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Chemical treatment of nettle ribbons and its effect on tensile property of extracted fiber

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Abstract
Stinging nettle (*Girardinia diversifolia*) is a cellulosic plant fiber which is abundantly available in the upper regions of Himalayas. The stem fibres are pliable which are used to weave or knit fabric. This fiber is very strong, but rigid and inextensible. This has restricted its usage in handmade textile products only. In order to process this fiber on a range of textile machines for development of a variety of products with enhanced performance, the fiber needs to be strong, flexible and extensible. This study was aimed at to increase the fiber tenacity by giving chemical pre-treatment. The effects of various experimental parameters, such as chemical concentration and time on tenacity of nettle fibres were studied. In the study, the chemical pre-treated nettle fibers were examined for their mechanical characteristics and compared to untreated fibers (control sample). The untreated fiber was less flexible and less extensible. A pre-treatment resulted in increase in tenacity and elongation at break. The increase in tensile strength was found to be 5.46 g/den from 3.84 g/den (as in case of control sample) and elongation increased to 3.33% from 1.66%.

Keywords: Himalayan nettle; chemical pre-treatment; tenacity

Introduction

Nettle is a cellulosic plant fiber which is abundantly available in tropical wasteland areas in the world. This fiber is very strong, but rigid and inextensible (Dreyer and Edom, 2005) [6]. It has been used to prepare many handmade textile products, but its rigidity and inextensibility have made it very difficult to process using machines. Nettle is a cellulosic plant which produces bast fibres for suitable textile uses. Amongst few varieties of available nettle plants all over the world, common stinging nettle (*Urtica diocia*) and Himalayan giant nettle (*Girardinia diversifolia*) are having properties which are suitable for textile end uses. Nettle production (*Urtica diocia*) began in Europe in the 19th Century and during the 1st and 2nd world War, nettle was promoted as substitute of cotton (Vogl and Hartl, 2003) [1]. Nettle is abundantly available in tropical wasteland areas around the world. The nettle family, Urticaceace, contains around 500 species. It is abundantly found on sides of the river, forest land and moist habitat in Nepal and this shrub grows wildly throughout the mountainous regions of India for e.g. Jammu & Kashmir, Sikkim, and Uttarakhand, Arunachal Pradesh. In Uttarakhand Himalayan nettle is locally known as bichchhu buti, nilgiri and kandali, it is found in Chamoli and Uttarkashi districts which it grows as undergrowth wild fibre. It is a perennial plant possibly best known as a source of strong, light-weight, sustainable natural fibers. (Badoni and Ghosh, 2015) [3]. The plant has a long history as fiber plants were widely used for bowstrings, fishing nets, sail cloth and even fine textiles in many cultures. Its use as a fiber disappeared after the arrival of flax and cotton in the 16th century, but now a day's its use is being revived. It is found better than jute and can be blended with other fibers. Different parts of the nettle plant have been used as food, fodder and raw material in cosmetics, medicines and bio-dynamic agriculture. Until few years back it was lying unexplored when on realizing its potential in the field of textiles. Many institutions in the region initiated research and developmental activities on the possibilities in the field of handloom based products from nettle. Many countries like United Kingdom and Germany have been involved in the development of nettle since 1999, and have made significant expansion in this field (UBFDB 2015) [4]. Nettle fiber is very strong, but rigid and inextensible. This has restricted its usage in handmade textile products only, though it has potential for replacing glass fiber as reinforcement in textile-reinforced polymer matrix composites (Yu and Franck, 2005) [2]. Natural fibers have the advantages of low density, low cost, and biodegradability. Therefore, chemical treatments are considered in modifying the fiber surface properties. Chemical treatments includes alkali, silane, acetylation, benzoylation, acrylation, maleated coupling agents, isocyanates, permanganate and others. The chemical treatment of fiber aimed at improving the adhesion between the fiber surface and also increase fiber strength (Xue and Lope, 2007) [5].

