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RESEARCH PAPER

Effect of retting methods on fibre quality characteristics of different varieties of flax

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Abstract: Stalks of flax varieties *viz*., DLV1, Himalini and No55 x NL115 were collected from AICRP, MULLARP Scheme, University of Agricultural Sciences, Dharwad for extraction of fibres in water, chemical and enzymatic retting methods. The extracted fibres were assessed for quantity and quality parameters *viz*., fibre yield, fibre length, microscopic appearance and fibre solubility in different chemicals. The results of the study revealed that, urea retting method yielded higher percentage of fibres, enzyme retted fibres gave longer length of the fibre. The fibre yield was higher in Himalini variety while the fibre length was higher in DLV 1 and No55 x NL115 variety. Flax fibres were soluble in 50 per cent sulphuric acid concentrated whereas insoluble in alkalies and solvents.

Key words: Fibre yield, Fibre length, Microscopic appearance, Retting methods, Solubility

Introduction

Natural plant fibres have been successful due to their positive impact on our ecological balance. Various plant fibres can be used for number of textile and technical applications as environmental awareness is most important concern nowadays. Fibres are extracted either from the outer part of the plant stem *i.e.*, the bast (*viz*., Jute, Kenaf, Flax, Ramie, Hemp *etc*), Leaf (*viz*., Sisal, Pineapple), Fruit (Coconut) or Seeds (Cotton).

Linseed or seed flax (*Linum usitatissimum*) belongs to the family Lineace, order Geraniales, series Disciflorae, subclass poly petalae and tribe eulineae. The common names are Lin, Linum, Linen, Agase. Flax is an erect annual plant growing upto 1.2 m tall, with slender stems. The leaves are glaucous green, slender lanceolate, 20-40 mm long and 3 mm broad. The flowers are pure pale blue, 15-25 mm diameter, with five petals; they can also be bright red. The fruit is round, dry capsules 5-9 mm in diameter, containing several glossy brown seeds of 4-7 mm long (Rastogi, 2009).

Flax is an annual herbaceous plant, with one dominant typical stem that branches at the top and grows to a height of 1 to 3 feet. The taller and straight flax varieties developed for fibre production are used by the textile industry is as speciality fibres to create a distinctive fabric with unique characteristics.

The main constituent of flax fibres are cellulose, hemicelluloses, lignin and pectin. A small percentage of wax, oil and structural water are also found. Flax fibre is a biodegradable natural composite material which exhibits good specific mechanical properties. These fibres have a long history of being utilized in clothing by the textile apparel industry due to their comfort level. Cellulose is the major component in flax fibre and ranges from 70-80 per cent.

Many varieties of flax are developed in University of Agricultural Sciences, Dharwad either for seed or for fibre purpose. In order to find out the varieties suitable for fibre production , this experiment is carried out with the objective to compare the best method of flax fibre extraction and to study the quality characteristics of extracted fibres

Material and methods

Selection of plant source

Three varieties of Flax *viz.*, DLV 1, Himalini and NL 55 x NL 115 developed at AICRP on MULLARP, University of Agricultural Sciences, Dharwad were selected for the study depending on the plant height. The experiment was conducted during the year 2019-20.

Ancillary properties of flax plant

Ancillary properties of selected flax plants was carried out to know the properties *viz.*, plant height (cm), number of capsules /plant, number of branches/plant and stem girth (cm)

Fibre extraction method

Retting was done for extraction of fibre from stalk. Bundles of plants were steeped in water for certain period depending on the type of retting to decompose, thus loosening gum that binds fibre to stem. This decomposition is called retting (Corbman, 1983).

The flax stalks were subjected to three types of retting methods *viz.*, water retting, chemical retting and enzymatic retting.

Water retting

The dried stalks of flax plant were steeped in tanks filled with fresh water for 4-6 days and then the fibres were removed by beating, then washed in running water and shade dried (Dhanalaxmi and Vastrad, 2013).

Chemical retting

Two per cent urea was sprayed on bundles of flax stalk and allowed for retting in tanks filled with fresh water and then the fibres were removed, washed and shade dried (Dhanalaxmi and Vastrad, 2013).

Enzymatic retting

Microbial consortium *Phenerochate crysosporium* maintained at Microbiology Laboratory, Institute of Organic Farming, UAS, Dharwad was used for the present study for retting of flax stalks. Two per cent of culture was sprayed on bundles of flax stalk and allowed for retting in tanks filled with fresh water and then the fibres were removed, washed and shade dried.

