Original Article

Characterization of cellulosic fibre from Phoenix pusilla leaves as potential reinforcement for polymeric composites

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Phoenix pusilla fibres (PPFs) were extracted from the leaves of a plant belonging to the family of Arecaceae, which is widely grown in Sri Lanka and Southern parts of India for some medicinal purposes. Their use as possible reinforcement in hydrophobic polymeric matrix composites is yet to be studied and for this reason, the better understanding of their microstructure, chemical composition, and mechanical properties becomes essential. In this view, the present investigation deals with the study on the effect of various chemical treatments on the mechanical, chemical structure, thermal and morphological behaviour of PPFs. The chemical treatment of fibres was initially involved with NaOH and then followed by benzoyl peroxide, potassium permanganate and stearic acid solution at different concentrations for the suitable time interval. The results revealed that chemical treatments help in diminishing the amorphous content from the fibres, while the FTIR analysis clearly indicates the removal of many polar impurities. The increase in crystallinity index and crystal size was seen in all the modified fibres when compared with the raw ones. The improved thermal stability behaviour in all the chemically treated fibres was demonstrated by thermogravimetric and differential scanning calorimetry analysis. The tensile properties of the fibres were analyzed through ASTM standard and finally, the surface morphology was analyzed using scanning electron microscopy. All the favourable results indicated that the PPFs could be used in the applications of natural fibre composites.

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1. Introduction

The present world scenario, where the petroleum resources get depleted at a rapid rate and also the need of more eco-friendly materials has made the investigators, scientists and industrialists to substitute the traditional man-made materials by sustainable natural fibres to be used as reinforcement in polymer matrix composites [1,2]. There occurs a large number of different lignocellulosic plant species that offer potential fibre and very few among them find utilized as reinforcements in composites [3]. Some of the fibres that have been successfully characterized by the researchers include bamboo, jute, banana, sisal, hemp, kenaf, century plant, date palm, needles grass and many others [4–7]. The merits of these natural fibres which have made them replace synthetic fibres, such as glass and carbon to be used as reinforcing agents in composites are their low cost, availability in abundance, biodegradability, fracture resistance, minimal health hazards and moderate mechanical properties [8–10]. Although the above-said advantages make natural fibres to be attractive materials, their lack of interfacial bonding with polymer matrix may result in poor adhesion when used as reinforcements and which in turn would affect the mechanical properties of the composites [11,12]. The polymer matrix offers high resistance and interfacial bonding properties to natural fibres while helping them to sustain their mechanical and chemical characteristics. Also, they help to keep the fibres in the desired orientation as well as protect them from any type of environmental damages [13]. The solution for this problem is to improve the bonding compatibility between hydrophobic fibres and hydrophobic matrices through fibre surface pretreatment techniques and/or coupling agents such as sodium hydroxide (NaOH), acrylates, peroxides, permanganates, silanes, stearic acid or acetylated reagents [14–16].

The main aim of the present study is to identify chemical composition, mechanical properties and microstructural behaviour of a new kind of fibres extracted from the leaves of Phoenix pusilla (P. pusilla) plant of the Arecaaceae family. The P. pusilla plant is widely found in southern India (Eastern Ghats of Tamil Nadu and southern region of Kerala), Sri Lanka and grows in the lowlands, ridges, and hills. Their stem usually grows up to 6 m height and are covered with persistent leaf bases vertically oriented on the trunk. Leaves are of 1.5 m long with their leaflets being very sharp leaflets stiff with very sharp needle-like apices, arranged at a distance of 2–5 cm, 16.5–20 × 1.5–2 cm, grouped into 4, basal leaflets spiny, not grouped, arranged in one or more planes of orientation; leaf sheath with fibres. Their distinguished trunks which are covered with distinct leaf-base scars have made them one of the most popularly cultivated flowering plants. They possess some features like drought tolerance, slow-growing and could be used in some medicinal applications such as burning sensations, cardiac debility, peptic ulcer, and general weakness. The leaflets are used in making mats [14–19]. Fig. 1 shows a P. pusilla plant and their leaf fibres.

