Evaluation of retting methods on the properties of *dhaincha* fibres

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ABSTRACT : Fibres from *Sesbania aculeata (dhaincha)* plant were extracted by biological and chemical retting. In biological retting both the stagnant and running water methods were utilized whereas in chemical retting, two methods were used viz., the combination of EDTA and NaOH; pretreatment of HCl followed by NaOH. The physical properties such as tenacity and fineness of extracted fibres were observed and compared for different retting methods. The chemical retted fibres produced fibres of relatively lower tensile strength as compared to biological retted fibres. The 15 days stagnant water retting fibres exhibited highest tenacity of 5.43 g/ denier followed by running water and combination of EDTA and NaOH with tenacity of 5.11 g/ denier and 3.54 g/ denier respectively. Further it was observed that the combination of EDTA and NaOH showed good fineness value (27.2 denier) followed by fibres retted with HCl and NaOH with fineness value of 29.4 denier. The fibres obtained after 15 days of stagnant water retting were coarser as compared to other retting methods. Overall, the fibres obtained after 15 days of stagnant water retting exhibited tenacity whereas the combination of EDTA and NaOH revealed good fineness value.

Key words: Biological retting, chemical retting, *Dhaincha*, extraction, fineness, natural fibre, *Sesbania aculeata,* tenacity

Plant fibers have been used by humans since ancient times for everyday need and their uses have expanded even to technical applications too. With the development of chemical technology, these fibres have been replaced by man-made fibers, mostly originating from fossil resources and have aggravated pollution to alarming level. Reduced availability of resources at affordable cost and the growing interest in sustainability have led to a renewed interest in natural materials in current situation. The celebration of year 2005 as international year of natural fibres further strengthened the concept of sustainability. Most common source of plant fibres is stem and the fibres are known as bast fibres. The bast fibres are also known as hard fibres and have characteristics namely good strength, length and low density that made them suitable fibre for building and construction as a form of geo-textiles, fibre board materials, insulation materials, reinforcement, filler, light-weight concrete and bricks. These fibres need a renaissance, not only for old industrial products but also for manufacturing new types of products, required in the various fields of technical textiles.

However to increase their uses and achieve good quality bast fibres, the appropriate extraction and processing methods are required. Now-a-day, various extraction methods i.e. water retting, chemical retting, dew retting and enzymatic retting are used. These methods yields fibres with varied set of physical properties, environment-friendliness and cost effectiveness. The use of dew and enzyme for retting are limited to specific regions because of the availability of dew during the few months of the year and the cost of enzyme that is too expensive whereas, water and chemical retting are commonly used method for natural fibres owing to the availability of sufficient water and low cost chemicals. Further, studies revealed that water retting yield good quality uniform fibres and the chemical methods makes retting to complete in an hour or two that normally takes 14-28 days in case of water retting. India is primarily an agricultural country. The favorable climatic condition facilitates the growth of wide variety of plants. The underutilized fibres are available in abundance and till now, have not received any economic importance owing to their harshness, brittleness and coarseness of fibres. Amongst numerous underutilized natural fibre sources in Uttarakhand state, one such potential source of textile fibres is *dhaincha (Sesbania aculeata),* popular as a green manure, useful in nitrogen fixation and wind barriers (Negi *et al*., 2016). The method of retting i.e., the retting condition and duration employs for the extraction of fibres strongly influences the quality and quantity of extracted fibres. Therefore, the study was planned to observe the effect of different retting methods on the properties of *Sesbania aculeata* fibres.

MATERIALS AND METHODS

The materials and methods used in the study are mentioned and described below:

Raw Materials Collection

The plantation of crop was done in the month of May in a farm at Ramnagar, Uttarakhand. The harvesting of plant, for stalk collection was done in the month of September. The branches, leaves and pods were removed from the fresh *dhaincha* plant and the bark was peeled out from the stems and allowed to dry. The dried bark was used to study the effect of different retting methods on the properties of *dhaincha* fibres.

Retting

According to Umoru *et al*., 2014 five types of retting processes namely; dew, flowing water, tank (stagnant), enzymatic and chemical retting are utilized for the extraction of bast fibres. Out of these, only two retting systems namely water and chemical were utilized to study the effect of extraction of *Sesbania aculeata (dhaincha)* fibres on the physical properties. The retting was done in the month of September at a temperature and relative humidity in range of 30^0C - 37^0C and 75 percent - 85 percent, respectively.

