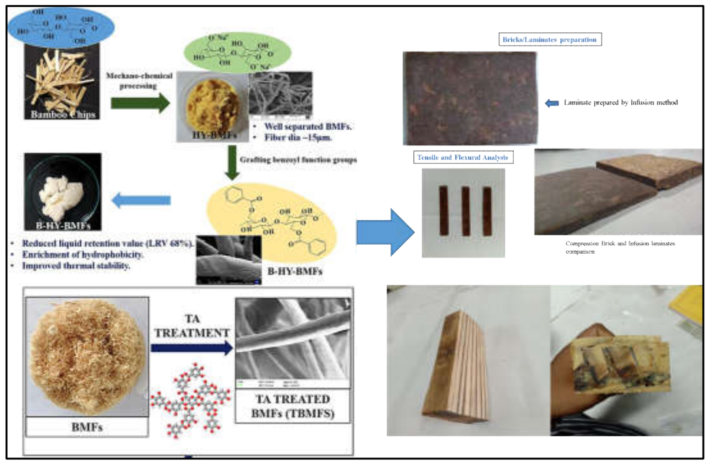
Standard Operating Procedure (SOP)
Bamboo Micron Fibres

NMHS Project: Bamboo bricks/laminates from BMFs (Bamboo Micron Fibres) for low cost housing structures for North Eastern Himalayan region

PI: Dr. S. Halder Department of Mechanical Engineering NIT Silchar Assam



This project was regarding the preparation of low cost housing structure for northeast Himalayan region. Objective of the project were to extract and treat BMFs from locally available bamboo culm and to prepare low cost and durable house by using BMFs. BMFs was extracted with an innovative Mechno-chemical process. Surface treatment of the BMFs was done using silanization, benzoylation and tannic acid. Bamboo strips laminates were prepared using BMFs and vinyl ester mixture as adhesive material. Flexural strength of 175.63 MPa was achieved for 10% BMFs in vinyl ester composite. One model house has been prepared with the help of these laminates at NIT Silchar. This research concludes that durable housing structure is possible with the help of bamboo fiber and laminated bamboo strip composite. More research is required to understand the environmental aspects on the BMFs laminates. The main problems with the traditional bamboo houses are:

- Once cut, insects may attack bamboo reducing its longevity.
- Durability and reliability as a structural material for building houses are less.
- In traditional housing, bamboo is in permanent contact with soil causing early degradation.
- Bamboo does not have an equal diameter over all its length and the thickness of the internal walls also varies. This results in only a few parts of the bamboo being useful
- Quality bamboo construction requires special techniques for joints and terminals.

The crux of this project was to overcome the above-mentioned problems by replacing the traditional bamboo housing with low-cost affordable shelter using bricks and laminates made from bamboo micron fibers. The aesthetics of such shelters are planned to make similar to that of buildings manufactured with costly traditional ingredients. The objectives and quantifiable deliverables of this project are mentioned below:

- Extraction of bamboo short micron fibers (BMFs) from the locally available bamboo column.
- Treatment of BMFs to reduce the environmental effect.
- Preparation of bamboo bricks and laminated boards.
- Investigation of bamboo bricks/laminated board performance with climate change.
- Sustainability analysis of bamboo bricks/laminated boards from BFFs.
- Techno-economic analysis of bamboo bricks/laminate boards.
- Demonstration of low cost and durable model house using bamboo bricks/laminates

Step 1: Collection of Bamboo:

