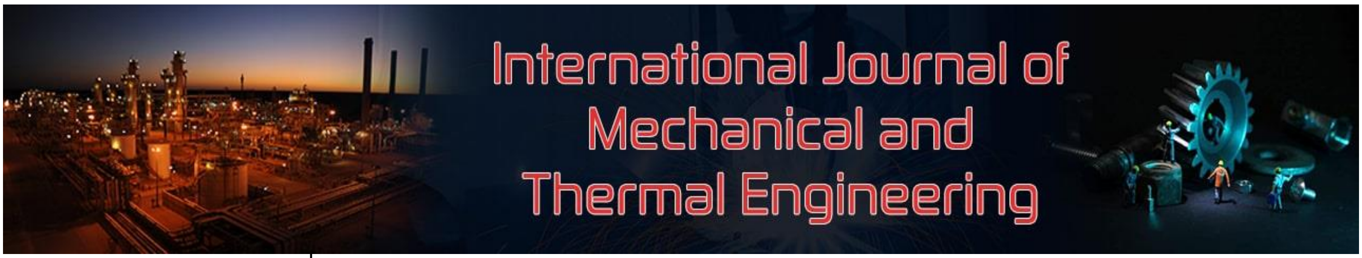

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SEM investigation for mercerization of Bhimal (*Grewia optiva*) Fiber

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Abstract
Mercerization is an alkalization procedure usually used to enhance the properties of any plant fiber. It involves dipping the fiber into a mixture of sodium hydroxide (NaOH) solution and water. This investigation explores the various constraints of alkalization along with their influence on bhimal (*Grewia optiva*) fiber surface structure. A mixture of NaOH to water was in the ratio of 1:10. Bhimal fiber was dipped into the solution for 24 hr. and then washed with warm water followed by distilled water and allowed to dry at room temperature. A gold coating was done over the sample to untreated and treated bhimal fiber regarding SEM examination for better conductivity. Alkalization results in an improvement in mechanical strength, surface morphology and porosity. Mercerization contributes to increasing surface roughness, decontamination, and developing strong interfacial adhesion bonds with epoxy in composite fabrication. Additionally, synthesizing bio-composites with bhimal fibers incorporation and bio-polymer matrix is having the capability to develop sustainable bio-materials.

Keywords: Bhimal fiber (*Grewia optiva*), SEM, mercerization, alkalization, natural fiber

Introduction
Biocomposites comprise natural fibers reinforcement and a matrix of biopolymer thus it delivers a sustainable composite material [1, 2]. Bhimal tree, a multipurpose tree, exhibits a good reinforcement material as bhimal fiber for biocomposites [3]. The synthesis of bhimal fiber composite involves bhimal fiber extraction, alkali treatments, selection of matrix, and fabrication methods, each of which influences the characteristics of the developed composites [4, 5]. Considering the deviation of composite properties is very important for optimizing, and diversifying their future applications sectors. The increasing requirements for sustainable materials are leading to the investigation of the renewable and low-carbon footprint of biocomposites along with biodegradability consideration [7]. Bhimal fiber is a highly demanding fiber to incorporate in composite because of its higher mechanical properties than other natural fibers [8]. This research aims to study the mechanical properties and morphology examination of bhimal fiber along with mercerization of bhimal fiber. A study of the chemical composition evaluation of bhimal fiber is tabulated in Table 1 when exposed to urea at varying percentages.

Table 1: Chemical composition of bhimal fibers [9] (Reproduce with permission)

Sample	Description	Neutral Detergent Fibre (%)	Acid Detergent Fibre (%)	Cellulose (%)	Hemicellulose (%)	Lignin (%)
A	The fibrous layer was manually pulled and removed	64.88	58.14	31.36	6.74	23.77
B	Extracted by water retting by the local people of Dehradun	64.33	59.86	35.91	4.47	22.78
C	Urea Retted @ 2.5% own solution	70.11	65.70	42.60	4.41	20.90
D	Urea Retted @5% own solution	65.56	57.58	41.65	4.98	20.80
E	Urea Retted @2.5% own and 5 gpl hydrogen peroxide treated	70.50	66.09	43.47	4.41	21.42
F	Urea Retted @5% own and 5 gpl hydrogen peroxide treated	70.40	65.07	43.08	4.33	21.04

Fig 1: Bhimal Fiber

Grewia optima is a Himalayan tree having a height of 10 m to 12 m in Himachal Pradesh and Uttarakhand state, India, naturally originating at 2000 m altitudes. The bhimal plant sheds its leaf during the calendar months of March to April, and meanwhile, the bhimal flowers bloom also along with fruit creation. Villagers in the calendar month of November extract bhimal fiber and consume the plant leaves for animals' fodder. The sticks are allowed to be dried under any shade and later exposed to direct sunlight for complete drying during the calendar month of April. The dried sticks further goes for the retting process up to three months in a water pond. Afterwards, the bark goes to uncovered, washed, and exposed to sunlight for drying to eliminate moisture content [10].

