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Extraction of sunnhemp fibre and its properties

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An effort has been made to extract sunnhemp fibre from stalks of sunnhemp plants which are considered as agriculture waste after harvesting seeds. Sunnhemp (*Crotalaria junceae*) stalks have been rippled and stacked for about 20-30 days to dry the stalks. The dried stalks are then pre-treated with different retting agents, viz. urea and compost culture. Fibre is extracted by water retting (tank) method. Physico-chemical properties, microstructure and solubility tests of extracted sunnhemp fibre are performed. It is revealed that the urea treated stalks produce higher fibre yield with lower retting period. Urea treated sunnhemp stalks give relatively longer, finer and stronger fibres than the compost culture treated and control stalks. Lower per cent of lignin is observed in compost culture treated stalks. Sunnhemp fibre constitutes oblong shaped cross-section with elongated lumen. Like cellulosic fibres, sunnhemp fibre is soluble in concentrated acids and is least affected by solvents.

Keywords: Compost culture, Physico-chemical properties, Retting, Rippling, Stacking, Sunnhemp fibre, Water retting method

1 Introduction

Sunnhemp is one of the leguminous plants grown in India as green manuring crop, because of its ability to produce large amount of biomass in 45 - 60 days. Moreover, it improves the soil properties, reduces soil erosion, conserves soil & water, and recycles plant nutrients. In India, sunnhemp is grown in 17.5 million hectares area with the production of 59.1 million bales, yielding 608 kg/hectares in the year 2012 -13 (agricultural statistics at a glance-2014). It is one of the important fibre yielding plants for small farmers since ages to produce strong ropes and twines which are needed for farming activities. Sunnhemp possesses fibre in its bast. Most of the bast fibres are cemented to the adjacent cells inside the stem with pectin and lignin, which can be extracted by retting process. Natural fibres are now considered as serious alternative to synthetic fibres for use in various fields[1].

Seed production of sunnhemp generates large amount of biomass (agriculture waste or fuel) after harvesting of sunnhemp seeds. Hence, an attempt has been made to extract fibre from stem portion of sunnhemp and assessed for its physico- chemical properties and microstructure.

2 Materials and Methods

2.1 Sourcing, Rippling and Stacking of Sunnhemp Plant

The stem of sunnhemp (*Crotalaria junceae*) grown in Institute of Organic Farming, University of Agricultural Sciences, Dharwad, Karnataka, was collected to extract fibre.

After harvesting of sunnhemp seeds, the stalks were collected and side branches were removed by the process called rippling to facilitate proper retting and decortication process. Subsequent to rippling, the stalks were stacked vertically in the field for about 20-30 days to dry and leaves to drop.

2.2 Pre-treatment of Fibre

Prior to retting process, sunnhemp stalks were pre-treated with different retting agents like urea and compost culture to reduce retting period and to get quality fibres.

Urea

Urea [$CO(NH_2)_2$], a nitrogen containing organic fertilizer, was procured from local market**.** Based on the earlier findings[2], 2% urea was sprayed on stalked bundles[2], which was then kept for retting in tanks filled with fresh water.

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Compost Culture

Microbial consortium *Phanerochaete chrysosporium* (crust fungi), maintained at Microbiology Lab, Institute of Organic Farming, University of Agricultural Sciences, Dharwad, was used for the study. It is a bacterial culture commonly used to decompose the compost at a faster rate. Two per cent compost culture was sprayed on stacked bundles, which was then kept for retting in cement tanks filled with fresh water.

2.3 Retting

Most bast fibres are cemented to the adjacent cell inside the stem with pectin, which can be extracted by retting process. Retting sometimes termed as degumming. Retting degrades the pectin rich bast and lignin in the middle lamella that is connected to the adjacent fibre cells, releasing individual fibres. Retting and extraction processes have a profound effect on the quality of fibre produced, and on the cost of fibre production. It affects the efficiency of manufacturing, the quality of the end products and their competitiveness in the market. Retting was carried out in cement tanks filled with clean water. The pre-treated stalks were dried and tied with big stones for proper immersion in water which otherwise causes uneven retting. Loosening of fibres was checked oftenly, and number of days for fibre extraction and per cent of extraction were recorded.

