Original Article

Characterization of new cellulosic fiber: Dracaena reflexa as a reinforcement for polymer composite structures

P. Manimaran\textsuperscript{a}, S.P. Saravanan\textsuperscript{b}, M.R. Sanjay\textsuperscript{c,\,*}, Suchart Siengchin\textsuperscript{c}, Mohammad Jawaid\textsuperscript{d,\,*}, Anish Khan\textsuperscript{e}

\textsuperscript{a} Department of Mechanical Engineering, Karpagam Institute of Technology, Coimbatore, Tamilnadu, India
\textsuperscript{b} Department of Mechanical Engineering, KIT and KIM Technical Campus, Karaikudi, Tamil Nadu, India
\textsuperscript{c} Department of Mechanical and Process Engineering, The Sirindhorn International Thai - German Graduate School of Engineering (TGGS), King Mongkut's University of Technology North Bangkok (KMUTNB), Bangsue, Bangkok, Thailand
\textsuperscript{d} Department of Biocomposite Technology, Institute of Tropical Forestry and Forest Products, Universiti Putra Malaysia, UPM Serdang, Selangor, Malaysia
\textsuperscript{e} Centre of Excellence for Advanced Material Research, Chemistry Department, Faculty of Science, King Abdulaziz University Jeddah, Saudi Arabia

\textbf{Abstract}

The search for novel bio-fibers in the field of the green composite can rise the invention of natural fiber composite and applications. In this work, the physical, chemical, structural, thermal, tensile and surface morphology properties of Dracaena reflexa fiber (DRF) are investigated. The chemical analysis results authorized the higher cellulose (70.32\%) and lesser hemicelluloses (11.02\%) and lignin (11.35\%) existing in DRF. XRD analysis proved that DRF has a relatively higher crystallinity index of 57.32\%. The free chemical functional groups presented in DRFs were determined by FT-IR. The DRF is thermally stable up to 230 \degree C which is greater than the processing temperature of thermoplastics resin. The C2, C3, and C5 peaks intensity of CP/MAS C\textsuperscript{13} NMR spectra once again confirmed that maximum cellulose present in DRFs. The lower density (790 kg/m\textsuperscript{3}) and higher tensile properties of DRF show the DRF is a suitable alternative to the synthetic fibers.

© 2019 Brazilian Metallurgical, Materials and Mining Association. Published by Elsevier Editora Ltda. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

1. Introduction

The current interest in materials and environmental related awareness drive the researchers to use of plant fibers as substitute materials for reinforcement in polymer composites [1–3]. Plant fibers reinforced composites can be used in various fields such as automotive, packaging, marine, constructions and military because of their attractive features such as less weight, biodegradable, eco-friendly, non-toxic, less cost and better mechanical properties [4–7]. Many researchers have documented that physico-chemical properties such as...
density, diameter, chemical composition, thermal stability and surface roughness of the plant fibers are depending on the source of the fiber such as stem, fruit, bark, root, stalk and leaf [8,9]. The most common chemical constituents of plant fibers are cellulose, hemicelluloses, lignin, pectin and wax. The expected properties of natural fibers are lengthy, lesser diameter and low spiral angle of the cellulose arrangement. The final properties of polymer composites are depending on the type of resin, form of reinforcement (nano or micro powder, short fiber, and continuous fiber), alignment of fiber and interfacial bonding between the fiber and matrix. Different kinds of plant fibers such as Arundo Donax L., Cissus quadrangularis, banana, hemp, sisal, Grewia tilifolia and others as potential reinforcement have been examined. Some researchers recently studied suitability of natural fibers, such as Furcraea Foetida, Coccinia grandis. L and Sida cordifolia, as reinforcement for polymer composites [10–20].

