Effect of Addition Microcrystalline Cellulose on Mechanical Properties of Jute/Glass Fibers Hybrid Laminated Composite

Jamasri 1*), Ferriawan Yudhanto 1,2)

1) Departement of Mechanical and Industrial Engineering, Universitas Gadjah Mada, Jl. Grafika No. 2, Yogyakarta 55281, Indonesia (* Corresponding author, E-mail: jamasri@ugm.ac.id)
2) Department of Mechanical Technology, Universitas Muhammadiyah Yogyakarta, Jl. Brawijaya, Kasihan, Bantul, Yogyakarta 55183, Indonesia

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ABSTRACT: This study aims to investigate the effect of chemical treatment of Jute fibers and the addition of MCC (microcrystalline cellulose) on the strength of the hybrid laminated composite (HLC). The HLC was fabricated using the press mold method with a pressure of 2 bar. The physical characteristic was evaluated by SEM, FTIR and XRD. The results show that the MCC’s filler addition to treated jute/glass fibers increased the interlaminar bonding. The HLC has the highest mechanical strength by a combination of treated jute/glass (JGJGJG), in which the resulted in tensile and flexural tests are 73 MPa and 183 MPa, respectively.

KEY WORDS: MCC, treated jute fiber, glass fiber, hybrid laminated composite [D3]

1. Introduction

Applications of laminated composites in vehicles have been developed, especially using synthetic fibers such as carbon fibers, glass, aramid, basalt, etc. Synthetic materials were widely used because of their properties such as high mechanical strength, good durability, high thermal stability, and suitable at chemical conditions. The use of synthetic materials has many impacts on the environment, especially after the vehicle becomes waste; some of the components must be recycled. The natural fiber is an abundant source of lignocellulose and an alternative for reducing synthetic materials. It is environmentally friendly, biodegradable, low cost, low density, good thermal resistance, and bio-compatible. Hybrid composites basically combine two or more materials with different properties, types, and sizes. The use of synthetic and natural fibers as composite reinforcement is a good combination considering both of them have advantages and disadvantages of the fiber’s basic properties as reinforcement.

Fig. 1 shows the use of a hybrid laminated composite in the body panel on a Mercedes Benz. The company has developed hybrid composite using hemp and glass fibers reinforced by polypropylene (PP). Hybrid composite was also manufactured for automotive components such as dashboards, door trim, door panels, seat back panels, etc. This composite manufacturing process usually uses compression molding and vacuum infusion methods.

Nawangsari et al.(15) developed the organic non-asbestos brake with the addition of microcellulose. The brake material consists of microcellulose, PAN (polyacrylonitrile), and rock wool, which increases the resistance coefficient of friction on high temperature (623 K) and reduces friction sound, and restores brake of performance.

Mishra et al.(6) has studied the structure, composition, and properties of sisal, and its resulted optimization of chemical modification by alkaline treatment 5 wt.% NaOH. This alkali treatment’s effect interlaminar bonding between fiber and matrix. It can increased the tensile and bending strength of sisal fiber treated-polyester by 28% and 7%, respectively, compared to untreated sisal-polyester.

Fig. 1 Hybrid laminated composite panel of Mercedes Benz Car from Hemp fiber (http://www.globalhemp.com/2011/02/automotive-composites.html)

Joseph et al.(7) carried out various modifications for the fiber surface by alkaline treatment using NaOH and KMnO4 on sisal short fibers with size of 6 mm length. The sisal fiber-reinforced LDPE (low-density polyethylene) resulted in increased tensile strength of 10% for NaOH and 25% for KMnO4.

Das et al.(8) performed alkali treatment on the bamboo fibers. The fiber immersion at various concentrations of 10, 15, 20, and 50 wt.% NaOH for 1 hour at room temperature with a ratio of fiber and solution of 1:15. The results showed that the best physical properties was at 15 wt.% NaOH which indicate an increase of CI
(crystallinity index) from 45.57% to 51.48%. However, the increase in concentration of 20% and 50% wt. % NaOH caused the CI to decrease to 47.55% and 17.82%, respectively. The decrease of CI indicated the damage of crystalline structure in cellulose.

