Summary of the work
Summary

Ecological or environmental problems have become global in character and there is an urgent need worldwide to tackle these problems. Environmental protection and production of quality textiles of international standards are two serious challenges before textile processors. Natural dyes/colors have been used historically throughout the world. However, their use has decreased to a large extent due to the arrival of synthetic dyes. Very few synthetic dyes that prove genotoxicity concerns are azo dyes derived from a relatively small group of aromatic amines (e.g. benzidine unit 2-naphthylamine). The associated azo dyes and parent amines are no longer commercially important. In view of the disastrous effects like allergy, mutagenicity and carcinogenicity of few synthetic dyes, including their non-biodegradability, the use of natural dyes has once again gained the interest. The plant kingdom offers a vast source of natural dyes/colours which can be obtained from many plant parts e.g., leaves, fruits, seeds, flowers, barks and roots by boiling, scrapping, powdering and mixing with other materials. In this regard, the western Himalayan region of India possesses various promising plant species for extraction of natural dyes/colors. Though reports are available on the extraction of color components from different dye bearing plant species of India but many species still remain unexplored.

There are some suggestions by various authors for use of natural dyes in textiles. However, the major hitch for use of natural colorants is the industry in the presence of different moieties (part or functional group of a molecule) in plant extracts as well as the variations of color active compounds due to environmental/seasonal changes. Further, the isolation of single molecule based color is also quite laborious and expensive.

Among many such natural dyes, tea derived from leaves of *Camellia sinensis*, turmeric from rhizome of *Curcuma longa*, madder from *Rubia cadifolia*, henna from *Lawsonia inermis* Linn. and baras from *Rhododendron arboreum*, are the most widely used dyes. The aqueous extract of tea has been reported to be used for dyeing of cotton and jute. The leaf extract of *L. inermis* are usually used to stain hands, nails and feet and has implication in herbal medicines. The major colouring agent in henna has been identified as lawson (2-hydroxy-1, 4-naphthaquinone), the property for which it finds its place in textiles. The presence of flavonoid colorant, luteolin has also been mentioned. Use of turmeric, madder, catechu, indian rhubarb, henna, tea and pomegranate rind on nylon has also been documented. The use of Baras dye derived from *R. arboreum*, as a natural dye due to the factors like its effective dyeability and fastness, has also been indicated. Despite the
fact that all the above mentioned species are rich source of colouring agents, one needs to explore the dyeing effects of their dyes on different fabrics by adopting different methods of extraction as well as to study their fastness in presence or absence of different mordants.

Along with the ecofriendly approach of choosing natural dyes, the consideration to other natural materials has also been enhanced. In textiles, market of bamboo clothing has suddenly raised due to the facts that bamboo fibres are an environmental friendly fibres extracted from bamboo which is renewable, fast growing and degradable. Some studies convey that bamboo, jute, ramie etc. fibres/fabrics can be manufactured or produced with use of ecofriendly chemical or additive. The present study is therefore aimed at pretreating the bamboo fibre/fabric in environmentally friendly ways and investigating the effect of pretreatments on the properties and dyeing behavior of the fibre particularly with natural dyes like turmeric, tea etc. In this work, a possible bamboo retting system is also developed and properties of retted bamboo are also discussed.

The background and results of our findings have been divided into six chapters.

Chapter 1: Introduction and review of literature

Chapter 2: Study on extraction of bamboo fibres from raw bamboo fibres bundles using different retting techniques

Chapter 3: Study on the performance of bamboo fibre modified with different concentrations of sodium hydroxide and chlorine containing agents

Chapter 4: Studies on the effect of bleaching agents on CAN retted & alkali pretreated bamboo fibres

Chapter 5 Studies on the effect of mordants on dyeing behavior of bamboo fibres using natural dyes

Chapter 6 Dyeing of Bamboo with Tea solution extracted by using different solvents

Chapter 1: Introduction and review of literature

This chapter deals with the introduction and review of literature about importance of natural dyes and uses of natural dyes and bamboo fibre in textiles. From the detailed literature review, the following conclusions were drawn:

- Considering the wide spread use of natural dyes in textiles, an attempt has been made in the present study to explore the dyeing effects of natural colorant extracted from fresh and dry leaves of tea as well as from rhizome of turmeric.
The literature survey revealed that although some reports are available on the use of these extracts on different textile substrates viz. cotton, wool, nylon, silk, polyester etc., but yet reports concerning the use on Bamboo fibre/fabric is scanty.