In this investigation, the bio-fibers extracted from Dracaena reflexa was considered as a potential reinforcement for polymer composite. These fibers have selected for numerous reasons:

1. It is commonly grown in all the climate conditions.
2. Its leaves are consumed as medicine for malarial fever, poisoning, dysentery, and diarrhea and dysmenorrhea. So, it is cultivated for commercial use.
3. Dracaena reflexa was used to clean and remove considerable amount of toxins from the air which was proved by the NASA (National Aeronautics and Space Administration).

However, there is no literature available on the fibers extracted from leaves of Dracaena reflexa. So, in this article, we analyzed physical properties, chemical composition, chemical functional groups, microstructure, thermal stability, single fiber tensile strength, crystalline properties and surface roughness of the Dracaena reflexa fiber by chemical analysis, single fiber tensile test, optical microscope, X-ray diffraction method (XRD), Fourier transform infrared (FT-IR) spectroscopy, thermogravimetric analysis (TGA), differential scanning calorimeter (DSC), scanning electron microscopy (SEM), atomic force microscopy (AFM) and nuclear magnetic resonance spectroscopy (NMR).

2.2. Extraction of leaf fiber from Dracaena reflexa plant

The Dracaena reflexa plants and its extracted fibers are presented in Fig. 1. The Dracaena reflexa leaves were gathered from the plant, then they were submerged into the water for retting (maximum period of two weeks). After two weeks, the fibers were mined using a comb with the metal teeth.

2.3. Physical analysis

It is very challenging to decide diameter of the plant fibers because the fiber is uneven in shape, so it is essential to calculate the average diameter of the fiber. With the aid of Carl Zeiss Optical microscope, the profile shapes and diameter of 25 samples of Dracaena reflexa fibers were identified. The diameter of each fiber was measured at five different places and the mean value was used for statistical analysis.

The pycnometer (toluene, \( \rho = 866 \text{ kg/m}^3 \)) experimentation is an suitable technique to observe the density of the bio-fiber. First DRFs were kept in a glass container filled with silica gel for 96 h to eliminate the moisture from fiber and then trim into 5 mm length whiskers for put in the pycnometer. In order to remove the bubbles in the fibers, DRFs were immersed into toluene for 2 h before the execution of test [21].

\[
\rho_{\text{DRF}} = \frac{(m_2 - m_1)}{(m_3 - m_1)(m_4 - m_2)} \rho_t
\]

where, \( \rho_{\text{DRF}} \) is the density of DRF in kg/m\(^3\), \( \rho_t \) is the density of toluene in kg/m\(^3\), \( m_1 \) is the mass of empty pycnometer in kg, \( m_2 \) is the mass of pycnometer with fibers in kg, \( m_3 \) is the mass of pycnometer with toluene in kg and \( m_4 \) is the mass of pycnometer with fibers and toluene in kg.

2.4. Single fiber tensile test and statistical analysis

The maximum tensile strength, Young’s modulus and elongation at break of single fibers of DFFs were estimated by single fiber tensile test with aid of an Instron 5500 R UTM (Universal Testing Machine). As per the guidelines of ASTM D 3822 standard, 70 mm gauge length (single fiber) and 5 mm/min crosshead speed was set for experimentation. The tests were executed at a room temperature of 25 °C with relative humidity of 65%. The microfibril angle (\( \omega \)) of DFF was determined by following the equation (2) [10].

\[
\varepsilon = \ln \left(1 + \frac{\Delta L}{L_0}\right) = -\ln(\cos \omega)
\]

where ‘\( \varepsilon \)’ represents the global deformation (or) strain, ‘\( \omega \)’ symbolizes the microfibril angle in degree, ‘\( L_0 \)’ indicates the gauge length (mm), and ‘\( \Delta L \)’ denotes the elongation at break (mm).

Commonly, tensile test results of plant fibers are scattered and depend on the age of the plant from which the fiber is extracted, extraction technique, testing environment, variation in the diameter and occurrences of defects in the surface of the fiber. It is essential to find the mean values of tensile properties through statistical analysis. Several researchers
used Weibull distribution for analyzing tensile properties of cellulosic fibers. In this way, the authors performed Weibull analyzing through Minitab 17 software [17].