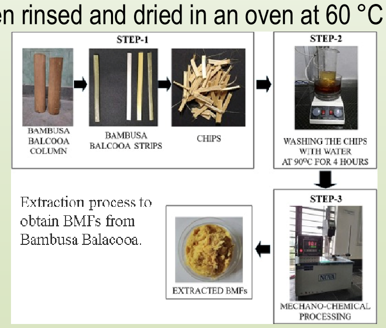
This section describes the insight into the optimization of processing parameters for the developed mechanochemical process to obtain high yield BMFs. It is well known that the use of alkali such as NaOH helps in the dissolving the lignin by forming a metal-lignin complex which is soluble in water. The reaction of lignin with NaOH results in the formation of sodium-lignate or Na-lignate. It was assumed that during the reaction process, the lignin present in the bamboo chips reacts with NaOH and the Na^+ ions get attached with the lignin by which bonding in the lignin is cleaved, resulting in the formation of lower molecular fragments akin to the observations as reported by others for different natural fillers. Furthermore, the mechanical agitation helps in removal of the Na-lignate from the chips and facilitate exposure of more fibers in the solution during processing, while the increase in reaction temperature can hasten up the reaction process. The above-said reaction between the chips and the NaOH can continue even after the fibers have been separated. However, long exposure could result in weak bamboo fibers as the alkali solution can degrade cellulose structures. Therefore, to attain maximum yield without much fiber weakening the reaction duration was fixed. Also, the reaction of the alkali and the lignin under high agitation is done to facilitate splitting the fibers to each individual strand of length ~3 – 5 mm. For the extraction of bamboo fibers, a green column of *Bambusa balcooa* was selected considering their soft nature that makes it easy to be machined manually. The node portion of the bamboo column was cut and removed first.

Step 2: Cutting:

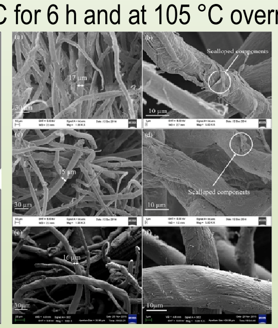
The columns were then cut longitudinally, and thick strips were prepared. From this, the inner wax rich layers, as well as the outer hard layers, were sliced off. It was evident that the fibers from the hard-outer layers are difficult to be processed for extraction, although the fibers are more densely packed at this region. Strips of ~2 mm thick bamboo were prepared from the previously prepared thick bamboo piece. Here the strips preparation was carried out manually by using an industrial blade.

Step 3: Extraction of bamboo micron fibers (BMFs):

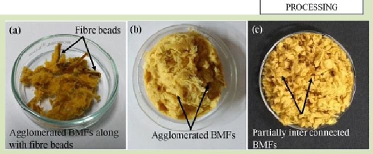
The extraction of BMFs from Bambusa Balcooa was accomplished in three steps. Under the first step, the hard epidermis outer layer and inner wax rich layer of the Bambusa Balcooa column was removed by mechanical stripping to obtain strips of bamboo. These strips were transformed to chips of size approximately 10 mm × 5 mm × 2 mm mechanically. In the second step, undesirable compounds from the chips were removed by soaking in distilled water for four hours at a temperature of 90°C. Dried chips were obtained by drying in a hot air oven at 70°C for 12 hours. Another washing was performed in acetone for the removal of water insoluble chemical compounds. In the last step, dried chips of ~10.5 g were initially treated in 17.2 wt.% NaOH aqueous solution. Finally, the resultant chips were processed under an innovative mechno-chemical process. The obtained fibers were then filtered by a mesh and washed several times by distilled water ensuring a pH value drop to 7. Fibers were then dried in an oven at 80°C for overnight as heating at higher temperature was found to burn the fibers on the surface as the fibers were not able to subsist the heating and the low evaporation rate at the same time. It was found suitable to make the drying at a lower temperature to yield a good result than to dry at the higher temperature. During the drying process, it was found that the fibers tend to agglomerate and create hard bits; which is most probably due to the presence of the hydroxyl ions on the fiber surface. Excess of moisture during drying tends to have this character. It was decided that the after the washing of the fibers, it was leached with ethanol in order to reduce the moisture content as well as improve drying. This lead to less agglomerated fibers and softer fibers. Fibers were then soaked in the solution for 30 min then rinsed and dried in an oven at 60 °C for 6 h and at 105 °C overnight.



Extraction process to obtain BMFs from Bambusa Balacooa.



Morphological evidence of BMFs (a) LY-BMFs at low magnification, (b) LY-BMFs at high magnification, (c) HY-BMFs at low magnification, (d) HY-BMFs at high magnification, (e) B-HY-BMFs at low magnification and (f) B-HY-BMFs at high magnification.



