Data Article

Dataset on mechanical, thermal and structural characterization of plant fiber-based biopolymers prepared by hot-pressing raw coconut coir, and milled powders of cotton, waste bagasse, wood, and bamboo

Mohammed Abdullah Hamad Alharbi*, Shinji Hirai, Hoang Anh Tuan, Shota Akioka, Wataru Shoji

Research Center of Environmentally Friendly Materials Engineering, Muroran Institute of Technology, Muroran 050-8585, Japan

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**ABSTRACT**

This article presents experimental data on visual, mechanical, thermal, and structural characterization by hot-pressing four sources of milled plant powders and coconut fibers. It correlates chemical composition obtained by (FTIR), particle size, and reports bending strength, water resistance morphological (SEM) and thermal stability, structural properties (FTIR and XRD). It further supplements findings of the influence of microfibrillation and chemical composition on hot-pressing plant fibers as presented in the research article “Effects of Chemical Composition, Mild Alkaline Pretreatment and Particle Size on Mechanical, Thermal, and Structural Properties of Binderless Lignocellulosic Biopolymers Prepared by Hot-Pressing Raw Microfibrillated Phoenix Dactylifera and Cocos Nucifera Fibers and Leaves” [1]. For more insights into the difference among non-lignin-, lignin-, and semi lignin-based adhesion refer to the research article [1]. This dataset is

* Corresponding author.
E-mail address: mohammedahalharbi@gmail.com (M.A.H. Alharbi).

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Specifications table

Subject | Materials Science
Specific subject area | Lignocellulosic biopolymers
Type of data | Images, Figures, Tables
How data were acquired | Scanning Electron Microscopy (SEM) micrographs captured using (JSM-6510, JEOL, Ltd., Japan), coated with thin layer of platinum using a magnetron sputter (MSP-1S, SHINKUJ, Tokyo, Japan), Tensile tester with 1 kN maximum loading capacity (Autograph AGS-X, Japan), analytical balance (AUX120, SHIMADZU, Co., Japan), thermogravimetric analyzer (EXSTAR TG/DTA 6300, Seiko Instruments, Inc., Japan), FTIR (ATR) (JASCO FT/IR-6600, JASCO Co., Japan), X-ray diffraction (XRD) (Ultima IV Protectus, Rigaku Co., Japan), graphing, data and statistical analysis (OriginPro, Version 2019b. OriginLab Co, USA), images (iPhone Xs Max, Apple Inc., USA)

Data format | Raw data, Analysed data, images
Parameters for data collection | Microparticles of cotton, recycled waste wood, bamboo and bagasse, and fibers of coconut coir were hot-pressed at 20 MPa until the optimum temperature was achieved. Subsequently, specimens were gradually cooled at room temperature and dried in an oven at 100 °C for 48 h before testing. Thereafter, the obtained samples were subjected to visual, mechanical, thermal, and structural characterization.

Description of data collection | Bending properties determined based on testing flexural properties JIS-K-7171 [2]. Density determined using Archimedes’ Principle. Water absorption and thickness swelling calculated according to JIS-A-5905, sections 7.10 and 7.11, respectively [2]. Statistical analysis determined using ANOVA. TGA/DTG measured at heating 10 °C/min to 700 °C under air. FTIR spectra measured in the range 700–4000 cm-1 at 4 cm-1 resolution and 45 scans. FTIR data acquired and analyzed according to methods [1,3–5]. XRD of samples were scanned at 0.2°/min in the 2θ range 5–50°, and analyzed according method [6].

Data source location | Institution: Muroran Institute of Technology
City/Region/Town: Muroran-shi, Hokkaido
Country: Japan

Data accessibility | The raw and processed data required to reproduce these findings are available with the related research article [1] and available to download from Mendeley Data http://dx.doi.org/10.17632/ng5289v2y8.3.

