailand and India etc., the people are fully trained in the retting process and sufficient water is available for extraction of the fibre. In Pakistan , the cultivation of jute is introduced recently. The farmers are not aware of the retting process with the result that the quality of jute produced cannot be maintained at a high level at present. The concerned authorities may start an elaborate programme to train the farmers in retting process, so that in future the quality of jute is not lowered due to retting process.

3.3 Chemical extraction of pine fibres:

The pine needle is very hard and it is not possible to extract fibre with the common retting process. Chemical method is best suited for the extraction of fibre from pine needles, but

TABLE NO. 3.1

Various types of fibres and the extraction process.

| S.No. | Name of Fibre | Botanical name | Extraction process |
|---|---|---|---|
| 1. | Jute | Carchorus Capsuloris colitrius | Retting |
| 2. | Flax | Linum usitatissimum | Retting |
| 3. | Sunn or Sunn-hemp | Crotalaria juncea | Retting |
| 4. | Hemp | Cannabis Sativa | Retting |
| 5. | Ramie | Bochmeria nivea | Hand/Scrapping |
| 6. | Sisal | Agave Sisalana | Crushing/Scrabbing |
| 7. | Henequen | Agave fourcroydes | Crushing/Scrabbing |
| 8. | Abaca | Musa textiles | Hand/ Scrabbing |
| 9. | Coir, Coconut | Cocos nucifera | Retting |
| 10. | Para Piassave | Leopoldinia piossaba | By hand |
| 11. | Kenaf | Hibiscus Cannabis | Retting |
| 12. | Abroma augusta | Abroma augusta | Retting |
| 13. | Pine Apple | Ananos Comosus | By hand /Crushing |
| 14. | Kapok | Cieba pentandra | Collection as such |
| 15. | Akund floss | Calotropis procera | Collection as such |
| 16. | Broomroot | Muhlenbergia macroura | Collection as such |

the chemical used should be cheap and the process should be simple. In the present work two methods of extraction are used. In the first method commercial sodium hydroxide is used while in the second method commercial sodium carbonate is used. The two processes have been discussed below:

1. Sodium hydroxide method:

The following methods were adopted for the extraction of fibre by varying the concentration of sodium hydroxide, temperature and time of treatment.

i) The pine needles as such were boiled for 30 minutes in 4% commercial sodium hydroxide solution. The sample to solution ratio was about 1:50. The sample was then rinsed thoroughly with water, dried at room temperature and hand carded. The colour of the fibre was brownish and the fibre yield was about 51% (Table No.3.2).

ii) In this method, the concentration of sodium hydroxide was kept at 2%, but the time of boiling was increased to one hour. The colour of the fibres obtained was brownish and the fibre yield was about 53%.

iii) In this method, the concentration of sodium hydroxide was kept at 4%. The needles were kept at a temperature of 35-40 $C^{o}$ for 24 hours. Usually this temperature occurs in the summer season. The fibres obtained by the method were brownish in colour. Fibre yield was higher (about 61%) than the other methods.

iv) In this method, the needles were treated with 2% sodium hydroxide solution for 48 hours at a temperature ranging from 35-40 $C^{o}$. Fibres obtained were brownish in colour and the fibre yield was about 58%.

Table No.3.2 shows the yield (%) of pine fibres by using 4% sodium hydroxide solution for 30,60 minutes boiling. The yield at 30 minutes boiling was 51%, while that of 60 minutes boiling was highest i.e. 61%. Fibre yield (%) of 2% sodium hydroxide at 45 minutes boiling was 53%.

Table 3.3 shows the strength and elongation by using 4% sodium hydroxide for 30 minutes boiling time. There are large variation in the strength of individual fibres ranging from

TABLE NO.3.2

Comparison of fibre yield (%) by sodium hydroxide and sodium carbonate methods.

| Particulars | Sodiun Hydroxide Method % yield | | | Sodium Carbonate Method yield % | | |
|---|---|---|---|---|---|---|
| Sample No.1 | 51 | 52 | 62 | 80 | 69 | 68 |
| Sample N o.II | 50 | 51 | 60 | 75 | 72 | 65 |
| Sample N o.III | 52 | 54 | 63 | 79 | 70 | 66 |
| Sample No.IV | 53 | 53 | 62 | 74 | 71 | 68 |
| Sample N o.V | 50 | 55 | 61 | 72 | 72 | 55 |
| Mean | 51 | 53 | 61 | 76 | 70 | 66 |
| Sodium hydroxide % | 4 | 2 | 4 | 4 | 4 | 4 |
| Time of treatment (Minutes) | 30 | 45 | 60 | 30 | 45 | 60 |
| Temperature $C^{o}$ | 100 | 100 | 100 | 100 | 100 | 100 |

TABLE NO.3.3

Strength and Elongation of Pine fibre extracted by using sodium hydroxide (4%) for 30 minutes.

| S.No. | Strength (gm.wt.) | Elongation % |
|---|---|---|
| 1. | 63.5 | 3.7 |
| 2. | 69.1 | 5.1 |
| 3. | 61.8 | 2.9 |
| 4. | 61.2 | 5.5 |
| 5. | 73.1 | 5.0 |
| 6. | 56.1 | 8.5 |
| 7. | 52.9 | 8.3 |
| 8. | 53.2 | 5.6 |
| 9. | 53.8 | 6.7 |
| 10. | 52.8 | 7.3 |
| Mean | 58.1 | 5.8 |

52.8 to 73.1 gm wt. The mean value of strength is 58.1 gm wt.