Alkali treatment with NaOH of any plant fiber is usually used to modify their surface structure specially for thermoset and thermoplastic resin composites [11]. The presence of waxy elements and various impurities over the surface of natural fibers affects and obstructs strong bonding with epoxy because of low surface tension [12]. Mercerization, also called alkali treatment, is used to increase bonding strength and reduce the fibers hydrophobically. Mercerization includes dipping the bhimal fibers in a NaOH-water mixture, allowing to conversion of the native cellulose to cellulose II, which results in enhancing the crystalline cellulose content [13]. In a study, bhimal (*Grewia optiva*) fiber had been dipped in a 10% (w/v) NaOH solution at room temperature for the next 4 hours. Mercerization enhanced the bhimal fiber crystallinity along with enhanced thermal stability compared with raw fiber [14]. In another study, cellulose microfibers were extracted from a bhimal plant having a mean diameter of 10 μm. The bhimal fibers' surface structure and morphological characteristics along with thermal stability got enhanced by alkalization and confirmed the effective removal of wax and impurities resulting in an increment of cellulose content and crystallinity index by 82.5% and 82% respectively. Additionally, fabricated composite with 40 wt% bhimal fiber incorporation exhibited higher tensile strength, impact strength, and hardness [15].

Materials and methods

Laboratory-grade NaOH pallets were used for Alkali treatment. The raw bhimal fiber collected from Uttarakhand were used. Distilled water was purchased from local market. Scanning Electron Microscopy (SEM) examination was done with model JSM-6610LV, JEOL Limited, Tokyo, Japan along with gold coating by model JFC-1600, JEOL Limited, Tokyo, Japan.

Fig 2: Scanning Electron Microscopy Equipment

Results and discussion

The mercerization chemical process exhibits water particle development at the end of the alkalization reaction along with the NaO compound (Equation 1).

$$\text{(Bhimal Fiber)-OH} + NaOH => NaO + H_2O \quad (1)$$

SEM is a very common analysis method for the morphology examination of any substance [16]. Gold/silver coating over the outer surface of the specimen improves fiber conductivity resulting in better SEM image capturing [17]. Microstructure and diameter of fiber, surface structure, along with defect analysis are studied with SEM examination [18].