Related research article | Mohammed Abdullah Hamad Alharbi, Shinji Hirai, Hoang Anh Tuan, Shota Akioka, and Wataru Shoji
Effects of chemical composition, mild alkaline pretreatment and particle size on mechanical, thermal, and structural properties of binderless lignocellulosic biopolymers prepared by hot-pressing raw microfibrillated Phoenix dactylifera and Cocos nuciferafibers and leaves
Polymer Testing
DOI:https://doi.org/10.1016/j.polymertesting.2020.106384 [1]

Value of the data

- The data are useful characterization for determining high-performance biopolymers using other various plant sources.
- The data help replicate processing conditions to produce lignin-based adhesion lignocellulosic biopolymers from other various plant sources.
Table 1
The prepared samples, their labels and processing conditions.

| Sample code | Raw material | Raw materials form (size) | Moisture content (%) | Ash content (%) | Hot-pressing temperature (°C) | Hot-pressing time (min) |
|-------------|--------------|---------------------------|----------------------|-----------------|------------------------------|------------------------|
| CCPB        | Coconut coir | Fibers (5 – 10 cm)        | 9.13                 | 4               | 180                          | 7–9                    |
| Cotton      | Commercial cotton | Microparticles (≤75–106 μm) | 6.25                 | 4               | 180                          | 7–9                    |
| Bagasse     | Bagasse waste | Microparticles (53 – 75 μm) | 14.54               | 7.57            | 140                          | 3–4                    |
| Waste wood  | Recycled waste wood | Microparticles (53 – 75 μm) | 7.96                 | 5.7             | 140                          | 5–7                    |
| Bamboo      | Waste Bamboo | Microparticles (~75 μm)    | 3.10                 | 5.7             | 140                          | 4–6                    |

Table 2
Mechanical and physical properties.

| Lignocellulosic Biopolymer | Bending strength (MPa) | Strain at break (%) | Young’s modules (GPa) | Density (g/cm³) | Water absorption (%) | Thickness swelling (%) |
|---------------------------|------------------------|---------------------|-----------------------|-----------------|----------------------|------------------------|
| CCPB                      | 71 ± 11                | 2.35 ± 0.10         | 3.54 ± 0.25           | 1.33 ± 0.01     | 35 ± 3               | 26 ± 3                 |
| Cotton                    | 24 ± 4                 | 0.91 ± 0.20         | 3.13 ± 0.55           | 1.50 ± 0.02     | –                    | –                      |
| Bagasse                   | 80 ± 4                 | 1.22 ± 0.10         | 6.24 ± 0.35           | 1.51 ± 0.01     | 32 ± 4               | 38 ± 6                 |
| Waste wood                | 37 ± 4                 | 1.37 ± 0.55         | 3.00 ± 0.49           | 1.34 ± 0.01     | 59 ± 13              | 46 ± 11                |
| Bamboo                    | 40 ± 9                 | 1.20 ± 0.16         | 4.51 ± 1.05           | 1.20 ± 0.03     | 61 ± 20              | 33 ± 10                |

• These data benefit other researchers to utilize lignin-based adhesion as a useful adhesive in environmentally friendly materials.
• FTIR, TGA/DTG, and XRD data are useful methods for predict chemical composition in hot-pressed plant fibers and their adhesion.