In spite of the above mentioned medicinal usage of P. pusilla plants, hardly any literature exists on the fibres extracted from these plants. All plant materials invariably possess cellulose content but a systematic analysis of them will help in identifying proper fibre source especially when required in large quantities for industrial scale applications. Moreover, such readily available data may find use as a quick reference for commercial level screening from fibres of different sources. Hence, in this study, an effort has been made to carry out four different chemical treatments of raw fibres like sodium hydroxide (NaOH), benzoyl peroxide, potassium permanganate, stearic acid on the surfaces of fibres and to understand the chemical composition, microstructure, thermal behaviour, mechanical properties, crystallinity index and crystallite size through various characterization techniques.

2. Materials and methods

2.1. Materials and extraction of P. pusilla fibres

The P. pusilla plant leaves were collected from a village in Virudhunagar district near Madurai road, Tamil Nadu, Southern India. The district has a humid climatic conditions and receives annual rainfall in the range of 780 mm approximately. The extracted fibres from the matured and fallen leaves of P. pusilla plant were initially soaked in water for three weeks, later the top and bottom layers from the leaf fibres were separated using a smooth brush. Then these separated fibres were thoroughly cleaned using purified water and dried in the sunlight for 7 days. Finally, the dried fibres were placed in an oven for a day to eradicate the residual moisture content [20,21]. The various chemicals used for surface modification were purchased from Sri Raghavendra Chemicals, Bengaluru, Karnataka, India.

2.2. Chemical modifications of PPFs

PPFs were chemically treated with four different solutions and treatment was carried out at the Department of Chemistry, Malnad College of Engineering, Hassan. The treatments applied for fibres are as below.

2.2.1. Alkali-treated PPFs

The treatment of natural fibres by NaOH is the most common. The PPFs were treated with alkali using a 6% (w/v) NaOH solution for 45 min of soaking time. Later, the treated PPFs are washed with deionized water, followed by the addition of a few drops of 0.1N hydrochloric acid to remove excess impurities [15].

2.2.2. Benzoyl peroxide-treated PPFs

For treatment with benzoyl peroxide, the PPFs were immersed in 6% benzoyl peroxide in acetone for 30 min. The treated PPFs were washed and air-dried for 24 h [15].

2.2.3. Potassium permanganate-treated PPFs

For potassium permanganate treatment, the PPFs were immersed in 0.5% potassium permanganate in acetone for 30 min. The treated PPFs were washed and air-dried for 24 h [15].
2.2.4. Stearic acid-treated PPFs
For stearic acid treatment, initially, 1% of stearic acid was liquefied in ethyl alcohol. Then this solution was added drop by drop on PPFs which were placed in a stainless steel container through continuous stirring method. Later, the treated fibres were dried in an oven at 80 °C [15].

2.3. Chemical analysis of PPFs
The cellulose and amorphous (hemicellulose and lignin) contents were estimated using standard analytical methods. The wax content was measured using the Conrad method, while the moisture content was determined using Sartorious MA45 electronic moisture analyzer. The ash content was quantified using the standard ASTM E 1755-01 method and density of PPFs was determined as per the literature method [22,23]. For each test, five PPF samples were considered and average values have been reported.

2.4. Fourier transform-infrared spectrometry (FTIR) analysis
FTIR analysis was carried out on raw and chemically modified PPFs using Shimadzu (FTIR-8400S, Japan) spectrometer in the frequency range 4000–400 cm⁻¹. A total of 32 scans were taken for every sample with a resolution of 2 cm⁻¹ and to record FTIR spectra, two mg of finely powdered fibres were mixed with KBr to form pellets.