2. Sodium carbonate method:

As in the case of sodium hydroxide method, in this method also, the concentration of commercial sodium carbonate, time of treatment and temperature have been varied for the extraction of pine fibre.

a) Known quantity of needles was boiled for 30 minutes with 4% commercial sodium carbonate solution. The ratio of sample to solution was kept at 1:40. After the treatment, the needles were washed with water repeatedly and softly beaten with wooden hammer and washed again. After partial drying, the fibres were carded. The fibre obtained were light brown in colour. The yield of fibres was in the range of 72-70%.

b) The needles were boiled for one hour with 4% commercial sodium carbonates solution. The fibres were thoroughly washed. While the treated semi-fibrous needles are still moistened, they are rubbed against a hard surface or thoroughly with hands with the result that the fibres are seperated out. The fibres were light brown in colour and the yield was in the range of 55-68%.

c) The needles were boiled for 45 minutes with 4% commercial sodium carbonate solution. The fibres were washed thoroughly and beaten lightly to obtain fibres. The fibres obtained were of light brownish colour and the yield was 69-72%.

Table 3.2 shows the yield (%) of pine needles using 4% sodium carbonate for 30-45 and 60 minutes boiling time. It is evident that as the time of treatment is increased , the yield decreases. At 30 minutes boiling, the yield is highest i.e. 76%. At 45,60 minutes boiling, the yield is 70 and 66% respectively.

TABLE NO.3.4

Strength and elongation (%) of fibres extracted by sodium carbonate method.

| Fibre No. | Sample No.I | | Sample No.II | | Sample No.III | |
|---|---|---|---|---|---|---|
| | Strength (gm.wt.) | Elongation % | Strength (gm.wt.) | Elongation (%) | Strength (gm.wt.) | Elongation (%) |
| 1. | 76.7 | 6.1 | 67.7 | 6.8 | 60.9 | 7.1 |
| 2. | 72.9 | 6.1 | 61.8 | 4.6 | 53.9 | 7.5 |
| 3. | 66.5 | 6.4 | 56.3 | 5.0 | 76.5 | 7.6 |
| 4. | 73.9 | 5.5 | 60.5 | 10.8 | 71.0 | 6.4 |
| 5. | 58.0 | 8.3 | 50.2 | 6.3 | 59.4 | 4.8 |
| 6. | 70.0 | 8.6 | 67.6 | 9.4 | 63.2 | 7.6 |
| 7. | 63.0 | 7.3 | 52.9 | 7.7 | 57.6 | 7.5 |
| 8. | 69.0 | 5.9 | 62.0 | 8.3 | 53.0 | 7.1 |
| 9. | 71.3 | 7.8 | 64.0 | 9.0 | 61.0 | 7.2 |
| 10. | - | - | 59.5 | 6.2 | 55.4 | 6.0 |
| Mean | 68.0 | 7.0 | 61.8 | 7.6 | 61.1 | 6.8 |
| Concentration% | 4 | | 4 | | 4 | |
| Time (Min.) | 30 | | 45 | | 60 | |

Table No.3.4 shows the strength and elongation using 4% sodium carbonate for 30,45 and 60 minutes boiling time. It is evident that the strength is maximum (68.0 gm.wt.) at 30 minutes boiling and about the same (61.0 gm wt) at 45 and 60 minutes boiling.

It is concluded that sodium carbonate method is superior to that of sodium hydroxide method as the yield (%) is highest at 4% sodium carbonate and 30 minutes boiling and about the same (61.0 gm. wt.) at 45 and 60 minutes boiling.

It is concluded that sodium carbonate method is superior to that of sodium hydroxide method as the yield (%) is highest at 4% sodium carbonate and 30 minutes boiling. The yield is 76%. The strength is also maximum (i.e. 68.0 gm. wt.) at this condition. Beside this, the fibres are superior in feel and lighter in colour in comparison with the colour and feel of the fibres obtained by sodium hydroxide method. Moreover, the use of sodium carbonate is not only economical but also harmless, whereas sodium hydroxide is expensive as well as corrosive and in certain cases it reacts with the vessesls used for extraction.

# 4. CHARACTERISTICS OF PINE FIBRE

## 4.1 Physical properties of pine fibre:

There seems to be little work carried out on the characteristics of vegetable fibre in comparison to such aspect as harvesting, retting and marketing etc. Moreover, the properties of vegetable fibre vary with the stage of maturity of plant, soil, climatic conditions, retting process etc. Therefore, large variation occurs in the properties of vegetable fibres which should be properly investigated. The word pine fibre has been used in this study to mean the technical fibre or the filament and not a ultimate fibre.

### 1. Fibre Fin-eness/Linear Density:

The fineness of a fibre is the diameter or thickness of its cross-section, normally this will be the thickness of a single strand or filament, which is itself made up (in cross section) of a number of ultimate fibres. The fineness will depend, therefore, on the number of fibre ultimates in the bundle and the diameter of the ultimates. The fineness depends on the plant from which the fibre is obtained and also whether it is a stem or a leaf fibre.

The fineness can be expressed in a number of ways, either in terms of the diameter of the cross sectional area or the weight per unit length ( linear density)[17]. The tex system has been adopted by the A.S.T.M. as a standard unit for designating the linear density of a textile fibre. The tex value is the weight in grams of 1,000 metres of the fibre. The linear density of the fibre is determined by direct weighing on a vibrascope. In the present work pine fibres of various length were weighted by an accurate chemical balance and the corresponding lengths were measured.

Table 4.1 gives the linear density of 17 samples of pine fibres. It is evident that there are large variations in the values of linear density between the samples (range 90 to 137 ug/cm), the mean value being 114 ug/cm.

2. Strength Characteristics:

A certain minimum strength is a necessity for any fibre that is to be used for making yarn.[18] Because of this fact attention has been paid to the measurement of s trength of fibres and to the effect of the method of manufacture. In the course of these operations the fibres are combed with steel pines and made to bend round various fluted rollers moving at fast speeds, so that unless the fibres are sufficiently strong, they will not be able to withstand such treatment and the strength of the final yarn will be unsatisfactory.

