Indonesian Journal of Forestry Research Vol. 11 No. 2, October 2024, 299-306 ISSN 2355-7079/E-ISSN 2406-8195

IDENTIFICATION OF LIGNOCELLULOSE-LIKE MATERIAL USING SPECTROSCOPY ANALYSIS

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Received: 01 November 2023, Revised: 04 June 2024, Accepted: 19 June 2024

IDENTIFICATION OF LIGNOCELLULOSE-LIKE MATERIAL USING SPECTROSCOPY ANALYSIS. Lignocellulose materials, such as bamboo, rattan, and wood, have been largely used for furniture and crafts. On the other hand, the utilization of lignocellulose-like materials, which have a similar texture and appearance to those from nature, has been increasing recently due to their superior durability. This research aimed to identify the lignocellulose-like material using spectroscopy analysis, such as Raman and Near Infrared (NIR), which is well-known as a non-destructive, quick, and accurate approach for material identification. We investigated four types of lignocellulose-like materials that were provided by Dewan Serat Indonesia (The Indonesian Fiber Council) from an industry that produces them. The NIR analysis was performed at wavenumbers 10,000-4,000 cm^{-1}. The natural lignocellulose (bamboo and wood) and the polymers (polyethylene and polypropylene) were used as standards. Raman analysis was further employed to identify the composition of selected lignocellulose-like materials by comparing their spectra with the library software. The results showed that the original NIR spectra of lignocellulose-like and those natural materials were different, indicating that the NIR analysis can differentiate those materials. The NIR spectra of lignocellulose-like materials were similar to those of polyethylene spectra. Those lignocellulose-like were also identified as polyethylene due to the similarity of the Raman spectra and their library spectra.

Keywords: Near Infrared spectroscopy, Raman spectroscopy, lignocellulose, lignocellulose-like materials, polyethylene

IDENTIFIKASI BAHAN SERUPA LIGNOSELULOSA MENGGUNAKAN ANALISIS SPEKTROSKOPI. Material lignoselulosa seperti bambu, rotan, dan kayu telah banyak digunakan untuk mebel dan kerajinan. Disisi lain, pemanfaatan material tiruan, dimana bahan tersebut mempunyai kesamaan tekstur dan penampilannya dengan lignoselulosa dari alam semakin meningkat. Hal ini juga didorong oleh sifat keawetannya yang sangat tinggi sehingga menjadi salah satu bahan pengganti yang potensial. Tujuan dari penelitian ini adalah untuk mengidentifikasi material tiruan lignoselulosa dengan analisa spektroskopi Near Infrared dan Raman, dimana kedua alat ini mempunyai keunggulan karena cepat, tidak merusak, dan memiliki keakuratan tinggi untuk melakukan identifikasi. Penelitian ini menggunakan empat jenis material tiruan lignoselusa yang diperoleh dari Dewan Serat Indonesia. Bahan ini merupakan salah satu produk yang dihasilkan oleh Perusahaan luar negeri. Analisa Near Infrared dilakukan dengan perekaman spektra pada nomor gelombang 10.000-4.000 cm^{-1}. Sebagai pembanding, digunakan material lignoselulosa alami dan juga polimer seperti bambu, kayu, poli etilen, dan poli propilen. Sedangkan analisa dengan Raman dilakukan dengan membandingkan spektra Raman dari material tersebut dengan spektra yang ada pada software Raman spektroskopi. Hasil penelitian menunjukkan bahwa spektra original Near Infrared dari bahan tersebut sangat berbeda dengan bahan lignoselulosa alami, yang mengindikasikan bahwa analisa tersebut dapat digunakan untuk membedakan bahan lignoselulosa alami dan tiruan. Selain itu, spektra

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Near Infrared dari bahan tiruan mempunyai kemiripan dengan polimer poli etilen. Hal ini juga dikuatkan dengan hasil dari analisa Raman, dimana spektranya sama dengan spektra poli etilen pada data base software Raman.