2.5. Chemical analysis

In general, the chemical composition of the fibers was strongly affected by the region, extraction methods, soil conditions, age of the plant and approaches used, to predict the composition [15]. The aim of this examination was to quantify the amount of cellulose, hemicelluloses, lignin, wax, ash and moisture content in the DFFs. The cellulose content of DFF was assessed by using technique which explained in previous work [16]. The hemicellulose cellulose content of DFF was confirmed through neutral detergent fiber method. APPITA P11s-78 method is used to compute the lignin contents [16]. The wax percentages was proven by Conrad method [16]. For the ash content measurement the way stated by TAPPI (Technical Association of the Pulp and Paper Industry) was applied [16]. The moisture present in the DRF was assessed by electronic moisture analyzer (model MA45).

2.6. Fourier transform-infrared (FT-IR) spectrum analysis

Fourier transform infrared spectroscopy (FT-IR) is the right method to detect the chemical functional group existing in the natural fiber. Particular amount of DRFs was crushed into the fine powder then blended with potassium bromide (KBr) and make pellets by applying pressure. The FT-IR spectra of DRFs obtained by a Shimadzu spectrometer (FTIR-8400S, Japan) in the wavenumber range of 4000–500 cm\(^{-1}\).

2.7. X-ray diffraction (XRD) analysis

X-ray diffraction is an excellent tool to notice the difference between amorphous and crystalline material present in the natural fiber. The examinations was done by the X’PERT-PRO diffractometer with the intensity of Cu K\(_\alpha\) radiation wavelength of 0.154 nm. The crystallinity index (CI) of the DRF was
determined by using following equation suggested by Segal et al. [22].

\[
C_I = \left( 1 - \frac{I_{am}}{I_{002}} \right) \times 100\% 
\]

(3)

where \(I_{002}\) is the intensity of crystalline peak (23°) and \(I_{am}\) is the intensity of amorphous peak (18°).

The crystallite size (CS) of DRF was calculate by using the following formula [23]:

\[
CS_{002} = \frac{0.9\lambda}{\beta_{002} \cos \theta} 
\]

(4)

where \(\beta\) peak's full-width at half-maximum, \(\lambda\) is the wavelength of the radiation (0.1541 nm) and \(\theta\) is Bragg angle.

2.8. Thermogravimetric analysis (TGA)

It is prerequisite to verify the thermal stability of the natural fibers to confirm the fitness of fibers for high temperature composite processing and applications. Thermogravimetric analysis of DRF was completed in \(N_2\) (nitrogen) environment using TGA SDT Q600 machine (TA Instruments, India) with the aid of Thermal Advantage software. The weight loss of DRF was investigated at a rate of heating \(10^\circ C/\text{min}\) in the temperature range of 30–1000°C. The kinetic activation energy (\(E_a\)) of the DRF is evaluated by following Broido's equation [24]:

\[
\ln \left[ \ln \left( \frac{1}{T} \right) \right] = - \left( \frac{E_a}{R} \right) \left[ \frac{1}{T} \right] + K 
\]

(5)

where, \(R\) denotes the gas constant (8.32 J/mol/K), \(T\) specify the temperature in Kelvin, \(y\) represents the normalized weight \((w_i/w_0)\), \(w_i\) indicates the weight of the sample at any time \(t\), \(w_0\) designate the initial weight of the sample.

2.9. Differential scanning calorimeter analysis (DSC)

The differential scanning calorimetry (DSC) analysis of DRF was performed by DSC SDT Q600 (TA Instruments, India). The heating rate was retained at \(10^\circ C/\text{min}\) and temperature ranging from 30°C to 1000°C. \(\Delta H\) is the melting peak and \(T_g\) is the glass transition temperature of DRF was finalized.