The strength and binding properties of the fibres are thus very important and in the case of hard fibres it is estimated that fibres in the sliver will have lost as much as 25 percent of their strength owing to the bending and piercing activities of the gill pins and the fraction of the fibre against fibre and of fibre against the pines.

Table No.4.2 gives the strength characteristics of individual pine fibre. The fineness is expressed in micron and in tex. systems. The tenacity is expressed in tex only. The fibre fineness ranges from 145.2 u to 165 o u, mean being 153.7 u. In tex value, it varies from 24.0 to 27.6. There are great variations in the strength at breaking point of pine fibre (range 41.0 g wt. 80 gm wt) the mean value is 58.7 gm wt. The mean tenacity is 2.17 g/tex and the mean tensile strength is 3.14 Kg/ $mm^2$. Elongation at break is 2.7% (range 2.0 to 4.1%). There is no relationship of

TABLE NO. 1

Linear Density of Pine fibres

| Sample No. | Length (cm) | Weight (mg) | Linear Density ug/cm |
|---|---|---|---|
| 1. | 18.9 | 2.20 | 116 |
| 2. | 20.8 | 2.40 | 115 |
| 3. | 19.2 | 2.25 | 117 |
| 4. | 17.8 | 2.10 | 118 |
| 5. | 21.2 | 2.33 | 110 |
| 6. | 19.3 | 2.38 | 123 |
| 7. | 15.9 | 1.43 | 90 |
| 8. | 20.5 | 2.49 | 121 |
| 9. | 18.1 | 1.90 | 105 |
| 10. | 17.1 | 1.80 | 105 |
| 11. | 10.3 | 1.13 | 109 |
| 12. | 13.4 | 1.40 | 104 |
| 13. | 9.1 | 1.06 | 116 |
| 14. | 10.4 | 1.26 | 121 |
| 15. | 10.9 | 1.50 | 137 |
| 16. | 11.5 | 1.25 | 108 |
| 17. | 9.9 | 1.15 | 116 |
| Mean | 15.5 | 1.76 | 114 |

TABLE NO.4.2

Strength characteristics of individual pine fibre

| Fibre No. | Fineness | | Strength | Elongation | Tenacity | Tensile strength $kg/mm^2$ |
|---|---|---|---|---|---|---|
| | Tex | Micron | gm.wt. | % | g/tex | |
| 1. | 26.7 | 152.8 | 44 | 3.1 | 1.65 | 2.38 |
| 2. | 26.2 | 151.6 | 50 | 2.0 | 1.91 | 2.76 |
| 3. | 31.0 | 165.0 | 65 | 3.1 | 2.10 | 3.03 |
| 4. | 26.9 | 153.8 | 73 | 2.0 | 2.71 | 3.91 |
| 5. | 27.5 | 155.4 | 80 | 4.1 | 2.91 | 4.20 |
| 6. | 25.3 | 152.0 | 75 | 3.1 | 2.85 | 4.12 |
| 7. | 24.0 | 145.2 | 34 | 2.0 | 1.41 | 2.04 |
| 8. | 26.0 | 151.2 | 73 | 4.1 | 2.81 | 2.05 |
| 9. | 27.6 | 155.8 | 41 | 3.1 | 1.49 | 2.14 |
| 10. | 26.0 | 154.0 | 52 | 2.0 | 1.93 | 2.78 |
| Mean | 26.8 | 153.7 | 58.7 | 2.7 | 2.17 | 3.14 |

breaking strength with diameter or area of cross section in the case of pine fibre. This is not in accordance with Stout and Jenkin's observation who have shown that the breaking strength of bast fibres in-creases as the area of cross section decreases.

Table 4.3 gives the mean values of strength characteristics of pine fibres. There are variations in individual samples, but the overall pattern is the same as given in Table N o.4.2. The overall mean elongation (%) is 3.1 and tensile strength is 2.87 $kg/mm^2$.

3. Comparison of wet and dry strength:

The strength of fibres alters when they become wet and it is important for some uses to know their wet strength as well as their dry strength. Although cellulose absorbs water, it is nevertheless insoluble in water. The fact that fibres are able to absorb water internally is extremely important from the point of view of their end-use; for if they could not absorb moisture internally in damp atmospheres, surface condensation would take place on them and clothing made from them would become clammy and unpleasant to wear.

It is important to know the behavious of fibres soaked in water. In most of the cases, the strength properties change by soaking in water for 24 hours. In the cases of Sisal and Elephant grass, the strength increases and also the elongation (%) increases. In the case of pine fibre (Table No.4.4) the strength of the individual fibre decreases and the elongation increases. The mean strength of 67.7 gm. wt. decreases to 32 gm. wt. thus strength of each fibre is decreased by overall 36 gm wt. The mean elongation (%) changes from 5.4 to 7.9% an overall increase of 2.6%. In

TABLE NO.4.3

Mean values of strength characteristics of pine fibres

| Sample No. | Fineness Tex | Strength gm.wt. | Elongation % | Tenacity g/Tex | Tensile strength $g/mm^2$ |
|---|---|---|---|---|---|
| 1. | 33.5 | 55.7 | 3.8 | 1.67 | 2.42 |
| 2. | 33.0 | 57.4 | 4.0 | 1.74 | 2.51 |
| 3. | 29.8 | 54.5 | 3.8 | 1.83 | 2.64 |
| 4. | 26.4 | 57.1 | 3.1 | 2.17 | 3.13 |
| 5. | 28.7 | 57.1 | 3.4 | 2.01 | 2.90 |
| 6. | 26.0 | 56.4 | 3.2 | 2.17 | 3.14 |
| 7. | 24.9 | 56.1 | 2.8 | 2.25 | 3.25 |
| 8. | 26.9 | 58.7 | 2.8 | 2.18 | 3.15 |
| 9. | 26.4 | 50.0 | 3.2 | 1.89 | 2.73 |
| 10. | 27.4 | 54.5 | 3.1 | 1.99 | 2.87 |
| Mean of means. | 28.3 | 55.7 | 3.1 | 1.99 | 2.87 |