Kata kunci: Near Infrared, Raman spektroskopi, . lignoselulosa, lignoselulosa tiruan, poli etilen

I. INTRODUCTION

Indonesia is well known for its mega biodiversity in its natural resources, including natural fibers or lignocellulosic materials, such as wood, bamboo, and rattan. For instance, almost 11.5% of all bamboo species in the world are grown in Indonesia, which consists of 161 species from 12 families (Widjaja et al., 2014). In addition, Indonesia has 4,000 wood species, and hundreds of those species have been traded with high commercial values (Damayanti et al., 2019). The abundance of natural resources plays an important role in helping developing countries maximize the potential utilization of those resources for their economic growth. Furthermore, natural resources are known as bio-renewable and eco-friendly materials (Karimah et al., 2021), thus their utilization can reduce the risk of environmental issues.

Lignocellulose materials have a wide range of applications, including structural uses in housing and building materials, furniture, and crafts. Moreover, the utilization of bamboo and wood as raw materials for crafts has been increasing and becoming one of the fastest-growing micro, small, and medium enterprises of the creative economic sector in Indonesia, which contributes to 60% of Gross Domestic Products (GDP) (Christine, Dachyar, & Nurcahyo, 2019; Herliana, Crestofel Lantu, Rosmiati, Chaerudin, & Lawiyah, 2022). As an organic material, lignocellulose has issues related to its durability, stability, and performance against weather and organisms (Himmi et al., 2017; Ismayati et al., 2011). On the other hand, the development of lignocellulose-like materials is also rising, which have similar texture, surface, and appearance to natural lignocellulose. One of the producers of lignocellulose-like materials claimed that their products are very resistant and durable against weather, moisture, mold, and termites, thus indicating the product is superior to the natural one. However, the increasing awareness of people toward their environment has led to the utilization of natural resources as the main raw materials for replacing hazardous and synthetic materials (Karimah et al., 2021). Thus, material identification to differentiate natural and lignocellulose-like materials is needed.

Direct investigation of the sample is usually destructive and requires a large sample, labor intensives, and time-consuming. Therefore, rapid analysis, non-destructive, and accurate method is urgently required to identify the natural and lignocellulose-like materials. Spectroscopy analysis using Near Infrared (NIR) and Raman spectroscopy might be able to meet all those requirements (Ayanleye & Avramidis, 2021; Deneva et al., 2020).

NIR is a region of the electromagnetic spectrum at the wave number 12,820-4,000 cm^{-1} (wavelength 780-2,500 nm) (Schwanninger, Rodrigues, & Fackler, 2011). The combination of chemometrics analysis and NIR spectroscopy is very useful as qualitative and quantitative methods (Tsuchikawa & Kobori, 2015). As a qualitative analysis, the principle of using NIR is comparing spectra of unknown and known materials (Wang, Xiang, Tang, Chen, & Xu, 2022), which could be used to identify some wood species (Adi et al., 2020; Hwang, Horikawa, Lee, & Sugiyama, 2016). The NIR range is suitable for measuring organic materials containing high moisture due to the smaller molar absorptivity compared to the infrared (IR) range (Tsuchikawa & Kobori, 2015). In addition, NIR spectroscopy is less strong than IR spectroscopy, which enables the thick polymer samples to be analyzed without suffering from the light. By using NIR spectroscopy, the absorption is very weak, making it possible to measure the NIR transmittance or reflectance without any pretreatment (Shinzawa et al., 2021). Moreover, analyzing the sort of plastic could be processed by NIR with high speed measurements, radiation penetration depth, and signal-to-noise ratio (Xia, Huang, Li, Xiong, & Min, 2021). On the other hand, Raman spectroscopy is popular as a non-destructive test for woody materials (Yamauchi, Iijima, & Doi, 2005). Raman spectroscopy is useful for detecting the vibrations of symmetrical structures and is very sensitive to polarizability (Gierlinger, 2018). In addition, it has important information regarding the structural and chemical composition (Amjad, Ullah, Khan, Bilal, & Khan, 2018). Raman spectroscopy is not only used to identify organic materials, but it is also used to identify polymers (Kida, Sasaki, Hiejima, Maeda, & Nitta, 2020; Zhou, Liu, Hao, & Liu, 2021). Therefore, this study was conducted to identify natural and lignocellulose-like materials by comparing their NIR and Raman spectra.