Scutching and cleaning

The retted mass of stalk was examined for optimum loosening manually and number of days was recorded. Process of extracting retted mass manually from slive is known as scutching. The fibre was finely cleaned under running water, brushed to remove adhered vegetative matter and dried under shade.

Pretreatment of flax fibre

The extracted fibres were further subjected for pre treatment *i.e*., scouring and bleaching to remove impurities from raw fibre and make fibre soft and white which can be easily blended with cotton for further spinning process.

a. Scouring

Fibres were pre soaked in 0.5 per cent solution of Turkey red oil (wetting agent) for 20 minutes. Then the fibres added to alkali solution containing NaOH of 2% with MLR 1:30 and added to the solvent and boiled for 45 minutes with subsequent stirring. Further, the scoured samples were later treated with 0.5 per cent acetic acid for neutralization. The fibres were then thoroughly rinsed in running water, squeezed to remove excess water and shade dried on flat surface (Brindha *et al.,* 2012).

b. Bleaching

Known quantity of scoured fibre were soaked in 0.5 per cent Turkey red oil for 20 minutes. The fibres were then transferred to the bleaching solution containing Hydrogen peroxide (2%) and 1-2% sodium silicate with the material to liquor ratio of 1:30 and then boiled for 60 min. Then the fibre samples were rinsed thoroughly under running water and dried under shade (Chattopadhyay *et al.,* 2012).

Assessment of fibre quality characteristics

Extracted fibres were assessed for fibre yield, microstructure, chemical composition, fibre length and solubility performance in different chemicals.

Table 1. Ancillary characters of flax plant at harvesting stage

Ancillary characters	Flax varieties			Mean
	DLV 1	Himalini	No55xNL115	
Plant height (cm)	47.5±5	54.4± 6	52.5±4	51.4±5
No. of branches/plant	6-8	5-9	6-10	6-9
No of capsules per plant	30-50	35-44	40-50	35-48
Stem girth (mm)	11-15	10-16	10-13	10-15

Results and discussion

Ancillary characteristics of flax plant

It is observed from Table .1 that, irrespective of flax varieties the mean height of flax plant was 51.4±5 cm at the time of harvesting. On an average 6-9 branches were observed with 35-48 capsules per plant with stem girth of 10 to 15 mm.

Retting period, method and extraction percentage of different flax varieties

The different varieties of flax stalks were retted with three different methods *viz.*, water, urea and enzyme retting at different period intervals during summer season (March to May). It was found that, DLV 1 and Himalini varieties took 7 days for loosening of fibre in water retting method whereas, No 55 x NL 115 variety took 8 days for loosening. In urea and enzyme retting methods, DLV 1 and Himalini took 5 days for loosening of fibres where as No55 x NL 115 took 4-5 days.

In case of urea and enzyme retting methods, the Himalini variety yielded higher percentage of fibre (11.07% and 10.2%) followed by No 55 x NL 115(10.51% and 9.72%) variety and DLV 1(7.39% and 6.75%). In general it can be concluded that, urea retting yielded higher percentage of fibre (11.07%) followed by enzyme retting (10.2%).

The process of fibre extraction from stems (straw) is called retting. There are few retting methods and apart from purely mechanical, the general rule is to remove and/or degrade pectin, and to some extent hemicelluloses (Dhanalaxmi and Vastrad, 2013). It is observed from Table 2 that, the yield of fibre produced by urea retting in shorter period of time (4-5 days) was higher in all the varieties *viz*., DLV 1, Himalini and No55 x NL 115 as compared to Enzymatic and Water retting methods. It may be due to the reason that, urea enhances the wetting action of water and the growth of microbes in water (Vanishree and Mahale, 2016) and however enzymes during retting helps to degrade pectin around flax fibres resulting in separation of flax plant fibres.

Microscopic appearance

The microscopic appearance of a fibre is an important criteria that helps to know the basic structure of the fibre both longitudinal and cross sectional view (Figure1). The