1. Data description

Photographs of raw materials used in experiments: small fibers of coconut coir, milled powders of cotton, recycled waste bagasse, wood, and bamboo and appearance of their corresponding hot-pressed biopolymers are shown in Fig. 1. The prepared samples, their labels, and processing conditions are given in Table 1. Table 2, shows the mechanical and physical properties. SEM micrographs captured of microparticles and biopolymers captured at 190, 600, and 1200 × magnifications are shown in Fig. 2 and Fig. 3, respectively. TGA and DTG curves generated to determine the thermal stability of hot-pressed plant-based biopolymers and milled powders, and to obtain moisture and ash contents of milled powders are shown in Fig. 4. The thermal properties of hot-pressed plant-based biopolymers are shown in Table 3. FT-IR spectra of the obtained biopolymers are shown in Fig. 5. Table 4, shows specific absorption-band wavenumbers (cm⁻¹) related to biopolymers of FT-IR band assignments (cm⁻¹) numbered in the research article [1]. The summation of major peak areas in the “fingerprint” region associated with cellulose, lignin, hemicellulose and pectin in lignocellulosic biopolymers are shown in Table 5. XRD diffraction patterns of hot-pressed biopolymers and assignment of X-ray diffraction peaks for the obtained biopolymers are given in Fig. 6 and Table 6, respectively. The raw and processed data required to reproduce these findings are available with the related research article [1], and available to download from http://dx.doi.org/10.17632/ng5289v2y8.3.
Fig. 1. Photographs of raw materials; (a) coconut fibers, (b) cotton, (c) bagasse, (d) waste wood, and (e) bamboo. Corresponding hot-pressed biopolymers; (f) CCPB, (g) cotton, (h) bagasse, (i) waste wood, and (j) bamboo.
Fig. 2. SEM micrographs of lignocellulosic microparticles showing particle distributions and orientation of each powder. (a) cotton, (b) bagasse, (c) waste wood, (d) bamboo microparticles captured at 190, 600 and 1200x magnification.

Table 3
Thermal properties of hot-pressed plant-based biopolymers.

| Lignocellulosic Biopolymer | Onset Temperature at 1% (°C) | Degradation Maxima Peaks (°C) | Endset weight Temperature (°C) | Remaining Residues (%) |
|----------------------------|-----------------------------|-------------------------------|--------------------------------|------------------------|
|                            |                             | I | ii | ii |                             |                          |                     |
| CCPB                       | 206                         | 266 | 300 | 421 | 491                          | 1.6                    |
| Cotton                     | 40∗                         | –  | 330 | 494 | 506                          | 6.22                   |
| Bagasse                    | 202                         | 261 | 323 | 484 | 572                          | 12.59                  |
| Waste wood                 | 182                         | –  | 312 | 385 | 441                          | 2.82                   |
| Bamboo                     | 180                         | –  | 295 | 361 | 662                          | 1.17                   |

∗ Hot-pressed milled cotton is sensitive to moisture and absorb moisture immediately after drying.
Fig. 3. SEM micrographs showing biopolymer morphologies of semi-lignin based adhesion; (a) hot-pressed coconut fibers (CCPB), lignin-based adhesion; (b) bagasse (BG), and non-lignin-based adhesion: (c) bamboo, (d) cotton, and (e) waste wood captured at 190, 600, and 1200x magnification.
Fig. 4. Mass-loss percentage curves for (a) lignocellulosic microparticles, and (b) the obtained hot-pressed biopolymers. Corresponding DTG of (c) lignocellulosic microparticles, and (d) the obtained hot-pressed biopolymers.
**Table 4**
Specific absorption-band wavenumbers (cm⁻¹) related to hot-pressed plant-based biopolymers according to FT-IR band assignments (cm⁻¹) numbered in the research article [1].

