

High Intensity Ultrasonication (HIU) Effect on Sunn Hemp Fiber Reinforced Epoxy Composite: A Physico-chemical Treatment towards Fiber

Chinmayee Dash, Asim Das and Dillip Kumar Bisoyi*

National Institute of Technology Rourkela, Rourkela-769008, Odisha, India

Email- dash2016@chinmayee@gmail.com, dkbisoyi@nitrkl.ac.in*

Introduction:

Sunn hemp (*Crotalaria Juncea*) fiber has reported to be more ecofriendly than synthetic fiber due to its high fiber aspect ratio (500-600), low density (1.4-1.6 g/cm³), high cellulose content (70-78 wt %), abundant availability, durability that serve a remarkable criteria in the use of composite fabrication[1]. But the inherently hydrophilic nature causes poor adhesion with hydrophobic matrix that leads to poor crosslinking [2]. So it is necessary to treat the fiber in order to reduce the hydrophobicity prior to develop composites. Many pretreatment techniques have been adopted to enhance sunn hemp fiber structure [3, 4, 5, 6]. The present paper focus an assessment on a physico-chemical treatment where a High Intensity Ultrasonicator (HIU) processor has used for physical treatment and alkali used for chemical treatment. High frequency electrical energy generated by HIU processor is converted to mechanical vibration by a piezoelectric transducer that connect to a probe tip. The vibration from the tip cause cavitation due to sudden formation and collapse of bubbles within the fluid where high temperature, pressure and shock wave are involved. Due to millions of bubbles formation, there is an etching effect associate on fiber surface which help to eliminate the amorphous element from fiber. Some author reported the fruitful outcome of the physicochemical treatment which proves as a suitable technique to enhance mechanical properties of composite [7, 8, 9]. Motivating from the literature work, the present work focus on the combine treatment of HIU-NaOH and their effect on fiber fine structures as well as composites were investigated.

Materials and methods:

Sunn hemp fiber were obtained from Central Research Institute for Jute and Allied Fibers (CRIJAF), Barrackpore, Kolkata-700120, India. General purpose epoxy resin (L-12) and Hardener (K-6) were acquired from Himedia Laboratories Private Limited, India. For treatment of sunn hemp fiber, Sodium Hydroxide (NaOH) was collected from Merck Life

Science Pvt. Ltd, Mumbai, India.

At first, the sunn hemp fiber were cleaned with deionized (DI) water to remove the surface dust and air dried for 24 hrs to extract the moisture. This dried fiber were named as Raw Sunn Hemp (RSH) fiber. The fiber treatment procedure has shown in Fig.1.