The chemical treatment of jute fiber has been carried out by Wang et al.\(^{(9)}\). The Jute fiber was composed of three main lignocellulosic component structures, namely cellulose (58-63%), hemicellulose (20-24%), lignin (12-15%), and some other small substance, i.e., pectin, fats, aqueous extract, etc. Chemical treatment (alkalization-bleaching) process consisted of a combine of sodium hydroxide (NaOH) by 1.6 wt.%, sodium silicate (Na\(_2\)SiO\(_3\)) by 0.5 wt.%, and sodium sulfite (Na\(_2\)SO\(_3\)) by 0.4 wt.% at 368°K along 120 minutes. It followed by bleaching using hydrogen peroxide (H\(_2\)O\(_2\)) by 1.2 wt.% and sodium silicate (Na\(_2\)SiO\(_3\)) by 0.3 wt.%, pH 10, at a temperature of 363°K along 90 minutes. The FTIR (Fourier Transform Infrared) and SEM (Scanning Electron Microscopy) physical tests showed that the content of hemicellulose and lignin on the jute fibers' surface were decreased. The XRD test showed an increase in the CI from 68.89% to 76.26%. Increasing the CI indicates that an increase in the amount of cellulose content.

The bleaching treatment using hydrogen peroxide on the agave cantala fiber was carried out by Yudhanto et al.\(^{(10)}\). Optimization for the combination treatment of alkali-bleaching was performed using the Taguchi method. The result was obtained for the optimal process in the alkali treatment using 5 wt.% NaOH solution at a temperature of 373°K for 1 hour then continued with bleaching using a 3 wt.% H\(_2\)O\(_2\) solution, pH 10, at a temperature of 333°K for 1 hour. This chemical treatment causes a 50% increase in the tensile strength of the single fiber.

Modification by adding fillers from synthetic materials such as fly-ash particles, carbon, clay, ceramics (Al\(_2\)O\(_3\), SiO\(_2\), TiO\(_2\), and Fe\(_2\)O\(_3\), etc. has been widely practiced in the composite industry.\(^{(11)}\) These materials are expensive and potentially harmful for the environment. The benefit of adding filler in polymer is to improve mechanical interlocking and create an interlaminar bonding. The use of biodegradable and environmentally materials is the best solution.

The natural fibers used as reinforcement or as filler is interesting for further research. The filler of Cellulose nanofibers (CNF) powder from the Helicters Isora plant was investigated by Chiraiyl et al.\(^{(15)}\). CNF powder as a filler in polyester was carried out with various weight concentrations of 0.5, 1, 3, and 5 wt.%. The best results were achieved for the addition of 0.5 wt.% CNF powder, which resulted in an increase in tensile strength and elongation break of the polyester composite by 57% and 17%, respectively. The addition concentration causes CNF agglomeration in which the stress concentration becomes uneven. This effect reduces the mechanical strength of the polyester composite. The addition of CNF powder filler also reduces water absorption in the composites.

### 2. Materials and Methods

#### 2.1. Materials

The materials used in this study were synthetic glass woven fiber (CWR-200), woven jute fibers, and UPE (unsaturated polyester) Yukalac 157 BQTN-EX which was obtained from PT. Justus Sakti Raya (Chemicals Manufacturer), Indonesia. The properties of UPE resin can be seen in Table 1. Chemicals treatment (alkali-bleaching) was consisted of NaOH (sodium hydroxide) and H\(_2\)O\(_2\) (hydrogen peroxide) solutions. The purity of NaOH and H\(_2\)O\(_2\) is 98% and 50%, respectively. MCC (microcrystalline cellulose) powder MERCK CAS-No: 9004-34-6 (Fig. 2), Darmstadt, Germany was obtained from CV. Multi Indonesia, Yogyakarta, Indonesia.