TABLE NO.4.4

Comparison of dry and wet strength of pine fibres

| Fibre No. | DRY | | WET | | Decrease in strengh | Increase in elongation |
|---|---|---|---|---|---|---|
| | Strength gm.wt. | Elongation % | Strength gm.wt. | Elongation % | | |
| 1. | 70 | 7.1 | 50 | 11.4 | 20 | 4.3 |
| 2. | 30 | 2.9 | 18 | 11.4 | 12 | 8.5 |
| 3. | 130 | 5.7 | 50 | 7.1 | 80 | 1.4 |
| 4. | 55 | 5.7 | 30 | 10.0 | 25 | 4.3 |
| 5. | 60 | 7.1 | 40 | 8.5 | 20 | 1.4 |
| 6. | 65 | 7.1 | 25 | 7.1 | 40 | 0.0 |
| 7. | 65 | 5.7 | 23 | 4.3 | 42 | 1.4 |
| 8. | 57 | 5.7 | 26 | 7.1 | 31 | 1.4 |
| 9. | 52 | 4.3 | 18 | 7.1 | 34 | 2.8 |
| 10. | 90 | 7.1 | 40 | 7.1 | 50 | 0.0 |
| 11. | 45 | 5.7 | 26 | 5.7 | 19 | 0.0 |
| 12. | 35 | 2.9 | 22 | 8.5 | 13 | 5.6 |
| 13. | 70 | 4.3 | 26 | 7.1 | 44 | 2.8 |
| 14. | 106 | 4.3 | 52 | 10.0 | 54 | 5.7 |
| 15. | 85 | 5.7 | 30 | 5.7 | 55 | 0.0 |
| Mean | 67.7 | 5.4 | 32 | 7.9 | 36 | 2.6 |

determing the suitability of any fibre for textile purposes a fibre which becomes strong in water or in sea water is suitable for fishing net and marine rope respectively.

4. Dimension of ultimate fibre:

The determination of dimensions of ultimate fibres is important as the properties of yarn of a vegetable fibre depend largely on the dimension of ultimate fibre. One strand of jute fibre as used in spinning, which may be about 5 to 10 feet long, will be made up of five to twenty or sometimes more ultimate fibres in its cross section, whilst a strand of flax will contain about 10 to 40 ultimates in its cross section. Such a fibre strand is, in fact, a single fibre bundle as it occurs in the plant. A single strand of jute about 6 feet long may be made up in its length of as many as 700 ultimate fibres overlapping each other along the length of the strand. However, as in the case of ramie, the ultimate cells are comparatively long, the single ultimate cells can be used for spinning on there own. Generally, the ultimate cells are too small to be used on their own except for paper making and the fibres used for spinning are strands consisting of numerous single ultimate cells bound togather.

Table 4.5 shows the length and diameter of ultimate fibres obtained from fibres. It is evident that the diameter varies from 27.2 to 36.2 u, a mean value of 32.3 u. There are variations in fibre length also (range 10 to 16 mm), mean being 13.3 mm.

4.2 Chemical composition of pine fibres:

All vegetable fibres are primarily cellulosic and in most instances, before fabrication into textile products, the non-cellulosic compounds are removed by various purification

TABLE NO.4.5

Ultimate fibre dimension of pine fibre.

| Fibre No. | Diameter u | Length mm |
|---|---|---|
| 1. | 36.6 | 12 |
| 2. | 36.2 | 14 |
| 3. | 27.6 | 11 |
| 4. | 31.8 | 15 |
| 5. | 32.4 | 13 |
| 6. | 34.6 | 16 |
| 7. | 29.0 | 14 |
| 8. | 29.4 | 10 |
| 9. | 34.6 | 16 |
| 10. | 31.4 | 12 |
| Mean | 32.3 | 13.3 |

procedures. Cellulose is the most abundant natural polymer, being a major component of all plants. In the solid state, cellulose is strongly hydrogen bonded, both inter and intramolecularly through many hydroxyl groups. Because of this hydrogen bonding and also in view of its molecular weight, cellulose is insoluble in normal non-reactive solvents. The present of cellulose in pine fibre is about 60.0% (Table 4.6). Bevan chlormation method is employed for cellulose determination.

The total number of units in a cellulose chain is known as its degree of polymerization (d.p.). Since cellulosic fibres consist of many chains of different lengths, the d.p. of any cellulosic fibre represents the average length of many chain molecules of which it is composed. The physical properties of cellulosic and other macromolecular fibres depend to a great extent on the d.p., the degree of crystallinity and the orientation. Crystallinity involves an orderly arrangement in only one direction or dimension. Usually high d.p. and high orrientation contribute to increased tensile strength. High crystallinity usually makes the fibre less swellable and less easily penetrated by dissolved substances such as dyestuffs. The cellulose content of the fibre is the residue after chlorination or exposure to chlorine gas for some time, followed by extraction with hot sodium sulphite solution. The higher the percentage of cellulose in a fibre, the greater will be its value.

The ash, which is obtained by igniting most native cellolosic fibres, contains sodium potassium, calcium and magnesium as its principle cations, together with traces of iron and other heavy metals. These traces may be present in the form of oxides, carbonates, phosphates or silicates. The percent of ash in pine fibre is high i.e. 3.3%. The percent of water and alcohol

TABLE NO.4.6

Chemical composition of pine fibre

| Sample No. | Cellulose % | Lignin % | Extractive % | Moisture % | Ash % |
|---|---|---|---|---|---|
| 1. | 60.2 | 17.5 | 11.2 | 7.7 | 3.3 |
| 2. | 59.3 | 18.0 | 11.0 | 8.3 | 3.2 |
| 3. | 60.0 | 17.2 | 11.4 | 7.8 | 3.4 |
| 4. | 60.5 | 18.1 | 11.1 | 5.9 | 3.3 |
| 5. | 60.2 | 17.7 | 11.0 | 7.6 | 3.4 |
| Mean | 60.0 | 17.7 | 11.1 | 7.7 | 3.3 |

Lignin is found mainly in the woody core of the stem, but also in the epidermal and cortical cells of the plant. It is, however, often found in the walls of the cells of the ultimate fibres. It occurs chiefly towards the outside of the walls and particularly when the plants have been allowed to become over mature before havesting. Lignin is insoluble in water and does not swell when wetted. It is not removed in the retting process to which the stems are subjected for the extraction of the fibres. The percentage of lignin in the pine fibre is 17.7%. The method of Ellis and co-workers was followed for lignin determination.