| Peak # | CCPB | Cotton | Bagasse | Waste wood | Bamboo |
|--------|------|--------|---------|------------|--------|
| 1      | 770  | 768    | 778     | 768        | –      |
| 2      | 806,817 | –     | –       | 811        | 812    |
| 3      | –    | –      | –       | 835        | –      |
| 4      | 852  | –      | –       | 857,868    | –      |
| 5      | 895  | 895    | 896     | 899        | 897    |
| 6      | 1030 | 1021   | 1025    | 1028       | 1029   |
| 7      | 1052 | 1053   | 1052    | 1054       | 1053   |
| 8      | 1112,1122 | 1113 | 1118 | 1109 | 1112,1122 |
| 9      | 1163 | 1159   | 1161    | 1161       | 1161   |
| 10     | 1233 | 1232   | 1236    | 1228       | 1236   |
| 11     | –    | –      | –       | –          | 1251   |
| 12     | 1267 | 1263   | 1261    | 1265       | 1258   |
| 13     | 1315 | 1315   | 1318    | 1315       | 1317   |
| 14     | 1337 | 1335   | 1331    | 1336       | 1331   |
| 15     | 1372 | 1370   | 1372    | 1370       | 1372   |
| 16     | 1421 | 1427   | 1419    | 1422       | 1421   |
| 17     | 1459 | 1456,1464 | 1457,1464 | 1456,1464 | 1463   |
| 18     | 1507 | –      | 1508    | 1509       | 1509   |
| 19     | 1541 | 1541   | 1542    | –          | 1542   |
| 20     | 1557 | 1559   | 1559    | 1560       | 1560   |
| 21     | 1591 | –      | 1593    | 1591       | 1592   |
| 22     | 1626 | 1628,1634 | 1635   | 1634       | 1631   |
| 23     | 1654 | 1653   | 1652    | 1652       | 1656   |
| 24     | 1736 | 1732   | 1716,1733 | 1731   | 1734   |
| 25     | 2851 | 2895   | 2894    | 2886       | 2854   |
| 26     | 2894,2923 | 2916 | 2902,2920 | –        | 2884,2920 |
| 27     | 3317,3342,3360 | 3310,3333 | 3309,3334,3356 | 3313,3337,3342 | 3312,3331,3359 |

**Table 5**
Summation of major peak areas in the “fingerprint” region associated with cellulose, lignin, hemicellulose and pectin in lignocellulosic biopolymers determine the difference in chemical composition among the obtained plant-based biopolymers.

| Sample          | ΣA (cellulose) | ΣA (lignin) | ΣA (hemicellulose) | ΣA (pectin) | C/L | L/(H+P) |
|-----------------|----------------|-------------|--------------------|-------------|-----|---------|
| CCPB            | 3.71           | 1.84        | 1.89               | –           | 2   | 1       |
| Cotton          | 5.25           | –           | 0.3                | –           | –   | –       |
| Bagasse         | 3.81           | 1.47        | 0.81               | 0.07        | 3.1 | 1.4     |
| Waste wood      | 3.17           | 1.65        | 0.40               | –           | 1.9 | 4.2     |
| Bamboo          | 4.17           | 1.63        | 1.18               | 0.08        | 2.6 | 1.3     |

|              | ΣA (cellulose) = (A_2895 + A_1317 + A_1370 + A_1320). | ΣA (lignin) = (A_1607 + A_1592). | ΣA (hemicellulose) = (A_1720 + A_1725 + A_1745). | ΣA (pectin) = (A_1324 + A_1346). | C/L | L/(H+P) |
|---------------|------------------------------------------------------|----------------------------------|-----------------------------------------------|----------------------------------|-----|---------|
| a              |                                                      |                                  |                                               |                                  |     |         |
| b              |                                                      |                                  |                                               |                                  |     |         |
| c              |                                                      |                                  |                                               |                                  |     |         |
| d              |                                                      |                                  |                                               |                                  |     |         |
| e              | The ratio of peak areas of cellulose to lignin.      |                                  |                                               |                                  |     |         |
| f              | The ratio of peak areas of lignin to hemicellulose and pectin. |                                  |                                               |                                  |     |         |

**Table 6**
Assignment of X-ray diffraction peaks of hot-pressed plant-based biopolymers.

| Sample | 2θ (Crystalline (002) (°)) | Intensity | 2θ (amorphous) (°) | Intensity | Crystallinity index (%) |
|--------|---------------------------|-----------|--------------------|-----------|------------------------|
| CCPB   | 22.56                     | 1247      | 19.07              | 578       | 54                     |
| Cotton | 22.42                     | 3412      | 17.98              | 1063      | 69                     |
| Bagasse| 21.43                     | 1033      | 18.02              | 660       | 36                     |
| Waste Wood | 22.55                  | 1922      | 18.87              | 809       | 58                     |
| Bamboo | 22.51                     | 1300      | 19.08              | 572       | 56                     |
Fig. 5. FT-IR spectra of hot-pressed plant-based biopolymers.