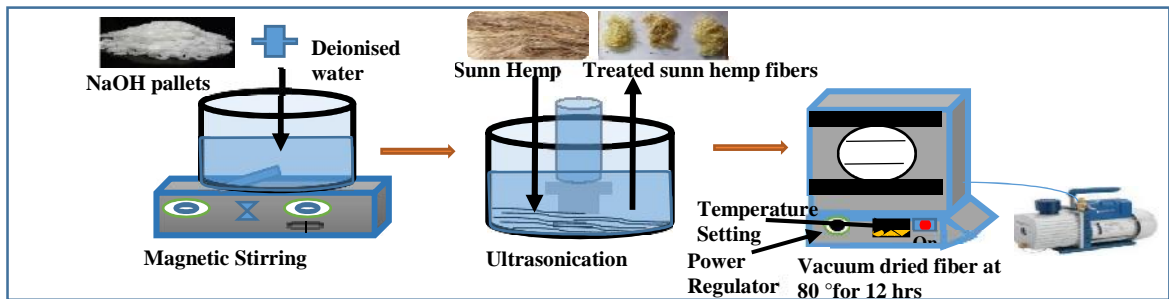


Fig.1 Treatment of Sunn Hemp fiber by HIU processor

For treatment, the RSH fiber were soaked in the 5% NaOH solution for 1hr in order to excite the reactive sites of cellulose hydroxyl groups. Aftermath, the fibrous solution exposed under HIU (OSCAR Ultrasonic Processor Sonapros, PR-1000) for 60, 90 and 180 minutes. Finally, the fibers were collected, washed and vacuum dried. The treated fibers were named as NRSH, NHIU1RSH, NHIU2RSH and NHIU3RSH where NHIU denote NaOH HIU treated RSH and the number denote the expose time period. X-Ray diffractogram patterns of untreated and treated RSH fiber were recorded by CuK radiation X-ray diffractometer (Ultima IV- Rigaku, Japan). Data were taken at 2θ range from $5^\circ - 50^\circ$ and scanning rate of $3^\circ/\text{min}$ with step size 0.05. Functional groups were investigated by Fourier Transmission Infrared (FTIR) spectroscopy (PerkinElmer Spectrum) from 4000 cm^{-1} to 400 cm^{-1} wave number and the surface morphology was carried out by using (Nova Nanosem 450, Japan) Field Emission Scanning Electron Microscopy (FESEM) at 10KV accelerating voltage.

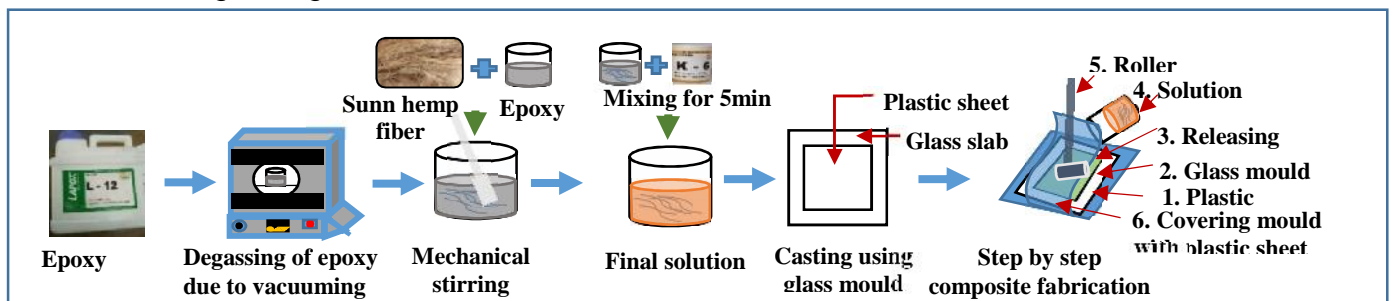


Fig.2 Composite preparation

The composites were prepared by Hand- lay out technique and the procedure has shown in Fig.2. The final solution was cured at room temperature. Two mould of dimensions (51×40×3) mm³ and (200×60×3) mm³ were prepared according to the ASTM-D3039 and ASTM-D7264 standard for flexural and tensile measurement respectively. Mechanical tests were carried out using a Universal Testing Machine (Instron-5967, UK) with Environmental Chamber. Specimens were cut with dimension (48×13×3) mm³ and (170×19×3) and tested at a loading rate of 5mm/min. The flexural strength of the composite were determined using the following equation.

Flexural Strength = $\frac{3FL}{2bd^2}$ where F= force at the fracture point, L= length of the support span, b & d= width and diameter of specimen

Results and Discussions:

XRD patterns for both untreated and treated RSH fibers have shown in Fig.3 (a). Two pronounced peak have appeared at 2θ angle of 22.6° and 15.6° which ascribe to crystalline cellulose I region where the minimum intensity of diffraction at 2θ of 18.6° ascribe to the presence of non cellulosic components in fiber. The calculated crystalline index (CI) [10] crystallite size (L) [12] for RSH fiber were found to be 72% and 31.2Å respectively. But after the treatment, the CI as well L were observed to be increased with increment of HIU vibration expose time period and showed the following trend RSH(72%, 31.2Å) < NRSH(80.6%, 42.6Å) < NHIU1RSH(80.7%, 42.7Å) < NHIU2RSH(81.4%,43.2Å) < NHIU3RSH(82.2%, 44.5Å). A very minimal increment of CI and L were seen for NHIU1RSH fiber than NRSH fiber and gradually increase with increase of expose time period. Highest CI and L value were observed in NHIU3RSH fiber. The etching effect caused by HIU vibration disintegrate cellulose within the fiber that eliminate the non cellulosic components from fiber. Due to treatment, the cellulose structure got modified by rearrangement of amorphous polymer chain in the diection of crystalline cellulose. So the combined treatment of HIU and alkali is effective to improve the crystallinity of cellulose polymer chain that can be an indication to enhance the strength of fiber. Increment in crystallinity lessen the chance of crystal defects that lead to increase the crystallite size.

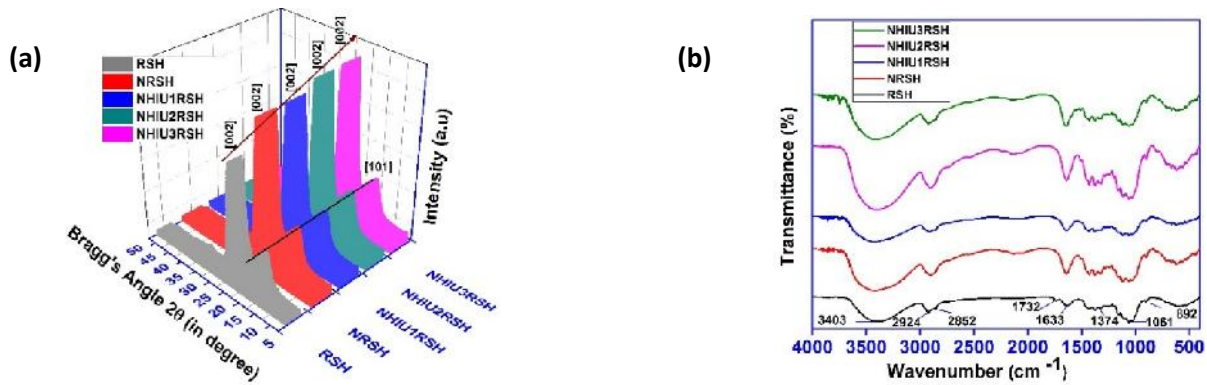


Fig.3 (a) XRD spectra (b) FTIR spectra of raw and NaOH+HIU treated sunn hemp fiber

FTIR spectra for both untreated and treated RSH fibers have shown in Fig.3 (b). The broad band around $3000\text{-}3500\text{ cm}^{-1}$ is referred to OH-stretching due to presence of hydrophilic substances in fiber. The peak at 2924 and 2852 cm^{-1} are due to C-H stretching which explain the presence of aromatic rings of lignin and wax respectively. But upon treatment, the lignin peak shifted for NRSH, NHIU1RSH, NHIU2RSH, NHIU3RSH and wax peak disappeared which indicate the removal of lignin and wax substances from fiber. Furthermore, peak at 1732 cm^{-1} in RSH fiber is due to C=O bond stretching which indicate the presence of hemicellulose but vanishes latter with treatment. Peaks at 1633 , 1061 and 892 cm^{-1} are due to O-H stretching due to absorbed water in cells, in plane C-O stretching due to carbohydrates and β -glycosidic linkage of holocellulose respectively. 1633 cm^{-1} peak have shifted towards higher wavenumber sides was observed in combined treated fiber due to lessening of hydroxyl groups. So, the combined treatment of RSH fiber was found to have good effect on elimination of amorphous compounds from fiber structure.