II. MATERIAL AND METHOD

A. Materials

This study investigated four types of lignocellulose-like materials, which have similar appearance and texture to wood and bamboo. These materials were obtained from Dewan Serat Indonesia (The Indonesian Fiber Council) in the form of raw materials in the square form, not as finished products (Figure 1. A-D). The samples were produced by a foreign company that it seems made from some plastic polymers. Several types of wood and bamboo species were selected at random and subsequently analyzed as the standard for natural lignocellulose. Eight commercial wood species from Indonesia were used for comparison, namely: Balau, heavy Red Meranti, White Meranti (obtained from commercial market around Bogor-Indonesia); Old Teak, Platinum Teak, Jati Plus (fast-growing Teak) (obtained from the National Research and Innovation Agency of Indonesia-BRIN); *Shorea bracteolata*, and *Shorea parvifolia* (obtained from *Xylarium Bogoriense*). Furthermore, four bamboo species from the bamboo collection field at the BRIN in Cibinong, such as *Dendrocalamus asper*, *Bambusa vulgaris*, *Giganthocloa atter*, and *Bambusa maculata* Widjaja, were also investigated. In addition, low-density polyethylene (PE) and polypropylene (PP) were also analyzed as standards for plastics.

B. Near Infrared and Raman Spectroscopy Methods

The samples were analyzed directly in the NIR machine (Frontier, PerkinElmer, Massachusetts, USA) to get the spectra at wavenumber 10,000-4,000 cm^{-1}, with 32 scans and a resolution of 16 cm^{-1}. The NIR spectra were set to provide a graph with absorbance and wavenumber. The determination of

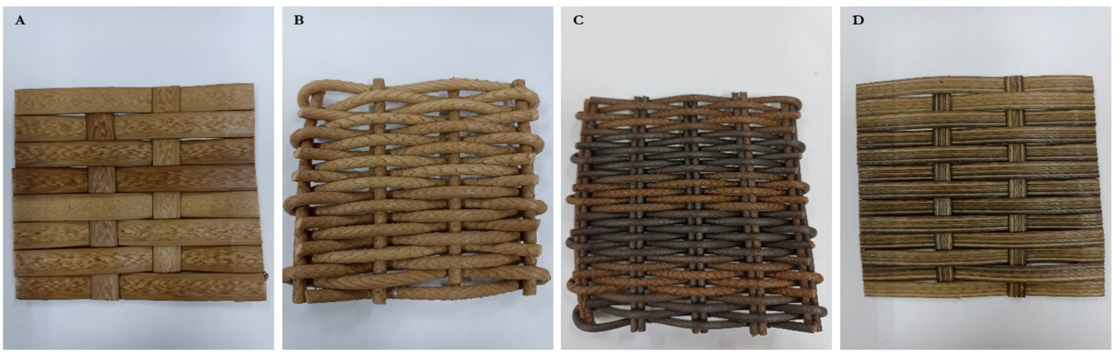

Figure 1. The samples of lignocellulose-like materials: A&D the shape is rectangular with different colours; B&C the shape is rounded with different colours

natural and lignocellulose-like materials was carried out by comparing the original spectra of those materials and the spectra of PE and PP. However, additional classification procedures would be applied if the original spectra exhibited comparable shapes in both natural and lignocellulose-like materials. Raman Spectroscopy analysis (LabRam HR Evolution Confocal Raman Microscope, Horiba France SAS, Longjumeau, France) was conducted at the Integrated Laboratory of Bioproducts (iLab)-BRIN, with the laser 532 nm and grating 600. The other parameters on the Raman analysis were ND filter 50%, objective 100x visible, acquisition time 12 seconds, and accumulation 10. Then, an integrated identifier software in Raman software was used to identify the composition of all samples by comparing their spectra to the database (Bio-Rad Laboratories).