4.3 Comparison of characteristics of pine fibres:

1. Comparison of the physical properties:

Literature on vegetable fibres, with the exception of jute, is limited, because jute is widely used among the vegetable fibres. The properties of vegetable fibres varies because the grades, maturity and retting methods differ considerably. Moreover, the different methods of measurements of vegetable fibres are applied so that comparison is difficult.

In physical properties, two types of fibre characteristics have been reported; one is for the filament or strand fibre and the other is ultimate fibre. In this report fibre generally means the former. Table No.4.7 shows the filament length, width and ultimate fibre length is generally shorter than the other fibres, fibre width being equal to that of Sisal fibre. The length and diameter of the ultimate fibres is comparable with other vegetable fibres. The ultimate length and diameter of pine fibre is less than that of jute.

Strength is one of the factors which is considered in evaluating the quality of fibre. Data available on strength

TABLE NO.4.7

Fibre dimensions of ultimate fibres of various vegetable fibres.

| Fibre | Fibre length (cm) | Fibre width (u) | Ultimate length (mm) | Ultimate diameter(u) |
|---|---|---|---|---|
| Flax | 20-140 | 40-620 | 4-66 | 12-76 |
| Jute | 150-360 | 30-140 | 0.8-5 | 10-25 |
| Hemp | 100-300 | - | 5-55 | 16-50 |
| Ramie | 10-180 | 60-904 | 60-250 | 16-126 |
| Abaca | 180-360 | 10-280 | 3-12 | 12-46 |
| Sisal | 75-120 | 10-460 | 1.5-4 | 16-32 |
| Pine | 9-12 | 140-160 | 11-16 | 27-36 |

M. Harris, Hand book of Textile Fibres, 1st Edition, (Harris Research Laboratories, Inc Washington DC 1954) P 115.

characteristics is scattered and different. In the present work no data is reported on the strength characteristics of ultimate fibres of pine fibre. Comparison with other vegetable fibres is not directly possible in some cases on account of differences in units etc. In Table No.4.8 strength characteristics of various vegetable fibres is given. The fineness (in denier) of sisal, henequene and abaca is given. The fineness of pine fibres is approximately the same as that of sisal fibre. The elongation (%) of pine fibre is almost the same as that of other vegetable fibres, but greater than that of jute and flax.

2. Comparison of chemical properties:

Chemical composition of a vegetable fibre is important in order to assess its suitability for end use. Moreover, the chemical constituents of bast and other vegetable fibre vary considerably. The relative proportion often varies remarkedly as a result of differences in crop, variety, state of maturity, soil, feltilizer treatment, climatic and enviromental conditions. Therefore, there are large variations in the chemical composition of the individual fibres as evident from Table 4.8. Cellulose is the main constitutent of a fibre and usually it varies from 60 to 70% in most of the vegetable fibres. Pine fibre contains 60% of cellulose. Other vegetable fibres such as jute, kenaf, abaca and kapok contain about 65% cellulose. Hemp, Sisal and Flax contain high percentages of cellulose i.e. 71 to 77%. All the vegetable fibres contain lignin which varies from 5 to 25%. Pine fibre contain 17.7% of lignin. As most of vegetable fibre is used as such i.e. without bleaching. Therefore, the presence of lignin will not effect the fibre quality. Water and alcohol extractives of the various vegetable fibres have also been given in Table 4.8. It

TABLE NO.4.8

Comparison of the physical properties of various vegetable fibres.

| Fibre | Fineness denier | Tenacity g/den. | Tensile strength PSI x 10-3 | Elongation % |
|---|---|---|---|---|
| Jute | 20 | 3 | 57 | 1.5 |
| Flax | 5 | 5 | 96 | 1.5 |
| Hemp | 6 | 4 | 76 | 2.0 |
| Ramie | 5 | 5 | 97 | 4.0 |
| Sisal | 290 | 4 | 74 | 3.0 |
| Henequre | 370 | 3 | 56 | 5.0 |
| Abaca | 190 | 5 | 93 | 3.0 |
| Pine | 242 | 0.24 | - | 3.1 |

H.F. Mark, N.G. Gaylord, Polymer Science and Technology Encyclopedia (Interscience Publishers, John Wiley & Sons, New York 1967.

TABLE NO.4.9

Comparison of chemical composition of various vegetable fibres.

| Fibre | Cellulose | Hemi-cellulse | Extractive & pectin | Ash | Moisture | Lignin & pentosan |
|---|---|---|---|---|---|---|
| Jute | 71.5-63.2 | 13.3-22.2 | 1.4-2.0 | 0.68 | 9.9 | 24.4-10.8 |
| Hemp | 77.5-74.3 | 10.1-17.9 | 2.7-4.0 | 0.82 | 8.8 | 3.7-9.3 |
| Sisal | 77.2-71.5 | 18.1-13.3 | 2.5-1.1 | 1.0 | 6.2 | 14.5-5.9 |
| Flax | 76.0-71.2 | 18.5 | 9.0-6.0 | 1.0 | 9.0 | 10.5-2.2 |
| Ramie | 91.0-76.2 | 14.5-8.0 | 6.2-6.0 | 0.12 | 6.4-5.0 | 5.6-0.7 |
| Kenaf | 65.7 | - | 1.9 | 1.0 | 9.8 | 21.6 |
| Abaca | 63.7 | - | 2.1 | 1.10 | 6.1 | 21.8 |
| Pine apple | 81.5 | - | 2.1 | 1.10 | 6.1 | 9.2 |
| Munn | 80.4 | - | 3.0 | 0.6 | 9.6 | 6.6 |
| Cypa Calophintia | 63.0 | 8.7 | 7.8 | 2.0 | 8.9 | 9.6 |
| Chindi | 80.0 | - | 3.1 | 0.64 | 10.2 | 9.4 |
| Capok | 64.0 | 23.0 | 2.3 | - | - | 13.0 |
| Pine fibre | 60.0 | - | 11.1 | 3.3 | 7.7 | 17.7 |