2.10. Scanning electron microscopy (SEM)

TESCAN model VEGA3 scanning electron microscope with an electron beam accelerating potential of 3 kV was utilized to examine surface of DRF at different amplifications. The platinum layer was formed on the surface of DRF to avoid electron beam charging influences during the examination.

2.11. Energy dispersive X-ray spectroscopy analysis

Energy dispersive X-ray spectroscopy (EDX) is an excellent method to assess the quantity of elements (C, O, N, Cl, Si, etc.) distributed on the surface of DRFs in terms of weight (%) as well as atomic (%). The elements existing on the surface of the DRFs were calculated in five trials and mean value has been recorded with support of EDX (INCAPentaFETx3).

2.12. Atomic force microscopy

Atomic force microscopy (AFM) can provide qualitative as well as quantities information of surface roughness parameters of natural fiber such as \(R_a\) – average surface roughness, \(R_q\) or \(R_{rms}\) – root mean square roughness, \(R_z\) – ten point average roughness, \(R_{sk}\) – skewness, \(R_{ku}\) – Kurtosis and \(R_t\) – maximum peak-to-valley height. This analysis was executed in atmosphere environment by Park XE-100 Modal Atomic Force Microscopy. This AFM analysis has given high resolution 2D and 3D images of a surface of sample.

2.13. \(^{13}C\) (CP-MAS) NMR spectroscopy

Solid state NMR is a helpful tool to characterize the occurrence of anisotropic (directionally dependent) interfaces in natural fiber. Solid-state NMR spectrometer (DELTAs2 Modal) available at IISc, Bangalore, India was used for this investigation. This analysis was conducted in the cross-polarization mode with MAS rate of 10 kHz at environmental temperature of 25°C and the operating frequency of \(^{13}C\) nuclei was set as 75.46 MHz.

3. Results and discussion

3.1. Physical analysis

Measurement of the bio-fiber diameter is complicated one because the fibers outer profile (diameter) is varying in nature. However, it is supposed to be circular to compute the tensile strength and Young’s modulus. The diameters of the DRF is \(172.5 \pm 7.897 \mu m\). Density of the natural fiber plays the virtual role in the weight of the composite. So, it is need to discover the low density novel fiber, suitably the density of DRF is \(790 \pm 22.78 \text{kg/m}^3\) which is notably lower than widely used other natural fibers such as sisal \((1500 \text{kg/m}^3)\), banana \((1350 \text{kg/m}^3)\), Palmyrah \((1090 \text{kg/m}^3)\), Cyperus pangorei \((1102 \text{kg/m}^3)\), Sansevieria ehrenbergii \((887 \text{kg/m}^3)\) and Sansevieria cylindrica \((915 \text{kg/m}^3)\).