Fig. 6. XRD diffraction patterns for the obtained hot-pressed biopolymers showing sharp peaks in bagasse- and waste wood-based biopolymers associated with high content of inorganic materials typically found in ash waste [9].

2. Experimental design, materials and methods

2.1. Materials

Cotton and coconut coir fiber are purchased from a commercial supplier, recycled waste wood milled powder donated by (Air Water ECOROCA Inc., Japan), bagasse waste fiber (Tsukishima Kikai Co., Ltd, Japan), and bamboo flour with average particle size $\approx 75 \mu m$ purchased from (Nakawood, Naga Wood Co., Ltd, Japan). Bagasse waste fibers are thoroughly washed with
warm water for 10 mins, then dried for 5 days, compared to other plants fibers, which are used as received.

2.2. Powder preparation

Cotton and Bagasse fibers were pulverized into powders, using a planetary alumina ball mill (Pulverisette 6, Fritsch, Germany) operating at 300 RPM for 45 repetitions of a 3-min milling cycle with a 2-min pause between cycles. Extracted milled cotton and bagasse, and waste wood microparticles were then sieved in a sieving shaker (Retsch AS 200, Japan) to microparticle sizes shown in (Table 1).

2.3. Biopolymer preparation

First, 0.9 g of each of the materials was placed in stainless-steel Ø25 mm cylindrical molds, which were placed in a hot-press (AS-1 AH-2003, Japan) wherein the top and bottom plates were set at 170°C and wrapped by a heating belt at 170–180°C. Processing pressure was maintained at 20 MPa until the optimum temperature was achieved for cotton and coconut coir fiber (~180°C), and bagasse, waste wood, and bamboo (~140°C). The total hot-pressing time was 3–9 min. Subsequently, the disc-shaped specimens were gradually cooled at room temperature and dried in a vacuum oven at 100°C for two days before testing.

2.4. Determination of apparent density

Density was characterized by an analytical balance (AUX120, SHIMADZU, Co., Japan) using Archimedes’ Principle.

2.5. Bending strength

Three-point bending strength, strain at break, and Young’s modulus were evaluated using a tensile tester with 1 kN maximum loading capacity (Autograph AGS-X, Japan) according to the Japanese standard for testing flexural properties, JIS-K-7171 [7]. Small plate-shaped samples were cut from each biopolymer into 3-mm-wide × 20-mm-long × 2-mm-thick test pieces.

2.6. Determination of water resistance

Water absorption (WA) and thickness swelling (TS) of the biopolymers were calculated according to Japanese industrial standard water absorption and thickness swelling JIS-A-5905, Sections 7.10 and 7.11, respectively [2]. Lignocellulosic biopolymer were completely dried to a constant weight in a vacuum oven at 105°C. The samples were then cooled to a constant weight, and sample weight and thickness were measured before the samples were immersed for 24 h in a flask containing 50 mL of distilled water. The immersed samples were wiped with a dry cloth, and water absorption and thickness swelling were weighed and measured using Eqs. (1) and (2), respectively, as follows:

\[
\text{Water absorption(\%)} = \frac{(M_2 - M_1)}{M_1} \times 10c
\]

\[
\text{Thickness swelling(\%)} = \frac{(T_2 - T_1)}{T_1} \times 10c
\]
where \( M_2 \) and \( T_2 \) are weight and thickness of the sample immersed for 24 h, and \( M_1 \) and \( T_1 \) are the initial sample mass and thickness, respectively.