M. Harris, Hand book of Textile Fibres, First Edition (Harris Research Laboratories Inc, Washington D.C. 1954).

is evident that most of the vegetable fibres contain less extractive i.e. the percentage extractive varies from 2 to 6%. Pine fibre contains the highest percent of estractive i.e. 11%. Similarly the percent of ash is also highest i.e. 3.3% in the case of pine fibre, other vegetable fibres contain about 1% Ash.

## 5. SOFTENING, BLEACHING AND DYEING OF PINE FIBRE

### 5.1 Softening of pine fibres:

Vegetable fibres, other then cotton, consist of bundles or bunches of ultimate fibres and as such their diameter is usually higher than the cotton fibre. Most of these fibres are, therefore, coarse and harsh. Softness, is one of the essential requirements for a fibre to be used for textile purposes. To make these vegetable fibres soft, they are treated with softners or batching oils in the textile industries where they are used for fabrics or bags. There is a wide variation in the structure as well as in the physical and chemical properties of these fibres. In view of these variations different softners or batching oils are used for different fibres under varying conditions. The softening operation not only induces suppleness to the fibres but it also makes the fibres suitable for spinning into yarn. Elasticity or stretchability of vegetable fibres is of significant importance in the textile industry. Unelastic or brittle fibres may break in the spinning operations. Therefore, greater fibre losses occur in the processing of such fibres.

Pine fibres also consist of bundles of fibres and as such they are harsh. Therefore, like other vegetable fibres they are to be treated with softeners, emulsifying agents or oil before being utilized for the textile purposes.

Keeping in view the physical structure of these fibres, it was considered necessary to apply various softners, presently used in textile industries. Softening experiments were carried out on eight softeners, listed in Table No.5.1.

TABLE NO.5.1

Pine fibres treated with different soaftners under varying conditions.

| Type of Softener | Wt. of Sample(g) | Softener applied% | Treatment Time (hr.) | Temperature (°C) |
|---|---|---|---|---|
| Softex (A) | 2 | 8 | ½ hr. | 30 |
| " (B) | 2 | 10 | ½ hr. | 40 |
| Irgamine S.F.C extra. (A) | 2 | 5 | 1 hr. | 80 |
| " (B) | 2 | 10 | 1 hr. | 80 |
| Sapamine O.C. (A) | 2.5 | 3 | 1 hr. | 18 |
| " (B) | 2.5 | 5 | 2 hr. | 60 |
| Cirrasoft XL (A) | 2.5 | 14 | 3 hr. | Room Temp. |
| " (B) | 2.5 | 14 | 24 hr. | Room Temp. |
| Cirrasoft PN (A) | 2 | 3 | 2 hr. | 30 |
| ". (B) | 2 | 4 | 2 hr. | Room Temp. |
| Cirrasoft ACN (A) | 2 | 3 | 2 hr. | 40 |
| " (B) | 2 | 1.5 | 2 hr. | 40 |

Procedure: Each softener was tried on pine fibres under varying conditions of temperature, time and concentration of the softner. Each softener was tested on fifteen samples of fibres. The treatment conditions of each sample varied from one another. Then the subjective method, for selection of two best samples out of fifteen for each softener, was used. The selected samples of each softner have been given in Table No.5.1. The selected samples shown in the Table 5.1 are further subjected to the same procedure of selection i.e. twelve selected samples (two of each softner) were again examined for selection of the first three best samples in the same way as done in the previous case. The procedure adopted for selection of the samples was as follows:

The samples treated with softeners were placed on a table and total of fifty research workers, one by one, were asked to select the first three best samples by hand touching. As it was not possible that every one could select the same first three best samples, therefore we followed the marking system i.e. Ist selected sample was alloted six marks and four marks to second sample and 2 marks to 3rd sample. The selection procedure is indicated in Table 5.2. It clearly shows that out of fifty research workers 25 have selected cirrasoft ACN as the first best softner. Fifteen research workers have given it the second position and five workers were of the opinion of placing it in the 3rd place. So the total marks for Cirrasoft ACN (A) are 220, therefore, it was obvious that this was chosen as the Ist selected softner. Similarly, the total marks shown in the last column in the Table5.2. indicate the 2nd and 3rd selected softners. It is evident that the Cirrasoft XL (A) and Cirrasoft ACN (B) are in the 2nd and 3rd place respectively.

TABLE NO.5.2

Subjective Score for selection of the best softner.

| Softner | No.of persons in favour in 1st position. | No.of persons in favour of 2nd position | No. of persons in favour of 3rd position | Total Score |
|---|---|---|---|---|
| Cirrasoft ACN -(A) | 25 | 15 | 5 | 220 |
| Cirrasoft XL -(A) | 10 | 5 | 5 | 90 |
| Cirrasoft ACN -(B) | 5 | 5 | 15 | 80 |
| Sapamine OC(A) | 5 | 5 | 5 | 60 |
| Sapamine OC(B) | 0 | 10 | 5 | 50 |
| Irgamine SFC(A) Extra | 5 | - | 10 | 50 |
| " (B) | - | 10 | 5 | 50 |
| Total | 50 | 50 | 50 | 600 |

1st - 6 Score
2nd - 4 Score
3rd - 2 Score

The results of strength and elongation, of the selected samples of pine fibres treated with the six softners, have been tabulated in Table 5.3.