2.5. X-ray diffraction (XRD) analysis
The phase changes in raw and modified PPF fibres in terms of crystallinity index and crystallite size can be analyzed by powder X-ray diffraction (XRD) technique. X’Pert-Pro diffractometer with a current flow of 30 mA with 40 kV and with a copper terminal material maintained at a temperature of 25 °C is utilized in the present XRD analysis. The diffracted intensity of Cu Kα radiation with a wavelength of 0.154 nm operating in the range of 10° ≤ 2θ ≤ 80° (step size = 0.05) condition is retained throughout the analysis. The crystallinity index (CI) was assessed by Eq. [1] [24]:

\[
CI = \frac{I_{200} - I_{AM}}{I_{200}}
\]

where \(I_{200}\) indicates the maximum intensity of 200 lattice plane at a 2θ angle between 22° and 23° and \(I_{AM}\) indicates the minimum intensity of amorphous material taken at an angle of 2θ at 180. While the crystallite size was estimated considering the crystallographic plane (2,0,0) and using Scherrer’s expression as mentioned in Eq. [2] [25]:

\[
CS_{200} = \frac{k\lambda}{\beta_{200}\cos\theta}
\]

where \(K = 0.89\), is the Scherrer’s constant, \(\lambda = 0.1541\) nm is the wavelength of the radiation, \(\beta\) represents the peak’s full-width at half-maximum in radians and \(\theta\) is the corresponding Bragg angle.

2.6. Thermogravimetric analysis (TGA)
TGA was performed to identify the thermal stability of PPFs and was carried out using Jupiter simultaneous thermal analyzer (model STA449 F3, Netzsch, Germany). The analysis was recorded under the nitrogen atmosphere and maintained at a heating rate of 10 °C/min and temperature ranging between 25 °C and 700 °C [26].

2.7. Differential scanning calorimetry (DSC) analysis
The thermal transitions of raw and modified PPFs were studied by DSC using a Mettler-model DSC 822. Two to three grams of fibre samples in each treatment group were enclosed in an aluminium pan. The samples were heat scanned at a temperature ranging from 50 °C to 700 °C with a heating rate maintained at 10 °C/min in this analysis [27].

2.8. Tensile testing
Fibre diameters were measured using a polarized light microscope (Nikon Eclipse LV 150, 50× magnification). The tensile
strength of the fibres was determined by selecting at least 20 samples in each group using a computer interfaced INSTRON universal testing machine (ASTM D 3379) of the crosshead speed 1 mm/min. The entire analysis has been carried at a relative humidity of 65% and the temperature maintained around 24°C.

2.9. **Surface morphological analysis by SEM**

The microstructure and morphology of the PPFs were examined by scanning electron microscopy (SEM) using VEGA 3 TESCAN. All the samples were coated with a thin gold covering to improve the image resolution as well as to avoid accumulation of electrostatic charge during analysis.

### 3. Results and discussion

#### 3.1. **Chemical analysis of PPFs**

The chemical composition as well as the density of raw and various chemically treated PPFs are shown in Table 1. The chemical constituents and physical properties of any natural fibre depend upon the climatic conditions and the characteristics of soil regions on which they grow, plant age and the extraction methods employed for their separation from plants [28]. The cellulose content plays an important role in contributing to the enhancement of mechanical properties of the fibres and various chemical treatments employed in the present analysis show that upon surface modification there was a vast increase in cellulose wt.% in the PPFs when compared to untreated fibres. On the other hand, hemicellulose and lignin are accountable for poor moisture absorption, thermal degradation, and rigidity respectively. The lower hemicellulose and lignin wt.% in all the modified PPFs compared to raw fibres confirms that chemical treatment helps in reducing the hydrophilic behaviour of fibres. The diminished moisture and wax content in all modified PPFs confirms that there would be improved bonding between the fibre and matrix when used in composites [29,30].