3.2. Single fiber tensile test and statistical analysis

The tensile properties of any natural fiber depending on chemical composition (presence of cellulose, hemicellulose and lignin), structure, growing conditions, harvesting time and extraction method. Low density fiber with improved tensile properties is preferred for lightweight composite structures. Twenty samples are tested for each gauge length ranging from 10 mm to 50 mm in steps of 10 mm using the universal test machine (INSTRON-5500R). The tensile properties of DRF are compared with other natural fibers and are presented in Table 1. From Table 1, it can be seen that the average tensile strength, modulus and elongation at break of Dracaena reflexa fibers was higher than the some other natural fibers. It is obvious from this table that the obtained tensile strength value is about 829.6 MPa. This value is higher than that found in some other natural fibers. This comparison table indicates the tensile properties of DRF are comparatively high enough for its application as reinforcement in polymer composite fabrication.
<table>
<thead>
<tr>
<th>Name of the natural fiber</th>
<th>Physical properties</th>
<th>Tensile properties</th>
<th>Chemical properties</th>
<th>Crystalline properties</th>
<th>Thermal Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Diameter (μm)</td>
<td>Density (kg/m³)</td>
<td>Young's modulus (MPa)</td>
<td>Elongation at break (%)</td>
<td>Microfibril angle (°)</td>
</tr>
<tr>
<td>Dracaena reflexa</td>
<td>176.20</td>
<td>790</td>
<td>829.6</td>
<td>46.37</td>
<td>2.95</td>
</tr>
<tr>
<td>Furcraea foetida</td>
<td>12.8</td>
<td>778</td>
<td>623.52 ± 45</td>
<td>6.52 ± 1.9</td>
<td>10.32 ± 1.6</td>
</tr>
<tr>
<td>Coccinia grandis</td>
<td>27.33</td>
<td>1243</td>
<td>273 ± 27.74</td>
<td>10.17 ± 1.261</td>
<td>2.703 ± 0.2736</td>
</tr>
<tr>
<td>Saharan Aloevera</td>
<td>80.61</td>
<td>1325.1</td>
<td>805.5</td>
<td>42.29</td>
<td>2.39</td>
</tr>
<tr>
<td>Sida Cordifolia</td>
<td>–</td>
<td>1330</td>
<td>703.95 ± 23.73</td>
<td>42.84 ± 2.1</td>
<td>2.89 ± 0.24</td>
</tr>
<tr>
<td>Arabicica Acacia</td>
<td>1102</td>
<td>1028</td>
<td>196 ± 56</td>
<td>11.6 ± 2.6</td>
<td>1.69</td>
</tr>
<tr>
<td>Cyperus</td>
<td>1102</td>
<td>1028</td>
<td>196 ± 56</td>
<td>11.6 ± 2.6</td>
<td>1.69</td>
</tr>
<tr>
<td>Salsirevia trifasciata</td>
<td>80-120</td>
<td>1414.7</td>
<td>348.6</td>
<td>15.3</td>
<td>2.3</td>
</tr>
<tr>
<td>Mendong Grass</td>
<td>33.4</td>
<td>892</td>
<td>452</td>
<td>17.4</td>
<td>22.9</td>
</tr>
<tr>
<td>Hibiscus sabdariffa.</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Borassus fruit</td>
<td>241.18</td>
<td>1256</td>
<td>175.52</td>
<td>31.34</td>
<td>–</td>
</tr>
<tr>
<td>Agave americana</td>
<td>1256</td>
<td>1200</td>
<td>175.52</td>
<td>31.34</td>
<td>–</td>
</tr>
<tr>
<td>Jute</td>
<td>26</td>
<td>1300</td>
<td>400-773</td>
<td>10-30</td>
<td>1.5-1.8</td>
</tr>
<tr>
<td>Rhetophyllum camerunense</td>
<td>1256</td>
<td>1200</td>
<td>175.52</td>
<td>31.34</td>
<td>–</td>
</tr>
</tbody>
</table>
The Weibull distribution curves of diameter, young’s modulus, tensile strength, elongation at break of DRFs were shown in Fig. 2. It can be deduced that the tensile test results of all the 20 trials were situated inside the line and fit perfectly the Weibull distribution. It can be clearly seen that the Weibull distribution with four parameters provide the mechanical properties close to the average values obtained experimentally.

3.3. Chemical analysis

The tensile strength, thermal stability, crystallinity index (CI), non-flammability and biological decomposability of the biofibers are influenced by the chemical composition of that fibers. DRFs is comprised of cellulose (70.32 wt.%), consisting of helically coiled cellulose microfibrils, bound together by an amorphous lignin matrix. Hemicellulose (11.02 wt.%) of the DRFs was acted as a compatibilizer between cellulose and lignin. Lignin content (11.35 wt.%) present in the DRFs may be act as a protection agent against the fungal attack. The Wax content of DRF is 0.23 wt.% which create disturbance to interfacial bond between fibers and matrices when fabricate the composite. The moisture and ash content of DRF is 5.19 wt. % and 6.23 wt.% respectively. The chemical composition of DRFs was compared with that of different natural fibers and conveyed in Table 1.