![Fig. 2 Microcrystalline cellulose powder](image)

### Table 1 Properties of cast-cured UPE (Unsaturated Polyester) Yukalac 157 BQTN-EX

| Item                              | Unit   | Typical Value | Note   |
|-----------------------------------|--------|---------------|--------|
| Specific Gravity                  |        | 1.215         | 298°K  |
| Gel Time                          | minutes| 20-30         | 303°K  |
| Heat Distorsion Temperature       | °K     | 343           |        |
| Water Absorption (room temp. 298°K) | %     | 0.188         | 24 hours |
| Flexural Modulus                   | MPa    | 92            |        |
| Tensile Strength                   | MPa    | 54            |        |
| Tensile Modulus                    | GPa    | 2.9           |        |
| Elongation                         | %      | 1.6           |        |
| Compressive Strength               | MPa    | 123           | ISO-604|

#### 2.2. Chemical Treatment of Jute Fiber

The chemical process on jute fibers was alkali and bleaching using a solution of NaOH and H\(_2\)O\(_2\). The fibers were immersed in 5 wt.% NaOH solution at room temperature (300±3)°K for 4 hours, then continued soaking in 3 wt.% H\(_2\)O\(_2\) solution at a temperature of 333°K, pH 10, for 1 hour\(^{(10)}\) with the ratio of fiber weight to the solution was 1:50. The fibers were then neutralized with distilled water repeatedly until reaching a neutral condition (pH 7). Drying of fibers was carried out in open condition for 1 day and continued in the oven for 2 hours at a temperature of 373°K.

#### 2.3. Hybrid Laminated Composite Fabrication

The hybrid laminated composite was fabricated used a press mold method, with a pressure of 2 bar. The molding material was made from A36 steel in which the size was fitted to the desired of HLC panel. The MCC filler of 0.5 wt.% was mixed using a mechanical stirrer in several stages for 20 minutes with speed of 350 RPM. The selection of low rotation speed aims to avoid air bubbles when mixing into the unsaturated polyester (UPE)\(^{(18)}\). MEKPO (Methyl Ethyl Ketone Peroxide) was used as hardener in

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UPE with percentage of 1% based on volume. Glass woven fibers and jute woven fibers were cut into rectangles size of 250 x 250 mm and arranged stratified in an arrangement as shown in Table 2 (note: Jute woven fibers are coded J and glass woven fibers are coded G). The different density of glass fiber (2.6 g/cm³) and jute fiber (1.4 g/cm³) causes the different thickness and volume fraction (Vᵢ) on hybrid laminated composite (HLC)(19).

| No. | Stacking sequence | Number of layer Glass fiber | Number of layer Jute fiber | Specimen Thickness (mm) | Vᵢ (%) |
|-----|-------------------|-----------------------------|----------------------------|-------------------------|---------|
| 1   | GGGGGG           | 6                           | 0                          | 2.26                    | 25.7    |
| 2   | JGGGGJ           | 4                           | 2                          | 3.04                    | 27.6    |
| 3   | JJGGGJ           | 3                           | 3                          | 3.06                    | 28.3    |
| 4   | JJJJJJ           | 0                           | 6                          | 3.80                    | 30.5    |

2.4. Physical Characterization

2.4.1. SEM (Scanning Electron Microscopy)

SEM was used to observe MCC’s morphological structure of raw jute fiber before and after chemical treatment. The SEM instrument was used Thermoscientific Quanta 250 model, which can enlarge the target with a magnification up to 20,000x.