Fibers extracted at (a) low yield condition, (b) medium yield condition and (c) high yield condition.

Step 4: Mass Production setup for BMFs

Mass production setup was required for the production of the BMFs as it was a major requirement. As per the literature for obtaining the maximum percentage yield of BMFs extraction, some parameter has to be maintained. The mass production setup is made by combining three necessary parts:

- **Temperature Controlling Unit**: As per the literature best % yield of BMFs extraction was obtained at a temperature of 90 degrees Celsius. So A temperature control unit is used which can cut the power supply when the temperature reaches 90 degrees Celsius and start the supply of power when the temperature falls to 89 degrees Celsius. The thermocouple is used to measure the temperature as shown in the figure-8.
- **Heating Unit and drum:** A drum of volume 30 liters is used as a container in which a Mixture of strips and 15% NaOH and water will be stirred. A heating immersion road (1500KW) is fixed at the bottom of the tank and perfectly sealed for leakage. The arrangement is shown in figure-4.
- **Mechanical stirrer:** Mechanical stirrer is used for stirring the strips, water and NaOH solution at 2100 rpm. It was placed overhead to the drum as shown in the figure.

Step 5: Functionalization of Bamboo micron fibers

The surface functionalization of the extracted BMFs was implemented with silane oligomers, as well as with plant phenol in the form of tannic acid (TA). During surface functionalization using silane oligomers, the unordered ethoxy groups are expected to be aggregated on the surfaces of BMFs due to the interaction of the amino groups of APTES with the surface hydroxyl group or with the adjacent hydrolyzed APTES via hydrogen bonding as schemed in Figure below. This functionalization likely produces randomly protonated amino groups on BMFs and elevates siloxane condensation between APTES and the BMF surface or between APTES molecules. These silane moieties on BMFs may act as an adhesion promoter. To modify the fiber surface by benzoylation onto the BMF. This attachment was done by reacting BMFs in a solution containing 100 ml of 10 wt. % NaOH solution and 50 ml of benzoyl chloride by keeping the ratio of fiber weight to the solution at 1:10. The reaction was carried out under high agitation of 3000 rpm for 15 minutes at room temperature. The processed fibers were then filtered by using a sieve and soaked in ethanol for one hour to remove the unreacted benzoyl chloride. Filtration followed by washing with distilled water was performed and then dried in an oven at 105°C for overnight, to remove the moisture. For silanization of BMFs, the fibers were soaked for 24 h in ethanol and distilled water (in a ratio of 80:20) with 10 wt % of APTES to that of the weight of BMFs. The ratio of the fiber to the silane solution was kept at 1:40. The silanized BMFs (SBMFs) was filtered and washed with ethanol to remove the unreacted APTES and dried overnight in an oven at 105 °C. For TA coating of BMFs (TBMFs), firstly tannic acid (TA) solution at a 5 mM concentration is prepared. Then, 0.851 g of TA was weighed and mixed with 100 ml double distilled water in a beaker and stirred by at 200 rpm till it gets dissolved. After that tris buffer solution was added at a concentration of 10 mM. The pH of the resultant solution was adjusted to 8.5 by using HCl and NaOH. Fibers were then soaked in the solution for 30 min then rinsed and dried in an oven at 60 °C for 6 h and at 105 °C overnight.