2.4 Fibre Quality Assessment

2.4.1 Microstructure

The microscopic appearance of a fibre is an important criterion that helps to know the basic structure of the fibre both longitudinal and cross sectional. This microstructure helps in identifying and explaining about the behaviour and properties of the fibre. The structure of sunnhemp fibre are captured under ×10 magnifications in Olympus projection microscope**.**

2.4.2 Physical Properties

Length (cm)

Each fibre was taken from the fibre mass and gently straightened over platform with tips of the finger. Length of the fibre was recorded in centimeter using a measuring scale. Hundred fibre samples were selected randomly, their lengths were measured and finally average value was calculated, since natural fibres posses wide variations.

Fineness

Sunnhemp fibre fineness(den) was assessed by using NIRJAFT Air-flow fineness tester which works on the principle of airflow method.

Bundle Strength

The bundle tenacity (g/tex) of fibres was measured under standard condition (65±2 % relative humidity and 27^{o}±2 % temperature) using NIRJAFT fibre bundle strength tester. Elongation per cent was also recorded.

2.4.3 Chemical Composition

Sunnhemp fibres were tested for the α-cellulose (%), hemicellulose (%), lignin (%), fat & wax (%), and ash (%) content at NIRJAFT, Kolkata. The methods employed for testing the above contents are ASTM:D1103-55T for α-cellulose (%); ASTM:D1103-55T and ASTM:D1104-56T for hemicelluloses; T-13 OS-54 for lignin; T-2040M-88 for fat and wax; and T-4130M-85 for ash.

2.4.4 Solubility Tests

This gives an indication of the extent to which the fibre polymers may react with common degrading agents such as acids, alkalies, solvents, laundering agents, atmospheric pollution, etc. AATCC Test Method-20-1990 was followed for analysing the solubility of sunnhemp fibre[3].

In Acids

Sulphuric acid (H_2SO_4), hydrochloric acid (HCl), nitric acid (HNO_3), acetic acid (CH_3COOH) and phosphoric acid (H_3PO_4) were selected using 25 %, 50 % and 100 % concentrations. At room temperature(20°), small sample of fibre (1 g) was placed in 50 mL beaker and covered with the test acid (1 ml of acid/10 mg of fibre). Fibre sample was stirred occa sionally for 30 min. Later treated fibre sample was washed, neutralized and dried. Further, the weight (fibre content) was calculated using the following formula:

$$Xi = \frac{G\text{-}Hi}{G}$$

where Xi is the content of sunhemp fibre (%); G, the weight of clean, dry sample; and Hi, the weight of dried residue after treatment.

In Alkalies

Sodium hydroxide (NaOH) and sodium carbonate (Na_2CO_3) were selected using 10 %, 20 % and 30 %

concentrations. At room temperature (20°), a small sample of fibre (1 g) was placed in 50 mL beaker and covered with the test acid (1 mL of acid/10 mg of fibre). Fibre sample was stirred occasionally for 30 min. Later treated fibre samples were washed and dried. Further, the weight (fibre content) was calculated using the above- mentioned formula, as used in case of acid treatment.

In Solvents

Methanol (CH_4O) and acetone $(CH_3)_2CO$ were selected using 100 % concentration. At room temperature (20°), a small sample of fibre (1 g) was placed in 50mL beaker and covered with the test acid (1 mL of acid/10 mg of fibre). Fibre sample was stirred occasionally for 30 min. Later, treated fibre sample was washed and dried. Further, the weight (fibre content) was calculated using the above-mentioned formula, as used in case of acid treatment.

3 Results and Discussion

3.1 Ancilliary Characters of Sunnhemp Plant

It is observed that the mean height of sunnhemp plant is 155.59 cm at the time of harvesting and plant posses 5-7 branches. Number of capsules observed in sunnhemp plant is found 35-55. Further, stem girth at the top and bottom of sunnhemp plant is found to be 8.0 mm and 14.5 mm respectively.