III. RESULT AND DISCUSSION

A. Near Infrared Spectroscopy

A graphical representation of the NIR original spectra of natural lignocellulose samples of 8 wood species (A) and 4 bamboo species (B) is visualized in Figure 2. The graphs indicate that there were identical shapes and bands for all-natural lignocellulose. The discrepancies of the original spectra are only for the absorbance value, where the wood species has a big variation compared to those bamboo species. Adi et al. (2020) revealed the relationship between density and their NIR absorbance values shows that the high density tends to have a positive correlation with absorbance, for instance, Balau (0.76 g/cm^3) has a higher absorbance value than red Meranti (0.64g/cm^3). This result is also similar to that of Belangke bamboo, in which the upper

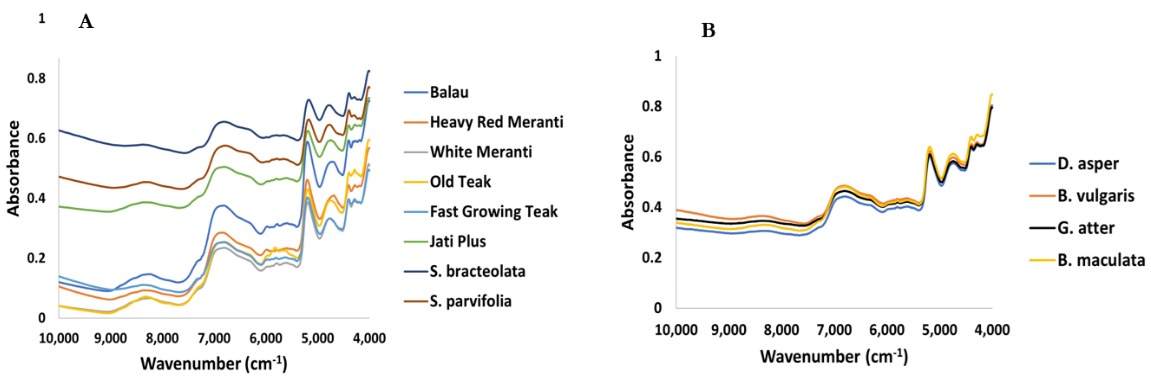

Figure 2. The NIR original spectra at wavenumber 10,000-4,000 cm^{-1} of wood species (A) and bamboo species (B)

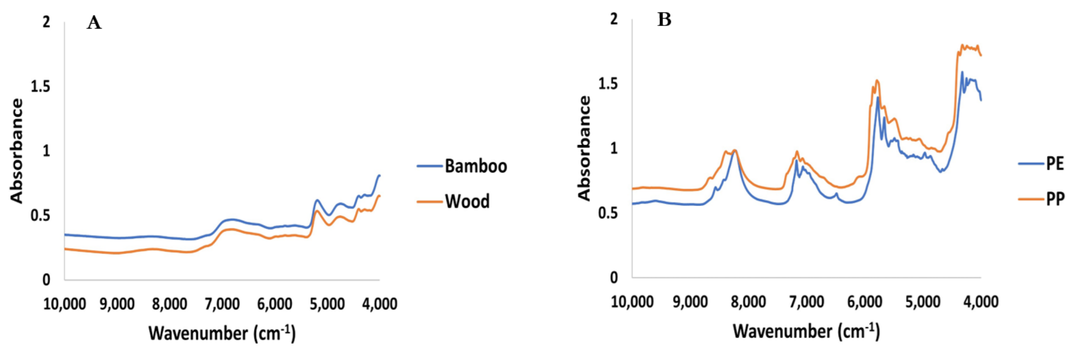

Figure 3 The average of NIR original spectra at wavenumber 10,000-4,000 cm^{-1} of wood and bamboo species (A); polymer standard for PP and PE (B)

part has higher absorbance than the other part since the top part has the highest density value (Iswanto et al., 2022). However, this study used several wood and bamboo species as a standard only.