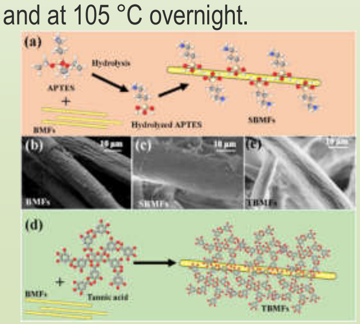
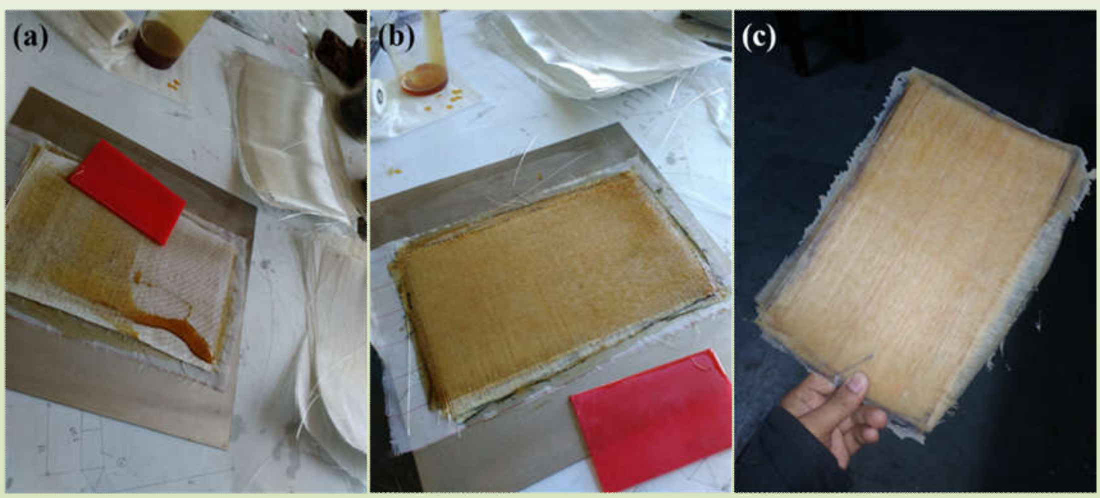


Fig. (a) Scheme showing siloxane condensation from APTES on BMFs. (b) Few scalloped structures on BMFs from FESEM morphology. (c) FESEM morphology of SBMFs depicting the appearance of a torn cell wall. (d) Scheme showing TA attachment on BMFs. (e) Smoother surfaces of TBMFs.

Step 6: Preparation of BMF Laminates

The preparation of GFRP laminates was carried out by hand layup method as shown in Fig.. A similar process for the preparation composite samples was carried out for making the matrix of the BMF based hybrid laminates. Epoxy of 60 grams was taken in a polypropylene glass and 95% weight of hardener to that of epoxy was mixed to it. Then it was mixed for 10 minutes and accelerator DY070 was taken at 1.5 wt.% of the epoxy and was added to it. The mixture was then mixed for another 10 minutes to get a uniform mix and bare and surface treated BMFs were at predetermined 1, 2.5 and 5 weight % to epoxy was added and samples were prepared. For preparing BNP laminate samples, epoxy resin LY556 was taken in a beaker and the particular weight ratio of the milled BNP was mixed. After mixing it with a mechanical stirrer at 3000 rpm for 15 minutes, the mixture was subjected to ultrasonication for 15 minutes at 70% amplitude at 10 seconds pulse-on and 30 seconds pulse-off time. Then harder, HY906 and accelerator DY070 was added to the mixture. The mixture was mixed again using a high-speed mechanical stirrer for 30 minutes to get a homogenous mixture and degassed to remove the trapped air due to aeration caused by high-speed mixing. A total of 8 plies of bi-directional 180 gsm glass fabric was used. A steel plate was taken to be the base material and an overhead projection (OHP) sheet made of cellulose acetate is taken and was fixed on the steel plate. The OHP sheet acts as a mold release film and samples can be easily removed from it. The hand lay-up process was carried out by applying the epoxy between the successive glass fabrics. Two major issues were observed during this preparation. Initially, it was observed that the epoxy tends to flow out of the laminate during the initial heating. At 80oC, gelation of the epoxy occurs. This can drain the matrix out of the laminate. To overcome this, a boundary of high-temperature butyl rubber was applied according to the laminate size. Secondly, the laminate tends to warp and bend during heating. A steel plate of suitable weight was put on top of the laminate after the hand layup process to have a flat laminate sample. The laminate was cured with curing cycles of 2 hours at 120°C + 8 hours at 160°C. The dimensions of the prepared laminate samples were made in such a way that the all the mechanical testing can be carried out from a single laminate sheet.



Preparation of hybrid GFRP laminates by hand lay-up process (a) applying of epoxy mixed with bamboo filler (b) finished stacking up of the layers before curing process and (c) finished GFRP laminate sample.