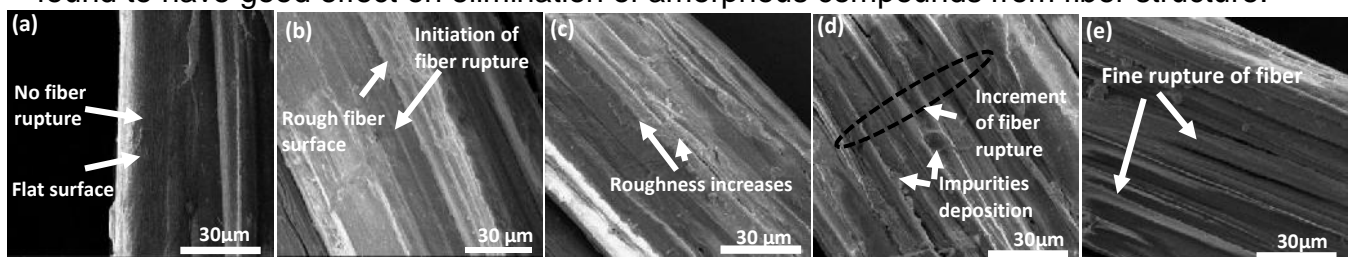


Fig.4 FESEM image of (a) RSH (b) NRSH (b) NHIU1RSH (c) NHIU2RSH (d) NHIU3RSH fiber

FESEM images for both untreated and treated RSH fibers have shown in Fig.4. Fig.4(a) shows a flat fiber surface with firmly arrangement of fibers due to presence of all chemical components in the fiber. On treatment with alkali, the fiber surface seem to be rough and fibrils starts to rupture as shown in Fig.4(b) which explain the removal of very least amount

non cellulosic compounds. In Fig. 4(c), the roughness of NHIU1RSH fiber surface increases. The fiber rupture increase in case of NHIU2RSH due to removal of more amount of inorganic materials as shown in Fig. 4(d). Finally a prominent fiber splitting with less impurities were observed in NHIU3RSH as shown in Fig. 4(e). This is due to reduction in fiber diameter by elimination of maximum amount of cementing materials that increased the effective surface area between fibrils. Therefore, the combined treatment of HIU and NaOH with high expose time strengthening the interfacial adhesion between fiber and matrix in composite.

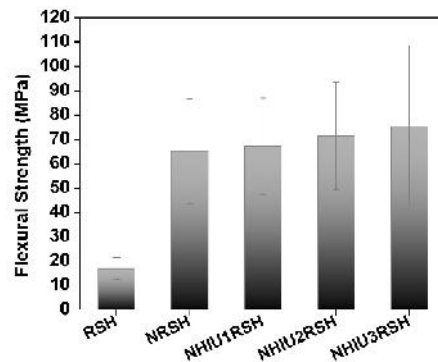


Fig.5 Flexural strength of untreated and treated RSH composite

Flexural strength (FS) for both untreated and treated RSH fibers composites have shown in Fig.5. It was observed that the combined treatment of HIU and NaOH show a better FS in comparison to RSH composite. The reason can be attributed to strong interfacial bonding between fiber and matrix due to formation of cavities and cell disintegration which causes the removal of cementing materials from fibers. Furthermore with increment of HIU vibration exposing time, the FS value is gradually increasing. This is due to the elimination of maximum amount of fiber impurities and increased surface area between the fibers. Along with this, the treatment increase the fiber aspect ratio by decreasing the fiber diameter which allowed the easy passage of matrix. A well chemical as well as physical bonding due to combined treatment of HIU and NaOH leads to a better FS in composite.

Conclusion:

In this study, the sunn hemp fiber was modified by means of combined treatment of HIU and NaOH where the time period of HIU vibration varied. XRD and FTIR study confirmed the increment of cellulose crystallinity and removal of non-cellulosic amorphous

components like hemicellulose, lignin, and pectin from fiber respectively. FESEM study of treated fiber showed a rough and ruptured fiber surface that increased the fiber surface area resulting better interfacial adhesion. The FS of combined treated fiber composite were found to be greater and more effective for NHIU3RSH composite due to better adhesion and increased cellulose crystallinity.

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