3.4. FT-IR spectrum analysis

The main chemical functional group presented in the DRF was detected through FT-IR spectrum is presented in Fig. 3. The peaks obtained at 3296 cm\(^{-1}\) is corresponds to O–H stretching of α-cellulose. The peak observed at 2914 cm\(^{-1}\) could be assigned to C–H stretching vibration of CH in cellulose component. The peak seen at 2796 cm\(^{-1}\) shows CH stretching of hemicelluloses. The peak attained at 2357 cm\(^{-1}\) represents C–C stretching of wax. The absorption peak at 1737 cm\(^{-1}\) corresponds to carbonyl group (–C=O) stretching vibration of the hemicellulose. The broad and strong absorption peaks noticed at 1642 and 1028 cm\(^{-1}\) were endorsed to –C=O stretching vibration of lignin. A small peak existing at 1346 cm\(^{-1}\) represents C=O stretching of hemicelluloses (Table 2).
3.5. XRD analysis

The XRD pattern of the DRF is displayed in Fig. 4. The two enlarged diffraction peaks at 2θ = 15.58° (110) and 22.41° (002), are clearly seen in most of the bio-fibers. The peak at 2θ = 15.58° point out the existence of amorphous constituents in DRF. The peak at 2θ = 22.31° denotes the amorphous constituents in the crystallographic plane. The crystallinity index of DRF was calculated as 57.32% which is higher than that of other bio fibers, such as C. grandis (52.17%), F. foetida (52.6%), Saharan Aloe vera (56.5%), S. cordifolia (56.92%), C. pangorei (41%), Acacia leucophloea (51%), Acacia Arabica (51.72%) and lower than that of Jute (71%) and Hemp (88%). In addition, the crystallite size (CS) of the DRFs was decided as 19.01 nm and higher CS minimizes the water absorption and chemical reaction ability of the fiber.

3.6. Thermal analysis

3.6.1. Thermogravimetric analysis

Generally, bio-fiber reinforced thermoplastic composites are manufactured under the high temperature, so it is important to evaluate the thermal stability of bio-fiber at various temperatures in order to: (i) choose the exact temperature range for manufacturing of composites, and to (ii) avoid the loss of fibers properties. The thermal degradation performance of DRFs was analyzed using TG and DTA curves shown in Fig. 5(a). From TG curve it is noticed that DRF has typically four stages of degradation. The initial degradation happened from room temperature to 232.20 °C with the weight loss of 11% which was associated with the elimination of moisture from the DRF. The next stage of degradation occurred from 232.20 °C to 295.39 with the weight loss of 2.61% which related to the reduction of the hemicellulose of the DRF. The combined degradation of the cellulose, hemicellulose and the huge quantity of the stable materials degraded between 295.39 and 348 °C with a weight loss of 51.43%. The fourth stage of degradation developed between the temperature range of 348 °C and 569.96 °C with a weight loss of 30.19% indicates the degradation of lignin. Comparable degradation performance was detected in the various bio-fibers such as C. grandis, F. foetida, C. pangorei and Agave Americana.

3.7. Differential scanning calorimetry

The kinetic activation energy is one of the noteworthy factor in the estimation of the thermal stability of DR fibers (Fig. 5(b) shows DSC curve). Broido’s plot of DRF is shown in Fig. 5(c). From this it is clear that the kinetic activation energy (Ea) is 68.784 kJ/mol. This activation energy of DRF is higher than the Coccinia grandis fiber (67.02 kJ/mol), Saharan Aloe vera cactus leaves fiber (60.20 k J/mol) and lower than the C. quadrangularis root (74.18 kJ/mol) and S. cordifolia (73.1 kJ/mol).

---

Table 2 – FT-IR peak positions and allocations of chemical stretching in the DRF.