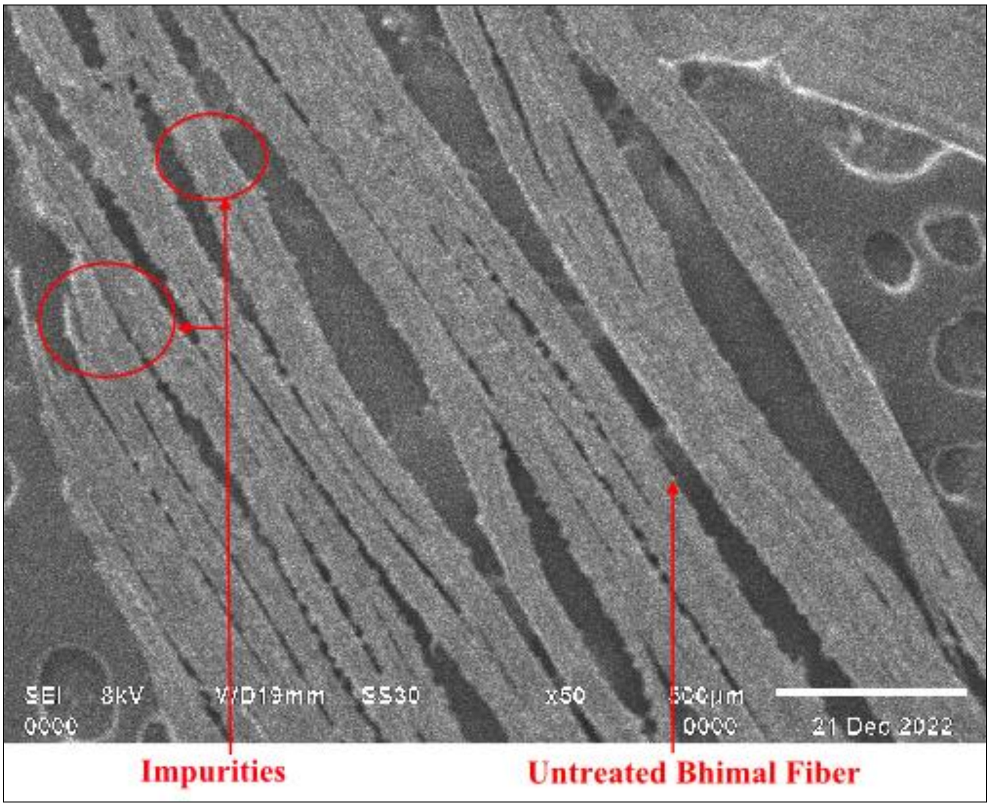

Fig 3: SEM image of bhimal fiber before alkalization

Untreated Bhmal fiber exhibited a waxy and smooth surface structure along with many natural impurities (Fig 3) which are not suggestible for any composite making [19]. SEM examination revealed that it may have poor interfacial adhesion with polymers, which will affect the mechanical strength. The cellulose I do not change, along with limited porosity [20, 21].

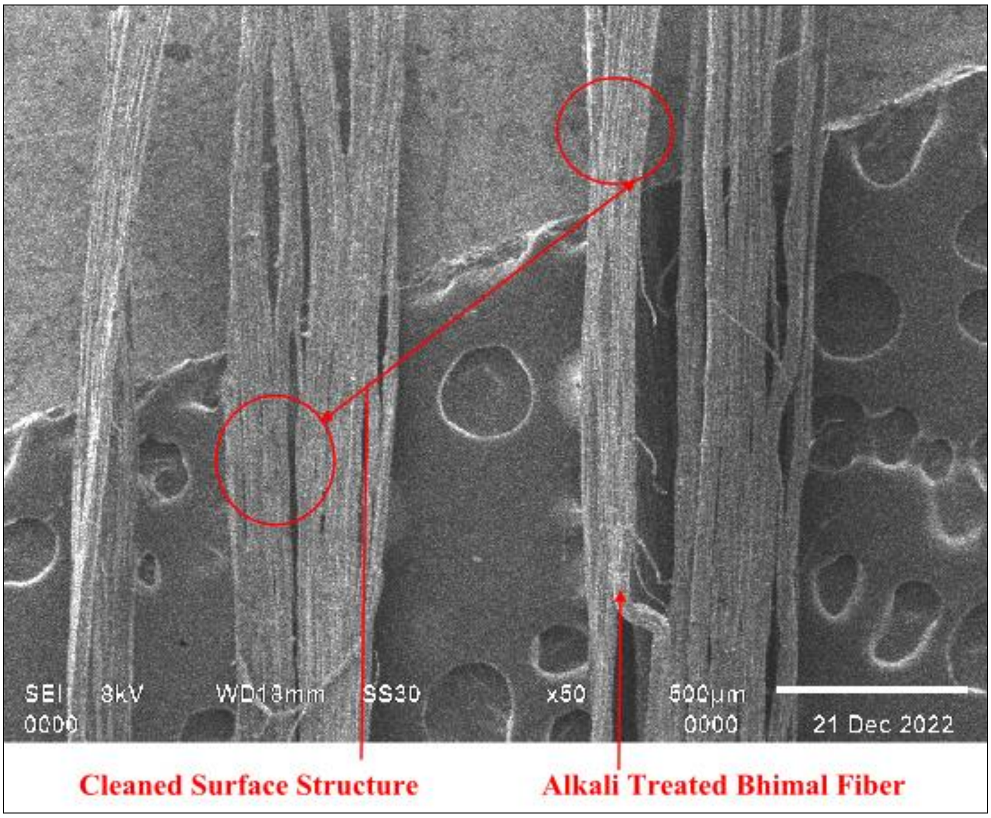

Fig 4: SEM image of bhimal fiber after alkalization

SEM examination of NaOH-treated bhimal fibers exhibits many morphological modifications. The bhimal fibers showed enhanced in diameter and surface roughness, which will increase interfacial adhesion bonding with epoxy resin by decontaminating of surface [22]. Changes in cellulose and swelling of the fiber microstructure is also observed [6].

Conclusion

The SEM examination of alkalized vs untreated bhimal fiber exhibited considerable results. Bhimal fiber diameter got increased because of swelling during alkalization. The surface structure has become roughish, and decontaminated after NaOH treatment while the effect of gold coating is also observed. The surface structure in SEM image confirms fiber suitability for better interfacial adhesion bonding with epoxy resin. Modifications in crystallinity, for cellulose I to cellulose II transformation, were detected. Additionally, the SEM results for the microstructure of bhimal fiber exhibited enhanced porosity with swell behaviour. These results support the constructive effects of mercerization regarding bhimal fiber inclusion in composite fabrication.

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