Water retting

This method of retting is one type of biological retting, in which the microbes present or developed on dried bark underneath water. These decomposed the vegetative matter and helped to dissolve the cementing matter i.e., lignin that lead to separation of fibres. The retting of fibre was done both in stagnant and running water for same duration.

In this method, the dried ribbons of *dhaincha* were submerged in water filled plastic tubs and in a water stream for 15 days (Negi and Rani, 2016).

Chemical retting

In this method, the fibres are usually treated with different acid and alkalis for 1 to 3 hours at 60-100^0C. For the present study, chemical retting procedure was selected on the basis of review of literature. The methods were slightly modified with the amount of alkalis that was changed from 1% to 5% to observe the effect of chemicals on fibres.

Two chemical methods were used, in first recipe the fibres were pretreated with 0.5% HCl solution and then treated with 5% NaOH in separate beakers while in second recipe the combination of 0.05% EDTA with 5% NaOH was utilized for treatment of fibres. The details of recipes are given in Table1.

On completion of retting period, the biological retted fibres (stagnant and running water) were covered with slimy material developed by the activity of microorganism. The slimy material was removed by thorough washing under running water. The fibres obtained from chemical retting were neutralized with 2 percent acetic acid followed by washing with water.

The fibres obtained from the water and chemical retting were separated by hand while washing and laid on flat surface for drying in the open air under shade. The fibres of *Sesbania aculeata (dhaincha)* were combed manually using combing brush to open the fibre bundles, and to remove remnant vegetative matter adhered to the surface. The fibres obtained after retting were assessed on the basis of physical properties and SEM analysis.

Assessment of physical properties

The affect of retting method on physical properties of *dhaincha* fibres was assessed on the basis of physical properties such as tenacity and fineness. One way ANOVA was applied using statistical software "SPSS 20".

SEM Analysis

The effect of different retting methods on *Sesbania aculeata* fibres was also investigated by Scanning Electron Microscope (SEM). It was done on FEI Quanta 200, at an accelerating voltage of 15KV with the

Table 1: Recipe for chemical retting of *Sesbania aculeata (dhaincha)* fibres

S. No.	Pretreatment	Retting	Rinsing	After treatment	References
1.	0.5% HCl 30 min @ 400 C	5% NaOH 60 min @ 100^0 C	10 min @ 60^0 C	2% CH_3COOH 10 min @ 60^0 C	Das Gumpta *et al.,* 1976
2.	-	5% NaOH, 0.05% EDTA 60 min @ 100^0 C	10 min @ 60^0 C	2% CH_3COOH 10 min@ 60^0 C	Kundu *et al.,* 1996

resolution of 100 microns and magnification ranging from 1000- 1200X. Prior to examination the fibre specimens were glued on separate aluminium stubs and sputter coated with carbon to avoid charging. After that, the samples were kept inside the instrument and the images were observed at different magnifications.

RESULTS AND DISCUSSION

The physical properties of retted fiber are shown in Table 2.

Tensile strength

It can be observe from the data given in Table 2 that the highest value of tenacity i.e., 5.43 g/denier was exhibited by biological retted (stagnant) fibres whereas chemical retted fibre (pretreated with HCl) showed lowest tenacity (3.22g/denier). Overall, the tenacity of fibres obtained from biological retting methods was better as compared to chemical retting methods. This might be owing to the strong action of chemical on the surface of fibres leading to break down the pectins and other vegetative matter completely making fibers free from bundle as compared to the action of bacteria that left behind the fibres with residual pectin i.e., binding component. Similar finding were observed by Dhanalaxmi and Vastrad (2013), that biologically retted *mesta* fibres exhibited higher strength as compared to chemical (urea) retted fibres and affirmed that the use of urea might have increased the wetting action of water that enhanced the growth of microbes in water.