2.4.2. FTIR Characterization

The FTIR (Fourier Transform Infrared) model used in this research was Shimadzu IR Prestige-21, working in the range of 400-4000 cm⁻¹. The pellet sample preparation was made by mixing potassium bromide (KBr) pellet and then pressing. The purpose of the FTIR test is to indicate the chemical groups. The content of MCC and jute cellulose can be predicted by this instrument. Besides, the wave peak also provides an information on the degradation of chemical group bonds at a certain peak so that the loss of noncellulosic material in the jute fiber due to chemical treatment can be observed.

2.4.3. XRD (X-Ray Diffraction) Characterization

The XRD test was carried out using the Rigaku Miniflex 600 with power of 40 kV and 20 mA. The crystallinity index was calculated using the formula introduced by Segal et al. (20):

\[ CI = \frac{I_{002} - I_{amorphous}}{I_{002}} \times 100 \]  

(1)

Where, I₀₀₂ is a crystalline structure region of cellulose (2θ=22°) and Iₐₐₘₒᵣₚₖᵢₛ is a non-cellulosic or amorphous region (2θ=18°) of the material.

2.5. Mechanical Characterization

Mechanical testing of HLC composites consisted of tensile and flexural tests, which refer to ASTM D630 and ASTM D790, respectively. Tensile testing used UTM (Universal Testing Machine) such as shown in Fig. 3, the dimension of HLC specimens was 50 mm in length and 13 mm in width. In addition, the thickness was varied depending on the type of stacking sequences. The speed rate of the UTM machine for tensile test was set at 2 mm/minute.

Flexural testing with a three-point method (Fig. 4) refers to ASTM D790 with the dimension of 12 mm specimen width and 56 mm bottom span length. The thickness of specimens was varied such as the tensile specimens. The recommended span length is at least 20 times the thickness of the test specimen. The speed rate of the UTM machine for flexural test was set for 1 mm/minute.
3.2. XRD Characterization

The XRD test results for MCC show a high crystallinity index of 77% (Fig. 7). The previous study of Wang et al.\(^9\), showed that a Jute fiber treated with alkali and bleaching had increased the crystallinity index by approximately 76.26%. The three prominent peaks at \(2\theta = 16.49^\circ, 22.84^\circ, \) and \(34.06^\circ\) represent the planes (111), (002), and (310), respectively\(^{4,24}\). The highest peak at \(2\theta = 22^\circ\) shows the crystalline structure of the cellulose. The high CI value suggests an increase in cellulose content. The increased cellulose content will increase chemical bonding, mechanical interlocking and thermal stability\(^{3,4}\).
3.3. FTIR Characterization

Fig. 8 shows the chemical bonds in the jute fibers, MCC, and UPE (unsaturated polyester). This bond is indicated by the wave crests in the wave number region from the range of 500-4000 cm⁻¹. The presence of cellulose in jute fibers shows three prominent wave peaks, namely 3435-3448 cm⁻¹ (O-H stretching), 2900-2924 cm⁻¹ (C-H stretching), and 1056 cm⁻¹. The fibers that had been treated with alkaline and bleaching showed a loss of peak at 1736 cm⁻¹ (C=O and 1250 cm⁻¹ C-O stretching). Both peaks indicate the presence of hemicellulose and lignin. Alkali treatment and bleaching are the effective way in removing these two components.

A peak of 1635 cm⁻¹ indicates absorbed water in the fiber. It can be seen that jute fiber before and after treatment. MCC has a relatively high water content because polyester is hydrophobic. In the UPE, there is a peak at 1635 cm⁻¹, which indicates that polyester is hydrophobic. For better application of jute and MCC, fibers need to be dried before being used as reinforcement and filler in the composites.