<table>
<thead>
<tr>
<th>Peak positions (wave number, cm⁻¹)</th>
<th>Functional group</th>
<th>Corresponding chemical constitution</th>
</tr>
</thead>
<tbody>
<tr>
<td>3296</td>
<td>OH stretching</td>
<td>α-Cellulose</td>
</tr>
<tr>
<td>2914</td>
<td>CH stretching</td>
<td>α-Cellulose</td>
</tr>
<tr>
<td>2796</td>
<td>CH stretching</td>
<td>Hemicellulose</td>
</tr>
<tr>
<td>2357</td>
<td>C=O stretching</td>
<td>Wax</td>
</tr>
<tr>
<td>1737</td>
<td>C=O stretching</td>
<td>Hemicelluloses</td>
</tr>
<tr>
<td>1642</td>
<td>C=O stretching</td>
<td>Lignin</td>
</tr>
<tr>
<td>1346</td>
<td>C=O stretching</td>
<td>Hemicelluloses</td>
</tr>
<tr>
<td>1028</td>
<td>C-OH of stretching</td>
<td>Lignin</td>
</tr>
</tbody>
</table>
From the above analysis it is proved that the DRFs can be potentially used as reinforcements in thermoplastic and polymers whose processing temperature is lower than 230 °C.

3.8. Scanning electron microscopy (SEM)

Fig. 6 shows the SEM micrographs of Dracaena reflexa fibers for morphology analysis. The figure displays a general view of cellulose fibers with a rough surface suitable for a good bond with matrix polymer. It can be clearly seen in the figure, there is no cracks, micro voids and helical fibrils on the surface of DRFs. So it is revealed that these fibers can be used as reinforcement in polymer composite manufacturing.

3.9. Atomic force microscopy

Fig. 7(a)–(d) shows the 3D, 2D images, line profile and parameters of atomic force microscopy analysis of DRFs. The average roughness (Ra) value of DRFs is estimated as 22.769 nm which is greater than the F. foetida (18.005 nm), S. cordifolia (6.712 nm), Acacia planifrons (0.708 nm), and lower than the C. pangorei (625 nm). The higher Ra value of DRF signifies the surface of the fiber has less impurity and lignin. The roughness skewness (Rsk) value of DRF is −0.0875. This negative value designated that DRF surface contain lot of pores. The nature of the surface (spiky or rough) of fibers is predicted through roughness kurtosis (Rku) value. Rku value is larger than 3 indicate that is spiky surface, if it is less than 3 directs that is rough surface. The Rku value of DRF is 3.110 nm which signify that surface of DRF is spiky in nature, so a slight surface modification needed to improve the surface roughness before DRF used as reinforcement in composite. Other surface parameters such as ten-point average roughness (Rt), maximum peak-to-valley height (Rh) and root mean square roughness (Rq or Rrms) of DRF were 113.013 nm, 152.772 nm, 28.527 nm, respectively.

3.10. EDX analysis

Fig. 7(e) offered weight and atomic percent of elements scattered on the surface of the DRF. C (carbon), O (oxygen) and Cl (chlorine) are the existing peaks of DRF. The elements existing in of DRF and other natural fibers such as Calotropis procera, C. grandis. L., F. foetida and sugar palm fibers were presented in Table 3. Carbon and oxygen are the major elements present in the EDX spectrum of DRF which is the usual outcome of plant fiber. DRF has 66.43% and 34.20% weight of C and O,
respectively, which is more than that of *C. procera*, *C. grandis*, and lower than *F. Foetida*, sugar palm fibers.