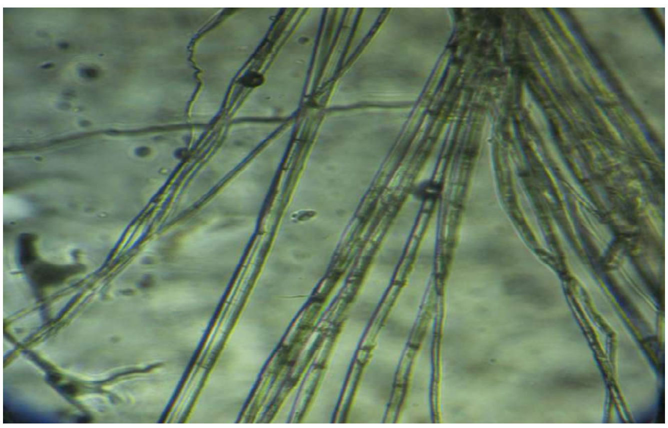

Fig. 1. Longitudinal view of flax fibre

Table 2. Fibre yield (g/kg stalk) obtained from different retting methods

Flax Varieties	Water retting		Urea retting		Enzyme retting	
	Retting period (days)	Fibre Yield (g/kg stalk)	Retting period (days)	Fibre Yield (g/kg stalk)	Retting period (days)	Fibre Yield (g/kg stalk)
DLV 1	7	56.12(5.61)	5	73.96(7.39)	5-6	67.5(6.75)
Himalini	7	87.78(8.78)	5	110.72(11.07)	5-6	104.0(10.2)
No 55 x NL 115	8	80.8(8.08)	4-5	105.12(10.51)	4-5	97.2(9.72)

(Figures in parenthesis indicates percentage)

microstructure helps in identifying and explaining about the behaviour and properties of fibre. It was observed that, flax fibre showed cross marking and nodes in longitudinal view, while polygonal structure in cross sectional view.

Longitudinal structure showed cross marking or nodes at intervals and the presence of some impurities on the surface of the fibre is observed and ultimate cells are cemented in non-cellulosic compounds. Further, cross section structure showed polygonal shape that varied notably from irregular to circular. The irregular cross-sectional shapes and uneven longitudinal surface of fibres such as cotton, flax and wool makes them dull or non-lustrous fibres; however, flax with its polygonal or many sided cross-section and relatively flat, regular longitudinal surface tends to display a slight lustre (Gohl and Vilensky, 1987).

Chemical composition of flax fibre

Table 3 explains the chemical composition of raw, scoured and bleached flax fibre. It is observed that, there was increase in cellulose (85.2%) and hemicellulose (11.5%) content and decrease in lignin content (1.92%) after scouring and bleaching flax fibre when compared to raw fibre. The traces of ash and wax content were also found in bleached, scoured and raw fibres.

Flax fibres are complex assembly of different polymers, polysaccharides such as cellulose, hemicelluloses and pectin, and the phenolic polymer of lignin. The amount of fibre polymers, proportions between them and monomer composition depend on many factors and vary among species and even within a single cell. This also explains the diversity of different fibre properties. Cellulose, a major structural polysaccharide in plants, is the most abundant organic compound. Table 3 explains the chemical composition of flax fibre. It is observed that, the highest cellulose content was obtained in flax fibre. The mechanical properties of fibres are largely related to the amount of cellulose, which is closely associated with crystallinity of fibre and the micro-fibril angle with respect to the main fibre axis (Sreekala *et al.*, 1997). However, the scouring and bleaching increased the cellulose and hemicellulose content in the fibre and decreased the lignin content. Cellulose provides strength and rigidity to the fibres and hence as cellulose content increases fibre strength increases. Unlike cellulose, hemicellulose molecules have short side chain which are acidic in nature and soluble in alkalies.

Table 3. Chemical composition of Flax fibre

Chemical composition	Flax fibre		
	Raw	Scoured	Bleached
Cellulose (%)	81.7	84.6	85.2
Hemicellulose (%)	9.51	10.63	11.5
Lignin (%)	7.32	3.14	1.92
Ash (%)	1.22	0.81	0.78
Fat (Waxes) (%)	0.25	0.72	0.6

Flax fibre length

It is observed from Table 4 that, irrespective of flax varieties the mean length of the enzyme retted flax stalk recorded significantly longer fibres (27.4 cm) compared to urea retted (27.0 cm) and water retted (25.6 cm) fibres. Among the flax varieties, irrespective of extraction method it was observed that, DLV 1 recorded significantly longer fibre (28.2 cm) followed by Himalini (26.8 cm) and No 55x NL 115 (24.9 cm) varieties.

Interaction effect of flax varieties and method of extraction on fibre length found to be nonsignificant. However, enzyme retted DLV 1 fibre recorded higher fibre length (28.6 cm).

Enzymatic retting of flax stalk produced longer length of fibre (Table 4) as compared to water and urea retting process. It may be due to its intrinsic property of enhancing wetting action and microbial growth and also may be due to less mechanical damage while retting.