Methodology

Sample collection and material used

The Himalayan nettle (*Girardinia diversifolia*) ribbons of 1 to 2 m length were procured from local market of Bageshwar district, Uttarakhand (India). The collected fibre ribbons were dipped in water for 4-5 hours and washed once with running tap water to remove the dirt and impurities. Then the sample was dried under shade until it was completely dried. The dried ribbons were cut into staple length, then directly used for chemical pretreatment. Analytical grade sodium chlorite, acetic acid and sodium hydroxide pellets were purchased from HiMedia Mumbai.

Optimization of chemical pre-treatment

The cellulose fiberes were extracted from nettle ribbons using the following process:

Five gram of ribbons were rinsed with tap water to remove foreign particles and dust. The cleaned ribbons were then soaked in 500 ml beaker containing 150 ml distilled water which was then transferred to hot plate at boiling temperature. Sodium chlorite and acetic acid were added to the beaker and treated for different time. The obtained ribbons were filtered, washed and rinsed with distilled water. The obtained ribbons were soaked in NaOH solution for one hour at room temperature (23^0 C ± 2^0 C). The ribbons were then filtered and immersed in 150 ml distilled water containing acetic acid to neutralize the fibers. The mixture was stirred for approximately 30s before it was allowed to settle for 5 minutes. It was then rinsed with water. Finally, the obtained fibers were dried under shade and the fibers were extracted.

Different experiment sets were prepared to optimize the pretreatment of nettle ribbons from the above process. The classical method involved optimization of pre treatment conditions to see effect on tenacity of nettle fiber by optimizing 'one variable at a time'. The process parameters thus standardized included sodium chlorite concentration (1-6% w/v, with an interval of 1), acetic acid concentration (0.5-3% v/v, with an interval of 0.5), treatment time (1-6 hours with interval of 1) and sodium hydroxide concentration (0.5-3% w/v with an interval of 0.5). R version 4.0.1 software was used to see the significant effect of different chemical concentrations on tenacity of fibre. The six different concentrations of each parameter were coded as I1, I2, I3, I4, I5, I6. Box plot graphs using R software were plotted to assess difference in tenacity at different concentrations.

Control Sample

The ribbons were dipped in water for 4-5 hours in water and then washed and dried under shade. After drying fibers were extracted and these untreated nettle ribbons were used as the control sample (Fig 1).

Fig 1: Untreated fibre (control sample)

Assessment of physical properties of nettle fibre

The fibre fineness, tenacity and elongation of nettle fibres was assessed by using digital instrument comprising of two units: Vibromat M and Fafegraph M. The drawn fibre specimen of 40 mm length was held vertically in a given clamp. A weight of 65 mg to 100 mg was attached on other end of fibre to keep it straight. The fibre specimen was subjected to an acoustic oscillation that resulted reading for fibre fineness on digital screen.

The fibre specimen with pretensile weight was shifted to Fafegraph M, a semiautomatic microprocessor with controlled tensile strength tester, based on the principle of Constant Rate of Extension (CRE). It estimates the breaking force, elongation and tenacity of the single fibre strand. The statistical value of DTE (denier, tenacity and elongation) was recorded after each test.

Results and discussion

Effect of chemical pre treatment on tensile property of nettle

Concentration of sodium chlorite (a)

The data relevant to the tenacity of the *nettle* fibres on varying concentration of sodium chlorite, acetic acid, time and sodium hydroxide is given in Figure 2.

It is apparent from Table 1 that tenacity of *nettle* fibres decreased with increase in the concentration of sodium chlorite ($NaClO_2$). This might be owing to the removal of noncellulosic matter and impurities from surface of nettle fibre. The highest tenacity i.e. 5.5 g/den was found at 4% sodium chlorite concentration as shown in the box plot graph Figure 2(a).

In the presence of acetic acid in aqueous medium sodium chlorite dissociates into chlorine ions and chloride ions which destroys lignin into simpler compounds and leads to the permanent whiteness of the nettle fibre (Rabetafika *et al*., 2014) [7]. The treated nettle fibres were shown in Fig. 3.

Concentration of acetic acid (b)

It is clear from Figure 2(b) that tenacity of *nettle* fibres decreased with increase in acetic acid concentration. The highest tenacity (5.45 g/den) was found at 2% acetic acid concentration as shown in the box plot graph (b).