3.2 Retting Period and Extraction Per cent of Sunnhemp Fibre

It is observed that the retting is faster in urea treated (UT) sunnhemp stalks (7 days) as compared to that in compost culture treated (CT) stalks (8 days) and control (9 days). This may be due to the fact that urea enhances the wetting action of water and the growth of microbes in water[2]

It is noted that fibre extraction percent of UT stalks of sunnhemp is greater (3.7 %) than those of CT stalks (3.62 %) and control (2 %). This indicates that retting treatment has remarkable effect on extraction per cent. Similarly Ghorpade and Balasubramanya[4] stated that microbial retting of banana pseudo stem has significant difference in extraction per cent as compared to control.

3.3 Microstructure

It is observed from the Fig. 1 that sunnhemp fibre bundle consists of several elementary fibres or ultimate cells. Longitudinal structure exhibites some lines throughout the length. Each cell depicts polygonal to oblong shaped cross-section with elongated lumen. The size of lumen and shape of cell varies within the fibre bundle. Similar cross-section is also illustrated in study conducted by Thygensen *et al*.[5] A study on bast fibre reveales that both leaf and bast fibres are multi-cellular with very small individual cells bonded together[6]. Further, longitudinal section reveals that cell to cell distance varies all along the length[7].

3.4 Physical Properties of Sunnhemp Fibres

The physical properties of sunnhemp fibres extracted by different retting treatments have been studied. Fibre obtained from urea treated stalks are longer (38.25 cm), finer (10.7 den) and stronger (12.5 g/tex) compared to fibre obtained from compost culture treated stalks wherein length, fineness and bundle strength are 32.34 cm, 10.8 den and 11.1 g/tex respectively. These results are at par with the study conducted by Dhanalaxmi and Vastrad,[2] who concluded that urea treated mesta stalks produced longer and finer fibre compared to biological retting. This may be due to its intrinsic property of enhancing

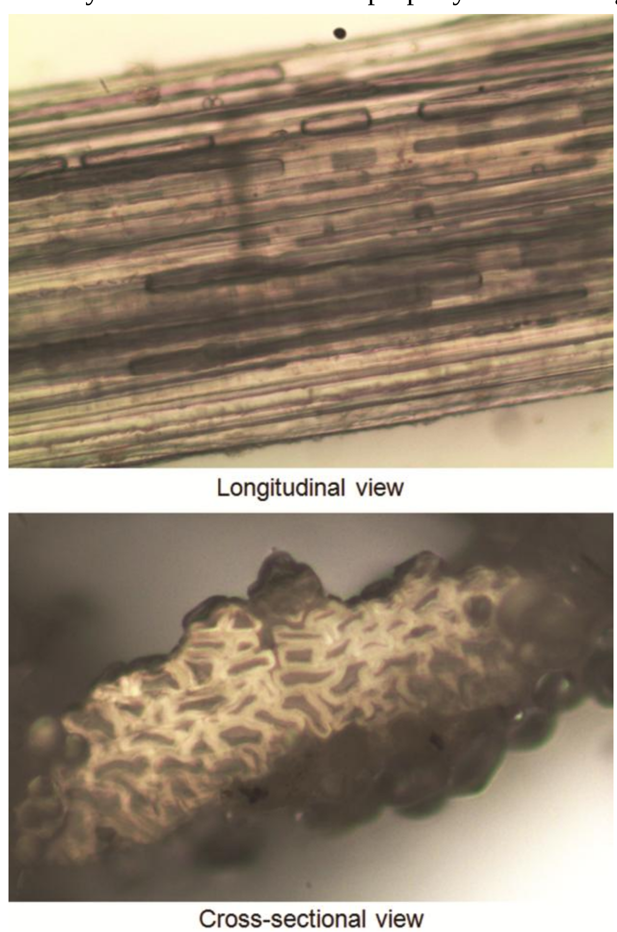

Fig.1 — Microstructure of sunnhemp fibre

wetting action and microbial growth. It is also observed that urea treated sunnhemp stalks produce stronger fibres than compost culture treated and control which may be due to the fact that low concentration of urea (2 %) does not affect strength. However, control stalks possessed 30.05 cm length, 10.82 den fineness and 9.8 g/tex bundle strength. Further, elongation per cent values of fibre obtained from CT stalks and control stalks are same (0.4 %) which is found to be slightly lesser than the fibre obtained from UT stalks (0.5 %).