In table 5.1, the different softners used for softening of the fibres are listed. Two selected samples of each softner are given in this table. It is evident that the softaning effects of different softeners on the fibres are subject to the adjustment of time, temperature and concentration. The results show that the last two softners (Cirrasoft PN and ACN) are comparatively economical as lesser amounts of the softners are used and also the treatment is done either at room temperature or lower temperature. As regards the time factor it is slightly greater than the first three softners. It is also clear from the table that higher percentage of the Cirrasoft XL has been used but its softening effects are quite distinct. This can be easily understood from the table 5.2.

The most economical result has been shown by the Capamine OC where minimum of the time, temperature and concentration have been indicated but in the overall selection (as shown in table 5.2) it is placed in 5th position.

The usefulness of the softeners has become more distinct in table 5.2 where in the final selection the three have occupied the Ist, 2nd & 3rd position. Here the extent of softness of Cirrasoft ACN (A) is evident by the last column of the table. The selection position, of Cirrasoft XL (A) and cirrasoft ACN(B) at second and third position, is not very marked as the marks indicate. So from this table it is clear that the pine fibres are softened to the maximum extent by 3% Cirrasoft ACN (A) when treated for two hours at a temperature of 40 $C^{o}$.

TABLE NO.5.3

Strength and elongation properties of pine fibres samples treated with various softners.

| S.No. | Softner used | Strength gm.wt. | Elongation % |
|---|---|---|---|
| 1. | Cirrasoft ACN (A) | 56.3 | 5.8 |
| 2. | " (B) | 48.2 | 6.0 |
| 3. | Cirrasoft XL (A) | 55.7 | 6.8 |
| 4. | " (B) | 71.4 | 5.3 |
| 5. | Irgamin SFC (A) | 50.7 | 7.5 |
| 6. | " (B) | 55.8 | 4.8 |
| 7. | Sapamine OC (A) | 62.4 | 6.2 |
| 8. | " (B) | 68.6 | 4.3 |
| 9. | Cirrasoft PN (A) | 69.9 | 6.1 |
| 10. | " (B) | 65.8 | 6.0 |
| 11. | Softex (A) | 72.7 | 5.4 |
| 12. | " (B) | 78.9 | 5.1 |

In Table 3 the strength and elongation of the pine fibres, after softening, have been recorded. It is clear from the results that samples Cirrasoft PN result in comparatively higher strength and elongation, whereas in the case of the samples of Softex, although the strength is maximum but the elongation is lesser. The samples of Cirrasoft XL also contain improved strength and elongation properties.

5.2 Bleaching of pine fibres:

Bleaching is one of the most important operations in the processing of textile materials. The main objective of bleaching of fibres is to remove the admixtures which are associated with the fibres. These include most of the non-cellulosic materials, such as carbohydrates, pentosan and lignin. By eliminating these substances, the wettability of fibres is substantially increased and this assists the fibres in successful dyeing and printing operations. Wettability is one of the requirements for dyeing.

The bleaching of bast fibres is done with difficulty as they contain higher percentages of non-cellulosic materials. The presence of higher percentage of lignin is one of the difficulties in the process of bleaching. Due to the presence of [illegible] materials, most of the bast fibres, even after bleaching, contain slight tint of yellow colour.

The pine fibres, which are brown in colour, were treated with the oxidising and reducing bleaching agents to impart whiteness to the fibres. The following bleaching methods were carried out:

1. The brownish pine fibres were treated with sodium hypochlorite (2% active chlorine on the weight of fibres) at a temperature of 20 $C^{o}$ for 2 hours at a pH of 11. The pH was

controlled with 4g/L solid sodium hydroxide while the bath ratio was kept at 1:6. The bleached fibres were rinsed throughly with water and subsequently with 2% sodium thiosulphate. As the fibres after this treatment contained yellow colour, these were, subjected to reductive bleaching to further reduce the yellow colour. Thus these fibre were treated with 3 g/L sodium dithionite at 85 $C^{o}$ for 30 minutes. The fibres were then thoroughly rinsed with water. The degree of whiteness of the fibre has been greatly improved by the second bleaching method.

2. The bleaching of fibres was carried out with hydrogen peroxide. The bleaching bath was prepared by addition of 40 cc/L hydrogen peroxide (20 vol%), 1.2 g/L sodium hydroxide and 3 g/L sodium slicate. The fibres were treated with the prepared bleaching solution for two hours at the temperature of 80-85 $C^{o}$. The pH of the solution was kept at 11. The fibres were thoroughly rinsed after completion of the bleaching period. The fibres obtained were still having yellow colour. To fully bleach the fibres, these were treated with 3 g/L sodium dithionite at 85 $C^{o}$ for 30 minutes. The fibres were then rinsed thoroughly with water. After this bleaching method almost white fibres were obtained.

5.3 Dyeing of pine fibres:

The affinity of cellulose fibres for dyes is much influenced by the way in which the cellulose molecules are linked together in the fibres. Usually a cellulose fibre contains both crystalline and amorphous regions. The crystalline regions are those in which the cellulose molecules are arranged in an orderly manner. As in the crystalline regions the cellulose molecules are density packed together, therefore, in the dyeing process, the comparatively coarse dye molecules are fixed between the cellulose molecules with great

difficulty. Whereas in the amorphous region, where the cellulose molecules are arranged in disorderly fashion, the water and the dye molecules moves in and between the cellulose molecules readily. This situation is thus more convenient for satisfactory dye absorption.