Treatment time (c)

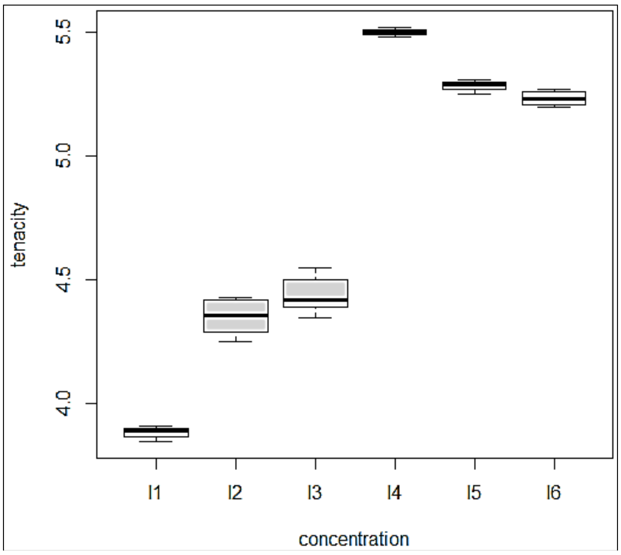
The increase in tenacity was found at 3, 4 and 5 hour treatment time. The treatment time 1 and 2 hours showed decreased tenacity i.e. 3.8 g/den and 4.09 g/den. This is because the fibre requires some time to swell so that the auxiliary agents can enter into the fibre. At lower treatment time and lower concentrations auxiliaries do not decompose the lignin and other compounds. The highest tenacity 5.45 g/den was found at 4 hour treatment time as shown in box plot graph (c).

Concentration of sodium hydroxide (d)

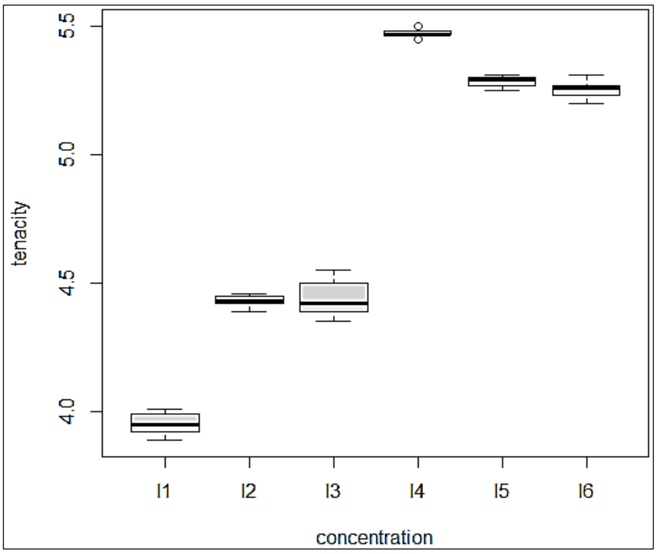
The highest tenacity 5.52 g/den was found at 2% concentration of NaOH as shown in box plot graph (d). In a study Kumar and Das (2017) [9] reported a mild alkali treatment which resulted in increase in tensile strength and elongation at break of nettle fibre without much changing initial modulus.

Table 1: Tenacity of nettle at different concentrations of sodium chlorite (a), acetic acid (b), treatment time (c) and sodium hydroxide (d)

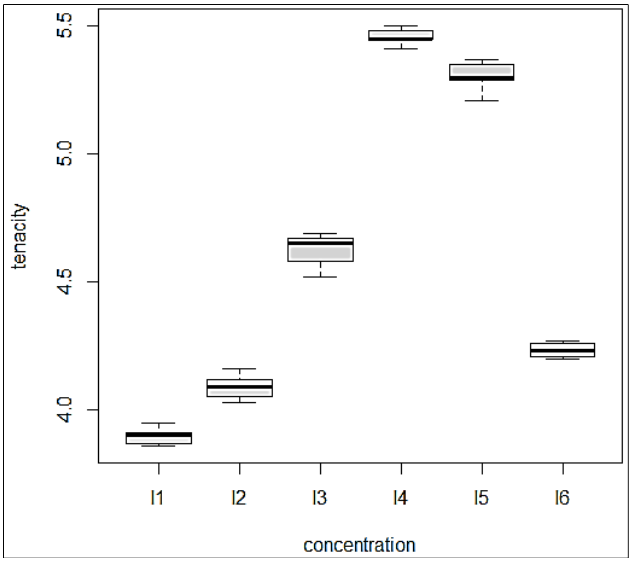
Code	Sodium chlorite (%)	Acetic acid (%)	Treatment time (Hours)	Sodium hydroxide (%)
I1	3.88	3.95	3.89	4.07
I2	4.35	4.43	4.09	4.48
I3	4.44	4.44	4.62	4.48
I4	5.5	5.47	5.45	5.52
I5	5.28	5.28	5.30	5.27
I6	5.23	5.25	4.23	5.28