Further on comparing two biological retting methods, it was found that fibres retted by running water dip exhibited slightly lower tenacity (5.11 g/denier) as compared to the fibres from tank water or stagnant water retting that showed the fibres tenacity of 5.43 g/denier, when submerged for same duration of 15 days. This might be due to the growth of various microbes cover fungi too in plant material submerged in water for a duration that acted on the surface of fibres and decomposed pectin, the vegetative, waxes and other cementing matter. According to Meijer *et al*. (1995), the microbes and fungi releases different types of enzymes that results in degradation of epidermis, parenchyma and lamella to split the bundles into fibres (Akin *et al*., 1996).

The fibres treated with combination of 0.05% EDTA and 5% NaOH exhibited higher tenacity (3.54 g/ denier) as compare to the fibres obtained from pretreatment with 0.5% HCl and treatment with 5% NaOH that revealed tenacity value of 3.22 g/denier. This might be due to the reaction of sodium hydroxide with the noncellulosic material that was slightly decomposed and dissolved by pretreatment of HCl. Wang *et al*. (2008) also found that the increase in the concentration of sodium hydroxide resulted in the decreased values of the breaking strength and the fineness. Kumar *et al*. (2011) also suggested that the removal of non-fibrous components and complete separation of fibres depends on the concentration, time and temperature of the reaction which affects the fibre strength and other physical properties.

The differences in strength of fibres extracted from different retting methods may be due to the difference in the efficiency of biological and chemical agents in removal of vegetative and non-cellulosic matter from the fibres. Overall it was revealed that the chemical retted fibres produced fibres of relatively lower tensile strength as compared to biological retted. Paridah and Khalina (2009) observed that the kenaf bast fibres retted with 5% NaOH and 5% sodium benzoate separately exhibited lower strength as compared to water retted fibres.

Significant difference was found between strength of *S. aculeata* (*dhaincha*) fibres extracted from different retting methods as the p value for tensile strength was smaller (p=.000) than 0.05. Hence it can be concluded that the type of retting method significantly affect the tenacity of fibre.

Fibre fineness

It can be envisaged from Table 2 that the fineness of *S. aculeata* fibres extracted from 15 days of stagnant retting exhibited maximum value (35.3 denier) followed by the fibres obtained from 15 days of running water retting i.e., 32 denier. The fineness value for fibres extracted from pretreatment of HCl and 1% NaOH was 29.4 denier and fibres retted with combination of 0.05%

Table 2: Tenacity and fineness of *Sesbania aculeata* fibres obtained from different retting methods

S.No.	Retting methods	Retting variable	Tenacity (g/den)	Fineness (denier)
1.	Biological retting (Stagnant water method)	15 days	5.43	35.3
2.	Biological retting (Running water method)	15 days	5.11	32
3.	Chemical retting (Pretreated with 0.5% HCl)	5% NaOH	3.22	29.4
4.	Chemical retting (combination with 0.05 % EDTA)	5% NaOH	3.54	27.2

EDTA and 5% NaOH exhibited lowest value for the fineness (27.2 denier). According to Angappan *et al*. (2002), higher the value of fibre fineness in denier, the coarser it will be and vice-versa. The principle of chemical retting was that the gum and other noncellulosic matter were removed layer by layer from the fibres by reacting with chemical and thus producing finer fibres. According to Kashayp *et al*. (2001) alkali helped to remove large amount of noncellulosic substance that facilitated to get clean fine fibres and it also removed cellulosic part of the fibres in a non specific manner along with non-cellulosic gummy material in a chemical treatment resulting in efficient removal of noncellulosic component.

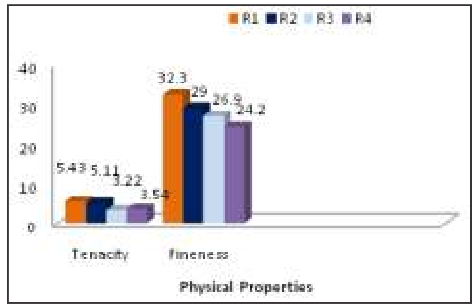
On the whole, the chemical retted fibres were finer as compared to biological retted fibres. This might be due to the enhanced digestibility of lignocellulosic material by the alkali. Contrary to this the incomplete separation of fibres by bacteria resulted in adhering of fibres into bundles in biological retting methods. This attributed to larger value of denier in case of biologically retted fibres. Hongqin and Chongwen (2010) also reported that water retted kenaf fibres displayed coarser fibres as compared to chemical retted fibres. Patak (2014) had also reported that water retted fibres were coarser than chemical treated fibres in case of all five bast fibres namely *semal, tesu, lasora, lantana and phalsa*.