3.5 Chemical Composition

Chemical composition of sunnhemp fibres with different retting treatments reveals that α-cellulose content of control fibres is higher (70.1 %) followed by fibre obtained from UT (69.8 %) and CT stalks (68.5 %). Similarly, hemicellulose content of control fibre is 12.2 % which is found to be higher than those of fibres obtained from CT stalks (11.1 %) and UT stalks (9.0 %).

Lignin content in control and fibre obtained from UT stalks is 4.3% which is slightly higher than the fibre obtained from CT stalks (4.2 %). Further, fat and wax of fibre obtained from control, UT and CT stalks are 0.58, 0.54 and 0.53 % respectively. However, 0.3 % ash is noticed in fibre obtained from control and CT stalks, whereas fibre obtained from UT stalks constitutes 0.29 % ash. Cellulose is the main component of natural fibres and also is organized into fibrils, which are surrounded by a matrix of lignin and hemicellulose, which contains three hydroxyl (OH) groups[8].

3.6 Solubility of Sunnhemp Fibre in Different Chemicals

In Acids

It is observed that sunnhemp fibres are dissolved completely in 100% sulphuric acid, whereas 70 % of sunnhemp fibre is dissolved in 50 % concentration of H_2SO_4. However, only 3.9 of sunnhemp fibre is dissolved in 25 % H_2SO_4. Solubility per cent is higher (12.5%) in concentrated HCl (100%) followed by 50 % HCl (2.8%) and 25 % HCl (2.6%).

Sunnhemp fibres of 7.2% are dissolved in 100% HNO_3 followed by 50 % HNO_3 (6.9%). However, solubility of sunnhemp is observed to be 1.5 % in 25 % HNO_3

Solubility of sunnhemp fibre in acetic acid is found 5 % in 100 % concentrated acetic acid (100 % CH_3COOH), 4.6 % in 50 % acetic acid and 3.1 % in 25 % acetic acid.

Similar trend is observed in phosphoric acid, where 6.3 % of sunnhemp fibre is soluble in 100 % phosphoric acid. However, solubility of sunnhemp fibre in 50 % phosphoric acid is found 3.6 %. Further, 3 % sunnhemp fibre is found to be soluble in 25 % phosphoric acid. Concentrated acids dissolve cellulose and forms cellulose hydrate[9]. Bast fibres are also cellulosic in nature and hence cause disintegration of fibres at higher concentration of acids.

In Alkalies

It is noticed that solubility of sunnhemp fibre is 6.1 , 5.7 and 4.3 % at 30, 20, and 10 % NaOH respectively. Similarly, solubility of sunnhemp is higher (1.3%) at higher alkali concentration (30%) followed by 20 % Na_2CO_3 (1.1% fibre solubility) and 10 % Na_2CO_3 (0.5% fibre solubility). Cellulosic fibres are resistant to alkalies and are relatively unaffected by normal laundering agents. The resistance is attributed to the lack of attraction between the polymers and alkalis[3]. Alkalies swell fibre and purify the cellulose which imparts permeability to the fibre. For smaller concentration of alkali, the diameter of hydrated ions is too large so that it cannot penetrate in to the fibre structure.

In Solvents

It is found that solubility (%) of sunnhemp is more in methanol (2.2% solubility) compared to acetone (2% solubility). Sunnhemp fibre is least affected by solvents. This may be due to the fact that cellulosic fibre has high resistance to solvents[9]. Similar trend is also observed by Pandey and Dayal[10].

4 Conclusion

4.1 Urea treated stalks produce higher fibre yield (extraction per cent) with lower retting period. Urea treated sunnhemp stalks show relatively longer, finer and stronger fibres than compost culture treated and control stalks. Sunnhemp fibre constitutes oblong shaped cross section with elongated lumen.

4.2 Lower per cent values of cellulose and lignin are recorded in fibre obtained from CT sunnhemp stalks. Relatively lower per cent of hemicellulose, fat and wax, and ash contents are observed in fibre extracted from urea treated sunnhemp stalks.

4.3 Solubility per cent of sunnhemp fibre is found maximum in concentrated sulphuric acid, hydrochloric acid and nitric acid as compared to that in acetic acid and phosphorous acid. Further, it is found unaffected by alklies up to a certain concentration, however higher concentration of NaOH affects greater per cent than that of Na_2CO_3. Solvents do not have much affected.

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