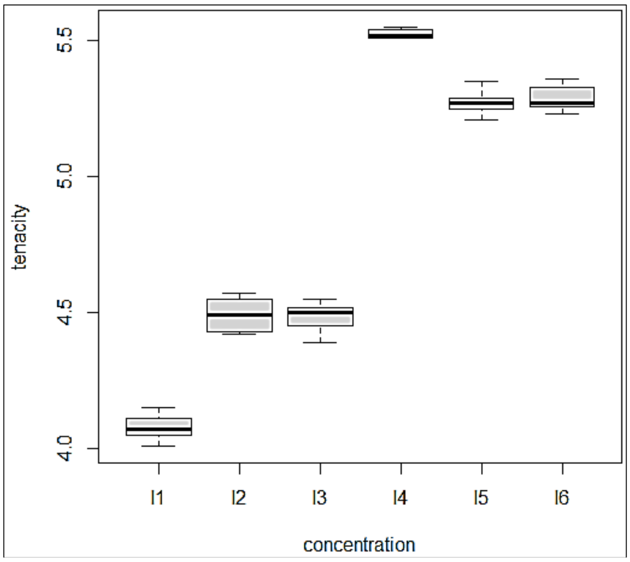
(a)



(b)



(c)



(d)

Fig 2: Sodium chlorite (a), Acetic acid (b), Treatment time (c) and Sodium hydroxide (d)

Mechanical properties of Himalayan nettle fibres before and after delignification

It is given in the Table 2 that nettle fiber (Treated sample Fig 3) exhibited decrease in the count of 14.77 denier to 11.60 denier, tenacity 3.84 g/den to 5.46 g/den, increase in elongation from 1.66% to3.33% and increase in breaking force from 54.93% to 77.84%.

Table 2: Mechanical properties of control sample and pre treated nettle fibres

S. No.	Mechanical properties	Fiber extracted from nettle ribbons (Control sample)	Chemical pre-treated nettle fiber
1	Fineness (den)	14.77	11.60
2	Tenacity (g/den)	3.84	5.46
3	Elongation (%)	1.66	3.33
4	Breaking Force (g)	54.93	77.84

Sodium chlorite and acetic acid treatment leads to break down fibres in both radial and axial directions. NaOH can disrupt the initial fibrillar structure which also leads to the disaggregation of microfibrils from their neighbouring fibres (Rezende *et al*., 2011) [8]. Therefore diameter of fibres was reduced.

The increased tenacity is due to the action of the sodium chlorite and sodium hydroxide on the non-cellulosic contents and their removal. These non-cellulosic contents i.e. hemi-cellulose and lignin are amorphous and are very disorderly in nature. They form pools in between the cellulosic regions which prevent the α-cellulose chains from positioning themselves in the direction of loading. The removal of these pools makes the fibres α-cellulosic regions to withstand higher stresses than untreated fibre (Vardhini, *et al*., 2016) [10]. Therefore increased tenacity of the nettle fiber also leads to the increase in breaking force. The removal of non cellulosic material in chemical treated fibre leads to the decrease in stiffness and hence elongation is increased.

Fig 3: Treated fibre

Conclusion

Investigating the effect of chemical pretreatment demonstrated that untreated nettle fiber was less flexible and less extensible. The chemical pretreatment resulted in increase in the strength and elongation-at-break, improved appearance and decrease in width of nettle fiber. Thus, chemical pretreatment leads to the removal of amorphous structure and also increase the crystallinity and purity of the cellulose obtained.

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