Significant difference was found between the fineness of *S. aculeata* (*dhaincha*) fibres extracted from different retting methods. The p value for fineness was smaller (p=.000) than 0.05, hence it can be concluded that the fineness of fibre varies with the retting methods used for their extraction.

It can be concluded that extraction of fibres through different retting methods had affected the fineness of *S. aculeata* (*dhaincha*) fibres i.e., fineness of fibres differed due to varied chemical and biological retting methods.

Effectiveness of retting method on fibre properties

From Figure 1, it can be envisaged that the stagnant water retted fibres exhibited relatively better properties as compared to other retting methods. According to USDA, 2014 water retting produces more uniform and high-quality fiber. Umoro *et al*.(2014), further explained that the chemical retting was a quicker process than the natural processes (dew, water and enzymatic) but it influenced several properties, including loss in tenacity, colour, and luster. According to Van Sumere (1992), the process of water retting yielded high quality fibres. The



Note:
R1: 15 days stagnant water retting;
R2: 15 days running water retting;
R3: chemical retting (HCl+NaOH);
R4: chemical retting (EDTA + NaOH)

Fig. 1: Comparison between the physical properties among retting methods

bacterial method was relatively better than chemical, because it gave better fibres quality and lower pollution whilst chemical retting requires huge energy and generates costly wastes.

Martin *et al*. (2013) who studied the effect of retting on tensile properties of flax fibre observed that the retting for a period of 19 days was considered good as the fibres exhibited standard quality and good tensile strength as compared to fibre retted for duration of 1 day to 14 days.

Hence, the *Sesbania aculeata* fibres obtained after 15 days of stagnant water retting yielded best fibres in terms of tensile strength and fineness. Ahmed *et al*. (2001) and Bengtsson (2009), also affirmed that the water retting is the cheapest and universally practiced that gave the best fibre quality when visually determining the quality parameters such as length, colour and smoothness Water retting is biological fermentation process which produces biodegradable waste. This enhances the soil nutrient without harming the environment. Therefore this process was selected for further study.

SEM

The surface morphology fibres obtained from different retting methods are shown in Figure 2. Comparison shows that the longitudinal views of stagnant and running water retted fibres were cylindrical in shape and surface was smooth owing to the less removal of vegetative matter that results in the joining of fibres at various places with pectin and lignin present in them.

On comparing 15 days stagnant and 15 days running water retted fibres with fibres treated with NaOH + HCl and EDTA + NaOH , it was observed that chemically retted fibres showed more opening of fibres leading to finer structure evident from increased fineness. Further it