Step 7: Preparation of Bricks/Laminates

Bricks were prepared using mould and hydraulic press. Firstly neat fibre is compressed to make the brick without resin. The neat BMFs brick is shown in figure.

The second brick was made by mixing the BMFs in Epoxy LY-556 resin. Hardener HY-906 (95 %) and accelerator Dy070 (2 %) were mixed in the resin. The BMFs-Resin mixture was compressed at 10 kN force for a Brick sample size of 100mm×100mm×7mm. The Brick sample is shown in figure.

BMFs-Epoxy resin Brick of size 100mm×100mm×7mm.

Step 8: Preparation of BMFs laminates

BMFs laminates were prepared by using the vacuum assisted resin infusion molding (VARIM) process. First of all the fibre is compressed in the mould with the help of a hydraulic press. After that, the resin is infused in the compressed neat fibre laminate. This process involves the good distribution of resin as a comparison to the compression method. The BMFs laminates are shown in figure.

FBMFs laminates and its comparison with BMFs brick

Step 9: **Vertical strips laminates (For beam and column)**

Thick laminate sample: Combining the strips of bamboo vertically and adhering them using some resin or Fevicol was the idea. For this two types of work were done. One is using thick bamboo strips and the second was using vary thin bamboo strips. Locally available bamboo (Bambusa Balcooa) is cut in pieces from nodes. Every node is divided into six equal strips with the help of a sharp tool (bill hook or locally called as Dao). These strips are curved in shape, so using a fine Dao; it is converted into a rectangular strip. After this, these strip has been heated up in the water at 90 degrees for two hours to remove unwanted compounds. After this, the strips are dried at 90 degrees for 6 hours to remove moisture contents. All surfaces are planned with the help of an electric hand planner. Tannic acid treatment was done on the strips as well as on the fibers. For fibre 2.5 g of tris buffer is added into 1000ml DI water and stirred at 400 rpm. The pH value of the solution was noted as 10.2. HCl is added with the help of a dropper (2 drops) to maintain the pH value to 8(~7.94). 10 g of tannic acid is added to the solution and stirred at 400 rpm for 15 minutes. 100 g of BMFs added to the solution and kept for 6 hrs. The BMFs dried at 90 degrees in the oven. For strips, 400 g of Tannic acid is added in 4 liters of DI water to make 10% composition and Stir at 2000 rpm by mechanical stirrer. Bamboo strips were dipped in the solution for 6 hours and after that strips were dried in the oven at 90 degrees. Strips were arranged in sequence by placing different joining adhesives between them. These strips were pressed with the help of a hydraulic press and kept for 4 hours. After this samples were planned using an electric hand planner. BMFs or TBMFs were mixed with resin (Epoxy L-12 and synthetic rubber adhesive) manually by hand. The samples are shown in figure.

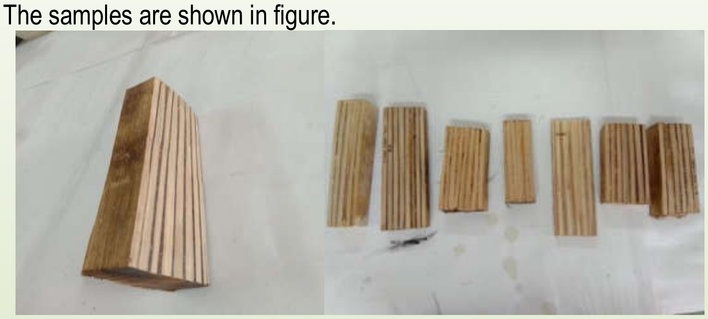


Figure: Vertical strips laminates

Mixed strips laminates

For the preparation of long beam and column, large strips of bamboo adhered horizontally as well as vertically. Firstly strips have adhered vertically with the help of vinyl ester and BMFs (10%) mixture. After that strips are joined with the help of Fevicol over joints. It will increase the joint strength of the laminates. The strips are shown in figure-27

Figure: Mixed strips laminate (Fevicol and BMFs + Vinyl ester adhesive)