The mean spectra for wood and bamboo species revealed that their typical spectra were in a similar shape (Figure 3A), although there were different values of absorbance, baseline shift, and some flat curves. This phenomenon usually occurs in the original spectra, which makes the interpretation of the bands very difficult. The chemical and structural characteristics of the cell walls have an impact on NIR spectroscopy because it is sensitive to functional groups (Horikawa, Mizuno-Tazuru, & Sugiyama, 2015). The spectra of plastic materials, such as PE and PP, are completely different from those of organic materials (Figure 3B). The basic concept of NIRS qualitative analysis is comparing the spectra of unidentified materials with those of known samples spectra (Wang et al., 2022). Consequently, it had been acceptable to easily distinguish between lignocellulose and plastic materials by just comparing the original spectra directly. The spectra of polymers contain broad overtones of stretching and bending vibrational bands due to the light reflectance of the polymers and the NIR light source (Michel et al., 2020). PE and PP have different bands in the region of 6,500 cm^{-1} and 5,500-5,000 cm^{-1}, which might be useful information for differentiating both materials. Some treatments, like the first derivative and

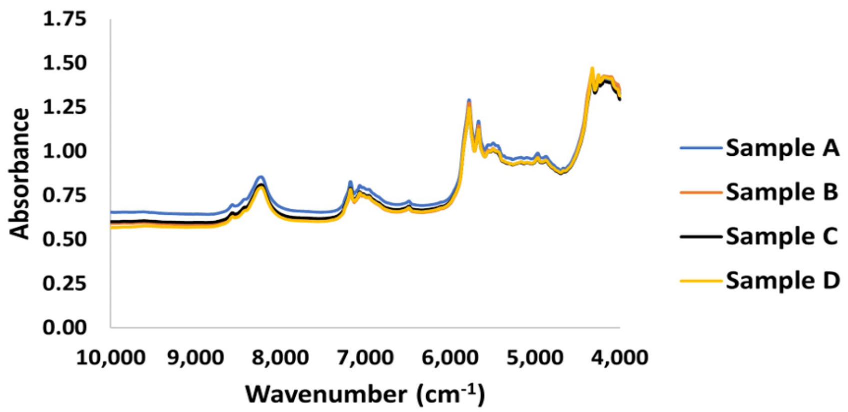

Figure 4. The average of NIR original spectra at wavenumber 10,000-4,000 cm^{-1} of 4 types of lignocellulose-like materials

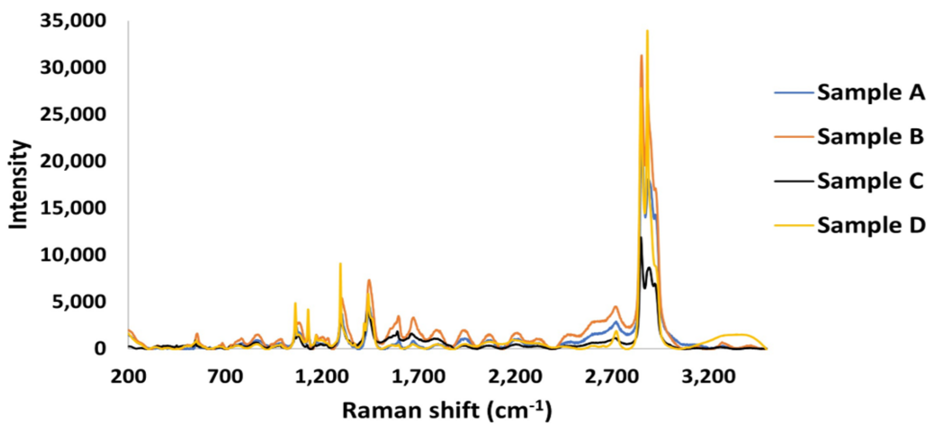

Figure 5. The Raman spectra of 4 types of lignocellulose-like materials

Savitzky-Golay filter, could make the bands more clearly visible (Duan & Li, 2021).