Solubility percentage of flax fibre

Table 5 revealed the effect of chemical reagents on solubility of flax fibre. It was observed that, flax fibres were completely soluble in 50 per cent H_2SO_4 concentration whereas, 15 per cent of fibres were soluble in 25 per cent sulphuric acid. However, only 10-15 per cent of fibres were soluble in 25, 50 and 100 per cent HCl. Further, in case of alkalies (10 and 20%), flax fibres were insoluble in Sodium hydroxide and Sodium carbonate but partially soluble (10%) in 30 per cent concentration of alkalies. On the other hand, flax fibres were insoluble in both Methanol and Acetone solvents.

From Table 5 it is observed that, flax fibres were completely soluble in 50 per cent Sulphuric acid concentration and partially soluble in 100 per cent HCl (10%).

Cellulose fibres are resistant to alkalies and are relatively unaffected by normal laundering agents. The resistance is

Table 4. Fibre length (cm) of flax varieties

Method of extraction	Fibre length (cm)			Mean
	DLV 1	Himalini	No55x NL115	
Water retting	27.6	26.1	23.2	25.6
Urea retting	28.5	26.8	25.7	27.0
Enzyme retting	28.6	27.6	25.9	27.4
Variety Mean	28.2	27.4	24.9	
	S. Em±	C.D @5%	C.V %	
A (Flax Varieties)	0.11	0.42	1.2	
B (Retting method)	0.32	0.97	3.54	
AXB	0.55	NS		

Table 5. Effect of chemical Reagents on solubility (%) of Flax fibre

Fibres	Acids						Alkalies						Solvents	
	Sulphuric Acid (H_2SO_4) (%)			Hydrochloric Acid (HCl) (%)			Sodium Hydroxide (NaOH) (%)			Sodium Carbonate (Na_2CO_3) (%)			Methanol (CH_4O) (%)	Acetone (C_3H_6O) (%)
	25	50	100	25	50	100	10	20	30	10	20	30	100 %	100 %
Flax	15	S	S	10	12	15	I	I	10	I	I	8	I	I

S: Soluble I: Insoluble

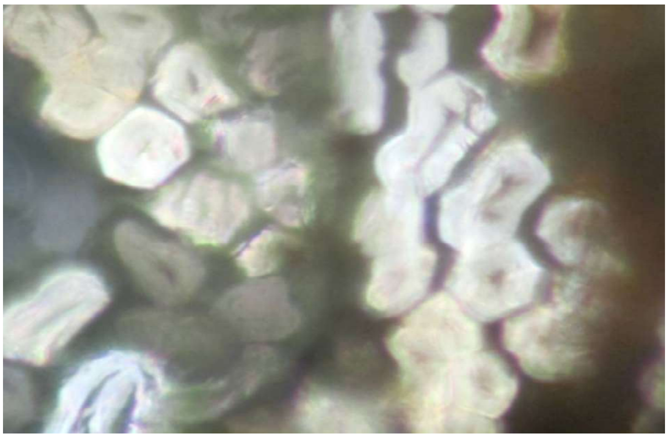

Fig 2. Cross sectional view of flax fibre

attributed to the lack of attraction between the polymer and alkalies (Gohl and Vilensky, 1987). Normal laundering will result in alkaline hydrolysis of the waxes and gums bonding the cells forming the flax fibre together. This results in cell ends projecting above the surface of the linen textile material, and is referred to as cottonising of linen. Severe cottonising will cause a noticeable weakening of the linen textile material. It is observed from Table 5 that, the flax fibre was insoluble in 10 and 20 per cent concentration of alkalies, only 10 per cent of fibres were dissolved in 30 per cent alkali concentration.

The flax fibres were insoluble in solvents as presented in Table 5. This may be due to the reason that, cellulosic fibres are resistant to solvents (Mishra, 2010). Similar observations were made by Pandey and Dayal (2008) and stated that, the flax fibre is resistant to the action of acetic acid, sodium hydroxide, cyclohexanone, metacrisol, carbon tetrachloride and acetone. Fibre disintegrates in hot concentrated H_2SO_4 and HNO_3.

Conclusion

Irrespective of flax varieties, urea retting method yielded higher percentage of fibres, whereas longer length of the fibres were obtained by enzyme retting method compared to urea and water retting methods. Among the flax varieties, irrespective of extraction methods, DLV 1 gave longer fibre than Himalini and No 55x NL 115 varieties.

The data generated from the present research depicts that, the flax fibre yield is more in Himalini and DLV 1 when extracted by urea retting and enzyme retting methods. However, with respect to fibre length, DLV 1 and Himalini varieties gave longer length of fibres. Thus, Himalini and DLV 1 varieties can be utilized for flax fibre production with either urea or enzyme retting. The extracted fibres can be utilized from cottage industries to technical textile industries.

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