Natural bamboo fibers have excellent properties suggesting that there is a good potential for them to be used in textiles; however, they have not received the attention that they deserve owing to their coarse and stiff quality. The high lignin content of the fibre is the major cause of its stiffness.

The invention of bamboo fibre is the biggest contribution of mankind to protect naturally rare minerals/resources and ultimately the environment as a whole. The property that bamboo is highly renewable grass has resulted in its being classified as ecofriendly, which in turn has resulted in its wide use in textile industry. Repeated tests have proved that the bamboo fibre has a strong durability, stability as well as tenacity. The thinness and whiteness degree of fibre obtained from bamboo has been found to be similar to that of viscose staple fibre. Moreover, this fibre being natural cellulosic fibre can achieve natural degradation in soil and can be blended with other materials such as cotton, hemp, lyocell, modal fibre and so on.

Chapter 2: Study on extraction of bamboo fibres from raw bamboo fibres bundles using different retting techniques

Subsequent to cellulose, lignin represents the second richest ingredient in the bamboo and processors have been focused on its chemical nature and structure. Bamboo lignin is a typical grass lignin. The rigidity in bamboo is basically due to its lignin content and acts as a permanent bonding agent. Lignin is a highly branched three dimensional polymer and is not swelled by the usual swelling agents. The chemical composition of bamboo fiber is the same as all bast fibers, that is, cellulose constitutes the majority and lignin needs to be reduced further for textile applications.

We followed the following mechanical method to produce the fibre bundles from raw bamboo:

- Nodes of raw bamboo (2-3 years old) were detached
- Residual part was cleaved in longitudinal direction to thin slabs with 15-20 cm in length & 1.5-2 mm in thickness by the slicer.
- Finally, they were converted manually into fibre bundles.
Extraction of natural bamboo fibres using following different retting techniques:

- Chemical Assisted Natural Retting (CAN)
- Acid Retting
- Alkaline Retting

The surface properties of original & treated bamboo fibres were examined by SEM. A large quantity of gum & lignin in untreated bamboo was found in untreated fibre bundles and it was observed that the natural bamboo fibre consists of numerous elementary surface fibres. After chemical assisted natural retting (CAN), there was a remarkable reduction in number of elementary surface fibres which was a clear indication that the technical bamboo fibre became smoother and finer than before.

Moreover, the inner structure of bamboo fibre became looser after different retting techniques. From SEM images, the clear cracks were also observed in retted fibre bundles which played an important role for maximum separation of fibres from the bundles easily in the subsequent wet processing of bundles due to ease penetration of processing chemicals.

Determination of lignin content & mechanical properties of fibres exhibited relationship between lignin content and mechanical properties. The removal of lignin in fibre structure led to a decrease in their tensile strength.

A detailed examination in terms of physical properties was carried out to determine the effects of different retting treatments on bamboo fibres. As it was found that the removal of non-cellulosic matter by retting process enhanced the fibre separation from the bulk, therefore, the retting process also improved the ability of the fibres to absorb higher degree of moisture. Acid retting process highly enhanced the fibre appearance as seen from its whiteness index value. The drop in tenacity of the fibre was also comparatively low. Chemical assisted natural retting caused highest loss in weight also at the cost of maximum loss in tenacity. The maximum loss in lignin was caused by chemical assisted natural retting process. Alkali retting gave an intermediate effect on fibre appearance and strength of fibre. The results showed that the difference of moisture content of the acid treated and the alkali treated fibres was much lower than that of the chemical assisted natural retted fibres. But physical fibre separation was achieved through natural retting.