Figure 4 shows the mean of original NIR spectra of 4 types of lignocellulose-like material at 10,000-4,000 cm^{-1}. At first glance, the absorbance values of all samples were identical, and it seems the graphs overlap. It was presumed that the raw materials used in all samples were comparable. The classification of unknown materials in optical spectroscopy usually uses visual inspection of the spectrum (Tuncer, Dogu, & Akdeniz, 2021). By comparing the spectra of lignocellulose-like material to the standard in Figure 3A, it was clearly seen that the raw material of lignocellulose-like was not made from the natural material. According to Figure 3B, the spectra of the 4 samples were compared to the PE spectra due to the appearance of some bands in wavenumber 6,490 cm^{-1} and the region around 5,500-5,000 cm^{-1}; thus, it was supposed that all the samples were made of PE plastic.

B. Raman Spectroscopy

Raman spectra of all lignocellulose samples are presented in Figure 5. In general, the spectra showed similar shapes, although there were some noise peaks on specific Raman shifts. Getting smooth spectra in Raman spectroscopy is challenging. The fluorescence interference has influenced the variation of baseline modeling; thus, choosing the appropriate window for polynomial fitting was required to minimize the noise (Amigo, Babamoradi, & Elcoroaristizabal, 2015). Furthermore, setting the proper accumulation and acquisition time is also needed in the analysis of Raman spectroscopy. This study found that there were at least 5 peaks in the Raman spectra, with different settings of acquisition times and accumulation. These peaks were at Raman shift 1,100 cm^{-1}, 1,250 cm^{-1}, 1,450 cm^{-1}, 2,700 cm^{-1}, and 2,850 cm^{-1}.

Micro Confocal Hyperspectral 3D Raman Spectroscopy in this study was completed with software to determine the spectra based on comparison with the spectra database from Bio-Rad Laboratories. The results revealed that 4 types of synthetic lignocellulose materials had different compositions. The sample 1 was identified as High-Density Polyethylene (HDPE), while sample 2, sample 3, and sample 4 were Low-Density Polyethylene (LDPE), LDPE + acrylic acid, and LDPE, respectively. PE was a base structure for that synthetic lignocellulose, even though 2 samples have different PE compositions. These findings were in accordance with those from the NIR spectra. Kniggendorf, Wetzel, & Roth (2019) reported that PE has Raman bands at 2,850 cm^{-1} and 2,884 cm^{-1}. All samples in this study also had bands at 2,850 cm^{-1}, which was the highest peak and contained -CH2 and -CH3 stretching vibrations (Gopanna, Mandapati, Thomas, Rajan, & Chavali, 2019).

Several factors might have contributed to the variances in PE structure in these samples, such as treatment processes to the Raman spectra, the surface of the sample, and some additives or colors to the samples. Selecting the proper window for polynomial fitting plays a crucial role. For instance, using a small window has the effect of generating noise, which means new bands appear in the spectrum. In contrast, choosing a wide window could eliminate some bands (Amigo et al., 2015). The surface characteristics have an effect on adding noise to the signal (Duan & Li, 2021). Adding some color and additives could change the spectrum (Michel et al., 2020). LDPE and HDPE have similar molecular structures (Duan & Li, 2021), so differentiating those materials by molecular vibrations is challenging.

IV. CONCLUSION

Determination between natural and lignocellulose-like materials was successfully introduced by using NIR and Raman spectroscopy. These techniques were non-destructive, fast, reliable, and traceable. The original spectra of natural and lignocellulose-like were very different, which indicated that the lignocellulose-like was made from synthetic material. By comparing the standard spectra of plastics, the composition of these materials was identified as LDPE. The Raman spectra corresponded to the NIR analysis since the spectra were similar to the LDPE spectra reference, even though Sample 1 was identical with HDPE and Sample 3 consisted of LDPE and acrylic acid.

ACKNOWLEDGEMENT

The authors thank Dewan Serat Indonesia for providing lignocellulose-like material samples, and Xylarium Bogoriense for supplying samples of various wood species.. This study is partially supported by the National Research and Innovation Agency (BRIN). The publication is partially supported by JSPS Alumni Association of Indonesia (JAAI).

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