3.4. Tensile Strength

Fig. 9 shows the comparison of tensile strength and elongation at the break due to the effect of adding MCC and chemically treatment of jute fibers. The MCC causes the mechanical strength of laminated composite slightly increased. The addition of MCC in neat glass-fiber composites (GGGGGG) is increased by 10%. The hybrid treated jute/glass composites, i.e., J(JGJJGJ) and J(JGIGIGG) are increased by 7%–11%, and the highest increase is obtained in neat treated jute composite (JJJJJJ) by 17%. The neat jute composite has lowest strength due to the hydropathic nature properties still exist in the jute fiber’s surfaces. This hydropathic nature causes the voids on the laminated composite product. For untreated fibers, the addition of MCC doesn’t have any significant impact. The reason is that the MCC doesn’t bind nicely, whereas the raw fibers are still covered by wax, hemicellulose, and lignin so it doesn’t influence the mechanical strength of HLC. The increase of HLC volume fraction also doesn’t affect the mechanical properties.

The addition of MCC and chemically treatment significantly affects the elongation of treated jute composite (JJJJJJ) by 71%. In contrast, for the HLC of J(JGJGJJG) and J(JGJGIGG) is increased slightly by 12% and 22%, respectively. The addition of MCC doesn’t significantly affect on elongation at break of glass fiber composites (GGGGGGG), it is only increased by 5%.

3.5. Flexural Strength

Fig. 10 shows the comparison of the flexural strength and deflection of HLC with different stacking sequences. The addition of MCC and chemical treatment have a very significant effect on the flexural strength of HLC. The composite of glass woven fibers (GGGGGGG) is increased by 22%. The J(JGJGJJG) which consists of two treated jute and four glass woven fibers, and J(JGJGIGG) which consists of three treated jute and glass woven fibers significantly increase the strength by 45% and 65%, respectively. The highest result is obtained in the neat treated jute composite (JJJJJJ), which is increased by 115%. The flexural strength of HLC is significantly higher compared to the tensile strength which is most possibly caused by the increase of the interlaminar shear strength due to the fibers chemical treatment. The chemical treatment on the jute fiber has changes the crystal structure of alpha-cellulose (Iα) to beta-cellulose (Iβ) in the molecular group. The hydroxyl group of Iα has a strong double hydrogen bond interlaminar cross-linked.

The addition of MCC and chemically treatment into the HLC causes the increase of deflection. The highest increase is achieved in the neat treated jute composites (JJJJJJ) by 191%, while in the hybrid treated jute/glass composites, (J(JGJGJ) and J(JGIGG), they are increased by 80% and 164%, respectively.
The smallest result of deflection is obtained by neat glass fiber composite (GGGGGG), which is only increased by 32%. The high crystallinity structure causes the excellent molecular bond of hydroxyl (-OH) groups between the MCC and fibers. Furthermore, the surface roughness on the treated jute fiber significantly influence the interlaminar shear strength at the flexural test.

3.6. SEM Image of HLC

The bonding between fibers, MCC and polyester were observed by photo SEM. Fig. 11 (a) shows that MCC filler doesn’t adhere well to glass fibers which is possibly caused by a weak interlaminar bonding of neat glass fiber. Fig 11 (b) shows that MCC in the treated jute fiber adheres well to the UPE matrix. It is due to the highest crystallinity index of MCC and treated jute fibers that indicates the both have a high cellulose content. The high cellulose content and roughness of surfaces area of treated jute fibers can improve the mechanical strength of hybrid laminated composite materials for automotive body application.
4. Conclusion

From the results and discussion, it can be concluded as follows.

1. The MCC's addition to treated Jute/Glass fibers laminated composite increases the tensile strength by 7-11% and the most significant increase is achieved in flexural strength by 80-164%.

2. The HLC has the highest mechanical strength by combination of three glass fibers and three treated jute fibers (JGGII) in which the resulting tensile and flexural tests are 73 MPa and 183 MPa, respectively.

3. SEM image shows the jute bundle fibers separated into smaller fibrils and the hemicellulose and lignin on the surface of the fibers are removed. In addition, XRD test results for MCC show a high crystallinity index of 77%.

4. From FTIR spectra, it can be seen that alkali treatment and bleaching can remove the presence of hemicellulose and lignin.

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