### 3.11. C\textsuperscript{13} (CP-MAS) NMR spectroscopy

To verify the structural features of DRF solid state NMR (C\textsuperscript{13} NMR (CP-MAS)) experiment was conducted. Fig. 8 displays the C\textsuperscript{13} CP-MAS NMR spectrum of DRFs. Amorphous cellulose of DRF was identified at 64.074 ppm (C\textsuperscript{6}). The resonance peaks from 76.158 to 73.936 ppm were assigned to the C\textsuperscript{2}, C\textsuperscript{3}, and C\textsuperscript{5} carbons ring of cellulose. Two peaks at 85.473 and 90.002 ppm were endorsed to C\textsuperscript{4} carbon in the crystalline region. The peak at 22.577 ppm allocated to acetyl groups of hemicelluloses. Among the peaks observed at 76.158–73.936 ppm are maximum intensity which indications that higher amount of cellulose present in DRF. The comparable peaks were noticed in the several bio-fibers such as *F. Foetida*, *Ficus religiosa*, *Thespesia Lampas*, Napier grass and coconut fiber.

<table>
<thead>
<tr>
<th>Elements</th>
<th>Calotropis procera Weight (%)</th>
<th>Atomic (%)</th>
<th>Coccinia grandis L. Weight (%)</th>
<th>Atomic (%)</th>
<th>Furcraea Foetida Weight (%)</th>
<th>Atomic (%)</th>
<th>Sugar palm fibers Weight (%)</th>
<th>Atomic (%)</th>
<th>Dracaena reflexa Weight (%)</th>
<th>Atomic (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>61.00</td>
<td>67.63</td>
<td>44.67</td>
<td>51.91</td>
<td>66.43</td>
<td>72.50</td>
<td>87.93</td>
<td></td>
<td>65.13</td>
<td>71.54</td>
</tr>
<tr>
<td>O</td>
<td>38.80</td>
<td>32.30</td>
<td>54.38</td>
<td>47.58</td>
<td>33.57</td>
<td>27.50</td>
<td>5.86</td>
<td></td>
<td>34.20</td>
<td>28.21</td>
</tr>
<tr>
<td>Na</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td></td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Mg</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td></td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Al</td>
<td>0</td>
<td>0</td>
<td>0.18</td>
<td>0.09</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td></td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Si</td>
<td>0</td>
<td>0</td>
<td>0.67</td>
<td>0.36</td>
<td>0</td>
<td>0</td>
<td>5.13</td>
<td></td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>P</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td></td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Cl</td>
<td>0.2</td>
<td>0.06</td>
<td>0.38</td>
<td>0.06</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td></td>
<td>-</td>
<td>0.67</td>
</tr>
<tr>
<td>K</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td></td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Ca</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td></td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>S</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td></td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

Fig. 6 – SEM images of DRF.
Fig. 7 – (a) 3-D roughness surface texture, (b) 2-D roughness surface texture, (c) 2-D line diagram for roughness measurement of DRF, (d) roughness parameters, and (e) EDX analysis of DRF.
Fig. 8 – $^{13}$C (CP-MAS) NMR spectroscopy analysis.

4. Conclusion

The physico-chemical, thermal, tensile, structural and morphological properties of DRF was examined in this research work. The density of extracted DRF 790 kg/m$^3$ was vaguely increased, however, it is lower than E-glass fiber (2500 kg/m$^3$) and carbon fiber (1570 kg/m$^3$). The result of the chemical analysis and FT-IR analysis showed reduction in hemicelluloses and greater cellulose content in DRF can use as a reinforcing material in polymer matrix composite. The single fiber tensile test revealed that, the DRF also good in tensile strength and the young’s modulus so it is advantageous to produce lightweight applications. From EDX analysis, it is confirmed that the carbon and oxygen were the main constituents of DRF and exposes that the fiber is organic in nature. Natural fibers with a rough surface essential feature for adhesion with polymer matrix was observed through SEM.

Conflicts of interest

The authors declare no conflicts of interest.

Acknowledgement

This research was partly supported by the King Mongkut’s University of Technology North Bangkok through the PostDoc Program (Grant No.KMUTNB-61-Post-001 and KMUTNB-62-KNOW-37). The authors are also thankful to Universiti Putra Malaysia for supporting this work through HICOE Grant No; 6369108.

References