### 2.7 Statistical analysis

Results were statistically analyzed using analysis of variance (ANOVA) carried out by data analysis software (OriginPro, Version 2019b, OriginLab Co. USA) to calculate mean results of 3 samples for the above tests. Mean comparison at 0.05 level of significance (\( \rho<0.05 \)) was performed according to Tukey's test.

### 2.8 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) micrographs captured using (JSM-6510, JEOL, Ltd., Japan) to investigate milled powder and biopolymer morphologies. First, the samples were fixed onto an aluminum tray with conductive tape and coated with a thin layer of platinum using a magnetron sputter (MSP-1S, SHINKKU VD, Tokyo, Japan). Then, the samples were placed in SEM chamber and recorded at 190, 600, and 1200 \( \times \) magnifications.

### 2.9 Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) were performed using a thermogravimetric analyzer (EXSTAR TG/DTA 6300, Seiko Instruments, Inc., Japan) to analyze the thermal behavior of the obtained hot-pressed plant-based biopolymers and their corresponding microparticles of samples weigh (13–25 mg) and (5–12 mg), respectively. The samples were heated at 10 °C/min to 700 °C under an oxidative atmosphere (air) to ensure complete decomposition. Mass loss percentage (%) of the hot-pressed plant-based biopolymers was determined from TGA curves, where onset temperature (\( T_{onset} \)) was defined as 1% mass loss from (TGA) curves, and endset weight (%) was determined at 700 °C, while three major degradation peaks were identified and selected from DTG curves, when possible. In addition, moisture and ash contents of microparticles were determined by weight loss (%) measured at 130 and 700 °C, respectively, according to method in the related research article [1].

### 2.10 Attenuated total reflectance fourier-transform infrared spectroscopy (ATR-FTIR)

FTIR spectra (JASCO FT/IR-6600, JASCO Co., Japan) for the biopolymers were generated using the attenuated total reflectance (ATR) method for wavenumbers in the range 700–4000 cm\(^{-1}\) at 4 cm\(^{-1}\) resolution and 45 scans per sample to identify functional groups and note cellulose, lignin, hemicellulose and pectin contents in the obtained biopolymers. All spectra were normalized similarly to method described in [8], then peaks attributed to cellulose, hemicellulose, lignin, and pectin were identified by the second derivative, using graphing and data analysis software (OriginPro, Version 2019b, OriginLab Co. USA) according to a specified range and quantitative method [4-5]. All absorption peaks attributed to cellulose, hemicellulose, lignin and pectin in the “fingerprint” region were calculated by the second derivative integration of the baseline curve for specified regions of interest using adjacent-averaging as a smoothing method. Whereas other peaks were identified by their maximum height. The summation of major peaks areas associated with cellulose (\( A_{895} \), \( A_{1160} \), \( A_{1317} \), \( A_{1370} \) and \( A_{1420} \)), lignin (\( A_{1505} \) and \( A_{1606} \)), hemicellulose (\( A_{770} \), \( A_{735} \) and \( A_{1745} \)), pectin (\( A_{34} \) and \( A_{1246} \)) and corresponding peak area ratios were used to note the differences in chemical composition of the obtained biopolymers.
2.11. X-ray diffraction (XRD)

X-ray diffraction (XRD) (Ultima IV Protectus, Rigaku Co., Japan) was conducted to determine crystallinity indices \( X_c \) of hot-pressed plant-based biopolymers. CuK\( \alpha \) radiation was used, and XRD was operated at 40 kV and 30 mA. Samples were scanned at 0.2°/min in the 2\( \theta \) range 5–50°. Crystallinity \( X_c \) was analyzed according to an empirical method of calculating crystallinity index \( X_c \) of lignocellulose fibers, as previously reported \cite{6}, represented by Eq. (3), as follows.

\[
X_c(\%) = \frac{I(002) - I(AM)}{I(002)}
\]

where \( I(002) \) is the maximum intensity of the crystalline region, and \( I(AM) \) is the minimum intensity of the amorphous region between crystalline peaks at 16 and 22°.

Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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