The natural, acid and alkaline retted bamboo fibers were further treated with sodium carbonate 5-30 g/L, Lissapol-D 0.5 g/L and kept at 80°C for 45 minutes with fiber to liquor
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ratio 1:20. The concentrations of sodium carbonate were 5g/L, 10 g/L, 15 g/L, 20 g/L and 30 g/L. At the end of scouring treatment, the fibers were neutralized with acetic acid and then thoroughly washed with distilled water. After that it was observed the effect of Na$_2$CO$_3$ concentration on weight loss, moisture content, whiteness index, yellowness index, tenacity of natural, acid and alkali retted bamboo fibres.

Chemical Assisted Natural & Acid retted and scoured fibres were selected for bleaching with H$_2$O$_2$ (10 g/L) in the presence of Clarite G (1gpL), Ultavon EL (2 g/L) & caustic soda (3 g/L) at 98°C for 60 minutes. These fibres were also selected for bleaching with Peracetic acid (10 g/L) in the presence of sodium meta silicate (5g/L), at 70°C for 90 minutes at neutral pH. It was found that with subsequent bleaching with hydrogen peroxide and peracetic acid of retted and scoured (with different concentration of sodium carbonate solution) fibres, the whiteness of all the samples is increased up to all concentration levels. From results, it was clear that these two processes of retting followed by treatment with different concentrations of sodium carbonate & bleaching had an excellent improvement in whiteness & reduction in yellowness. H$_2$O$_2$ bleaching found to result in better whiteness & yellowness in comparison with PAA bleaching.

Chapter 3: Study on the performance of bamboo fibre modified with different concentrations of sodium hydroxide and chlorine containing agents

In this study, the bamboo fibre bundles (CAN retted fibre bundles) were treated with sodium hydroxide at different processing conditions, in combination with potassium hydroxide and chlorine containing agent sodium chlorite solution by considering previous different studies from literature survey. The purpose of this study was to develop efficient method (five methods A, B, C, D & E) for the extraction/loosening of bamboo fibres for further textile applications. An optimum method for extraction of fibres from the bundles were standardized here and the extracted fibres were further characterized by calculating weight loss, whiteness & yellowness indices, lignin content measurement, tensile strength, Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD) and Scanning electron microscope (SEM).

Natural bamboo fibres extracted by CAN retting technique were modified during this study and best processing parameters were optimized to improve the fibre quality for textile application. Reduced contents of noncellulosic substances and improved fibre fibrillation after different chemical methods of modification were observed from experimental results. Method B produced reasonable reduction in lignin content and weight loss with acceptable
whiteness and tensile strength. From FTIR spectra, it can be observed that all the bands mentioned over were notable weaker for the fibres treated with alkali at low temperature for prolonged time (method B). Furthermore, from their XRD pattern, it was found that fibres had the similar diffraction peaks with different intensity. SEM pictures showed that the fibres treated with methods B & E had more uniform geometry of fibrils arrangement than the fibres treated with methods A, C & D.

Hence it can be concluded that chemical amendment as carried out in method B may propose a opportunity of using natural bamboo fibres for further textile applications.

Chapter 4: Studies on the effect of bleaching agents on CAN retted & alkali pretreated bamboo fibres

This chapter reports the bleaching of CAN retted and alkali treated bamboo fibres with Peracetic acid (in-house prepared PAA) [See Section 4.2.2.3. Preparation of PAA] and comparison with bleaching with H$_2$O$_2$ in respect of some important properties. The bamboo fibres were treated with different concentrations of sodium hydroxide viz. 0.05 N, 0.1 N, 0.2 N & 0.3 N) at 40°C for 72 hours with fiber to liquor ratio 1:20. After that the fibers were neutralized with acetic acid and then thoroughly washed with distilled water. At the end of treatment, these fibres were bleached with hydrogen peroxide and peracetic acid separately.