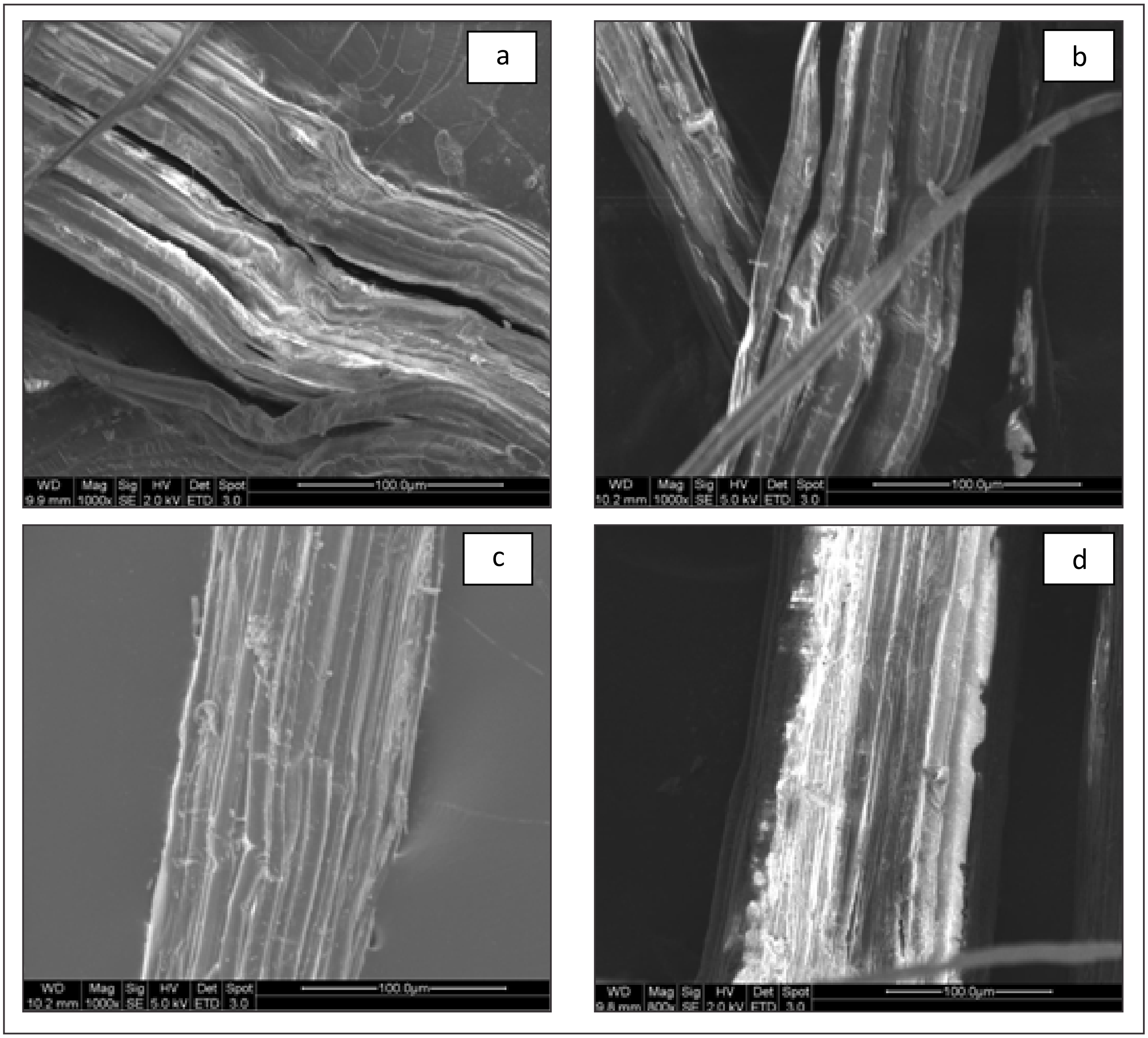


Fig. 2: SEM analysis of retted fibres [(a) With HCl and NaOH magnification X 1000 (b) with EDTA and NaOH magnification X 1000 (c) After 15 days stagnant water magnification X 1000 (d) After 15 days running water magnification X 1000

can also be attributed to the removal of lignin content after the alkali treatment. Balogun *et al*. (2015) also affirmed that NaOH treatment can completely remove lignin without leaving any residue left on the fibre, but the rate of lignin removal depends on the NaOH concentration.

Stagnant retted fibres show minimal damage to fibre structure, fibres appear clean and smooth though intact because separation of individual fibres did not occur as in case of chemical retting. Whereas in fibres retted in running water, it showed uneven surface suggesting negligible opening of fibres and removal of pectin and lignin. Hossain *et al.* (2014) also claimed that alkali treated fibres have a finer fibre bundle with rougher surface compared to those of the untreated ones.

CONCLUSION

Global trends towards sustainable development have brought natural, renewable, biodegradable raw materials, into the focus. *Sesbania aculeata* (*dhaincha*) can be a promising underutilized agricultural resource for the replacement of petroleum based fibres used extensively in technical textile sector. For this reason, there is need to search for the extraction methods to remove the non-fibrous components to completely free the bast fibres from other impurities. Out of the two retting methods, biologically retted fibres exhibited better properties in term of strength and fineness. Hence, the properties exhibited by fibres extracted from biological retting were considered good for technical textile uses. Therefore stagnant water could be considered as ideal medium of retting. It can be concluded that the quality of the fibre depend on the manner of the retting conditions. Hence, it is essential to choose appropriate retting method according to the ultimate use of fibres in different industrial applications. Finally it can be stated that *Sesbania aculeata* (*dhaincha*) fibres can be explored as a potential source for technical textiles.

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