The bleached pine fibres were dyed with direct dyes without any preliminary processes. The following procedure of dyeing was followed:- A cold dye bath was prepared by addition of 2% direct dye and 20% common salt. Then 5 g. of pine fibres, after wetting in water, were placed in the dye bath. The bath was stirred and the temperature was raised to 85-95 $C^{o}$ in 15 minutes. The temperature was maintained between 85-95 $C^{o}$ for one hour. After completion of the dyeing period, the sample was removed and rinsed thourghly with cold water.

Good shades of blue, red and orange were obtained by treating the pine fibres with direct blue, red and organge dyes. Other types of dyes such as acid, sulfur, azoic and vat may be used with varying degrees of satisfaction.

## CONCLUSIONS

1. Pine needles are abundantly available in North Western areas of Pakistan. These needles are a waste product of the forests and constitute a hinderance in the development of forests. The forest department collects these needles annually and subjects them to burning. The main disadvantages of these needles are that they are the cause of fire in the forests and check the growth of new plants. At the moment they are not put to any economic use and in fact their very removal will be a great service to the forest growers.

2. Among the vegetable fibres jute is the most widely used, and its world production is next to cotton. Pakistan is rich in vegetable fibres, but so far some of the vegetable fibres have been studied at the laboratory level only. Except jute, no vegetable fibre is being used commercially on any large scale. The main reason is that no data is available on the availability of these vegetable fibres. Moreover, these vegetable fibres need detailed study on their actual processing. Pine fibre has not been investigated on a pilot plant scale because no pilot plant facilities are available in the country. We will be able to utilize these fibre after carrying out due pilot plant studies on individual fibres.

3. Jute cultivation has recently been introduced in the country, especially in the Punjab and Sind, but with little success, because retting and other field operations of home produced jute are not yet being carried out properly by the farmers. The country's seven jute mills depend on the imported jute. The quality of

fibre depends on variety, agro-climatic conditions and the time of harvesting. But above every thing else, the quality depends on the efficiency of retting. If the fibre is properly retted, many of the defects found may be eliminated. The present inferior quality of home produced jute is due to the unawareness of the farmers in respect of retting and other preparatory processes. The quality of jute is lowered if care is not taken during retting process. The Government may start on elaborate programme to train the farmers in the retting process, so that in future the quality of jute is not affected due to the retting and associated processes.

4. Pine needle is very hard and therefore mechanical methods for the extraction of fibres cannot be of much use. The only method of extraction of fibre is the chemical process. Two types of chemical extraction methods have been developed. One is extraction of pine fibre with the help of commercial sodium hydroxide solution. In this process 4%, 2% solution of sodium hydroxide were applied at the boil for 30 and 60 minutes respectively and pine fibres were extracted. Longer period was required for keeping the needles at 35-40 $^{o}$C. In the second process, sodium carbonate is used. The needles were boiled for 30, 45 and 60 minutes in 4% sodium carbonate solution. Pine fibres were extracted by mild beating. Sodium carbonate method is superior to that of sodium hydroxide method as the yield is highest (76%) at 4% sodium carbonate solution and 30 minutes boiling and the strength of the fibre is also maximum at this condition.

5. Properties of pine fibre show that the fibre length is 15.5 cm and width is 154 microns. Ultimate fibre length is 13.3 mm and diameter 32.3 micron. The mean strength of fibre is 55.7gm

wt.and elongation 3.1%. The strength of pine fibre on wetting is decreased and elongation increased. The fineness is approximately the same as that of sisal fibre. However, the strength is a bit low than that of jute. Chemical composition of pine fibre shows that it contains 60% of cellulose, 17.7% lignin, 11.1% extractive and 3.3% ash. It is comparable with jute, kenaf, abaca and kapok which contain about 65% cellulose. Extractive matter and ash content of pine fibre are highest among the available vegetable fibres.

6. Pine fibres as such are harsh and are not suitable for conversion into yarn. The pine fibres can be conveniently softened by softeners which are economical and easily available in the market. These softners are already being used in the vegetable fibre industries for softening. A number of softeners were applied by varying time of treatment, temperature and concentration of the softeners. It was found that pine fibres are softened to the maximum extent by 3% Cirrasoft ACN when treated for two hours at a temperature of 40 $^{o}$C. The strength of fibre is not affected by the softener but the elongation (%) increased considerably.

7. The pine fibres can be bleached to almost whiteness by the ordinary bleaching procedures. Two methods were applied. The first method is by bleaching with sodium hypochlorite (2% active chlorine at a temperature of 20 $^{o}$C for two hours at a pH of 11). The fibres were further bleached with 2% sodium thiosulphate. After this the fibres still contain yellow colour and were, therefore, subjected to reductive bleaching. By this treatment the degree of whiteness is improved considerably. In the second method, the bleaching were carried out with hydrogen peroxide

(20 vol.%), 1.2g per litre sodium hydroxide and 3g per litre of sodium silicate. The fibres were treated for two hours at a temperature of 80-85 °C at 11 pH. To fully bleach the fibres, these were treated with 3g per litre sodium dithionate at 85 °C for 30 minutes.

8. Generally vegetable fibres are not bleached and dyed. But in some cases, as in the case of carpet yarn and for producing fancy effects, the yarn is dyed. The bleached pine fibres were dyed with direct dyes without any preliminary processes. Good shades of blue, red and orange were obtained by treating the pine fibres with direct, blue, red and orange dyes. Other types of dyes such as acid, sulphur, ozoic and vat may also be used.

9. Vegetable fibres of importance in the textile industry, other than cotton and flax, are jute, hemp, ramie, abaca and sisal. Jute and hemp are used extensively in package industry and in carpets and to a lesser extent in fabrics for draperies and upholstry. The principal use of ramie and flax fibres is in the production of various household fabrics. Abaca is extensively used in ropes and twine. The present studies reveal that pine fibre may be used for most of these purposes and especially in ropes and matting. It can also be blended with jute and other vegetable fibres for use in package industry.

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