This two-stage delignification of bamboo fibres with alkali and different bleaching agents could obtain fibres with good physical appearance & properties. Alkali pretreatment prior to bleaching could significantly reduce H$_2$O$_2$ & PAA loading in subsequent stage by partially removing the lignin and swelling the fibres. The maximum loss in lignin was found in case of NaOH→H$_2$O$_2$ process along with no acceptable weight loss. On the other hand, the pretreated fibres with higher concentration of sodium hydroxide i.e., 0.3 N followed by PAA bleaching step (NaOH→PAA) produced reasonable reduction in lignin content and weight loss with acceptable whiteness and tensile strength.

Chapter 5: Studies on the effect of mordants on dyeing behavior of bamboo fibres using natural dyes

In this study, the experimental work was planned to deal with the following objectives: (1) extract color rich fraction from the source (2) examine the dyeing characteristics on mordanted bamboo fibres. For this work we have selected (A) Turmeric (rhizomes of Curcuma longa) and (B) Tea (Camellia sinensis) as the plants of selection
because these are abundantly available throughout the northern and western Himalayan region of India.

From this work, it was clear that the exhaustions were satisfactory for all the three mordant systems used viz. Alum, Tannic acid and Alum→Tannic acid for dyeing of bamboo fibres with Turmeric and Tea as natural dyes. Among the three mordant systems, (Alum→Tannic acid) system produced highest colour uptake on bamboo fibres. Among the two dyes used, Tea produced higher colour yield compared to Turmeric for the identical dyeing conditions. The pre-mordanting treatment enhanced the dye uptake for all the samples. After critically analyzing the results, it can be said that among the three types of mordants, Alum and Tannic acid combination gave best result as the percent exhaustion with acceptable fastness properties.

**Chapter 6: Dyeing of Bamboo with Tea solution extracted by using different solvents**

Dyeing of bamboo fabric with green tea as a natural colorant had been investigated in this study. The dyeing process was carried out (with and without mordanting) using pre-extracted dye stock employing either 100% water or solvent: water (1:9) mixture as the extraction medium. Three different qualities of tea leaves were used as source of color extraction. The dyeing properties on bamboo fabric had been evaluated. Dye exhaustion, color depth in terms of K/S, washing fastness, light fastness, etc. were compared for dyeing performed at 90°C for 1hour using four extracted dye stocks on pre-mordanted and unmordanted samples. The following results were observed.

- Significant increase in the brightness of the dyed samples was observed in case of aqueous extracted dye stock. This comparison eliminates the requirement of solvent pre-extraction of tea leaves and motivates us to use directly the aqueous extract of tea which also reduces the cost of dyeing. The produced shades were ranged from medium brown to dark brown.

- The resulting wash fastness of the dyed samples was good to excellent in all the cases. The colour staining rating has been observed very good (4-5) in all the dyeing trails. Among the three qualities of tea, the light fastness of Kawa was found to be the best which was further improved after mordanting in case of Hyson & IHBT due to the formation of coordination complexes of the dye with the mordants.

The structural characteristics of the bamboo fibre were different from those of other textile plant fibres. A detailed examination in terms of physical properties was carried out to determine the effects of different retting treatments on bamboo fibres. Results showed the
physical changes after different treatments & the removal of non-cellulosic matter by retting process enhanced the fibre separation from the bulk. Chemical assisted natural (CAN) retting caused highest loss in weight also at the cost of maximum loss in tenacity. The maximum loss in lignin was caused by CAN retting process. Further, two-stage delignification of CAN retted & scoured bamboo fibres, with alkali and different bleaching agents could obtain fibres with good physical appearance & properties. Alkali pretreatment prior to bleaching could significantly reduce $\text{H}_2\text{O}_2$ & PAA loading in subsequent stage by partially removing the lignin and swelling the fibres. The maximum loss in lignin was found in case of NaOH→$\text{H}_2\text{O}_2$ process along with no acceptable weight loss. On the other hand, the pretreated fibres with higher concentration of sodium hydroxide i.e., 0.3 N followed by PAA bleaching step NaOH→PAA produced reasonable reduction in lignin content and weight loss with acceptable whiteness and tensile strength. Further study on dyeing, demonstrated that tea and turmeric could be used as a colorant for dyeing of bamboo using aqueous extraction of Tea & Turmeric.