The descending ash wt.% in treated fibres will have a direct impact on the mechanical properties. The raw PPFs were found to have a density of around 0.211 g/cm³, while the various chemically treated fibres showed improved density values ranging from 0.228 to 0.362 g/cm³. The slight improvement in the density values of chemically treated PPFs may be due to partial removal of hemicelluloses and lignin from the fibres and also could be attributed to the densification of fibre cell walls and filling up of pores by grafted molecules [31,32].

#### 3.2. **FT-IR analysis**

Fig. 2 shows the FTIR spectrum of raw and modified PPFs, in which only the most interesting absorbance peaks have been identified and highlighted. A broad absorption peak in the region 3454–3100 cm⁻¹ is attributed to the characteristic O–H stretching vibration and hydrogen bond of the hydroxyl groups [33]. The broad peak at 2928 cm⁻¹ corresponds to C–H stretching vibration from CH and CH₂ in cellulose and hemicellulose components [34]. The absorbance band at 2347 cm⁻¹ relates to C≡N stretching of wax and is seen in raw PPFs, which demonstrates the existence of waxy and other impurities in them. The two peaks observed at 1760 and 1566 cm⁻¹ is ascribed to C=O stretching vibration of the amide group in hemicellulose and lignin respectively. A noticeable peak at 1028 cm⁻¹ in raw and modified PPFs is attributed to C–OH stretching of lignin and confirms that the surface modification helps in eliminating hydroxyl and carboxyl groups from the fibres, thereby making them have good adhesion properties [4,16]. The peak positions and their possible allocations are depicted in Table 2.

#### 3.3. **XRD analysis**

The X-ray diffraction patterns of raw and modified PPFs are illustrated in Fig. 3. As seen in Fig. 3, the main characteristic peak occurred at 2θ = 15.07° and 2θ = 16.09° and correspond to (002) crystallographic plane. While the main and least crystalline peaks were experienced at 2θ = 22.67° and 2θ = 18.12° respectively. The CI of fibres were quantified using Eq. [1] and are tabulated in Table 3. The improved CI values in all the modified PPFs validate that the chemical treatment essentially results in better packing of cellulose chains in the fibres by removal of amorphous contents and increasing the cellulose

![Table 1 - Chemical composition of raw and modified PPFs.](image)

<table>
<thead>
<tr>
<th>Fibre</th>
<th>Cellulose (wt.%)</th>
<th>Hemicelluloses (wt.%)</th>
<th>Lignin (wt.%)</th>
<th>Wax (wt.%)</th>
<th>Ash (wt.%)</th>
<th>Density (g/cm³)</th>
<th>Other content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw</td>
<td>59.46</td>
<td>18.56</td>
<td>8.28</td>
<td>0.33</td>
<td>4.56</td>
<td>0.211</td>
<td>8.45</td>
</tr>
<tr>
<td>NaOH</td>
<td>72.14</td>
<td>2.32</td>
<td>5.14</td>
<td>0.18</td>
<td>4.65</td>
<td>0.323</td>
<td>3.46</td>
</tr>
<tr>
<td>SA</td>
<td>75.37</td>
<td>2.76</td>
<td>4.06</td>
<td>0.11</td>
<td>5.56</td>
<td>0.346</td>
<td>4.42</td>
</tr>
<tr>
<td>BP</td>
<td>73.24</td>
<td>2.14</td>
<td>3.92</td>
<td>0.22</td>
<td>4.83</td>
<td>0.362</td>
<td>6.43</td>
</tr>
<tr>
<td>PP</td>
<td>72.88</td>
<td>2.98</td>
<td>4.26</td>
<td>0.26</td>
<td>5.01</td>
<td>0.228</td>
<td>4.31</td>
</tr>
</tbody>
</table>

![Fig. 2 – FTIR spectra of raw, NaOH, SA, BP and PP treated PPFs.](image)
3.4. Thermogravimetric analysis

The main factor that is responsible in any natural fibre for limiting its use as reinforcement in biocomposites is its low thermal stability. In order to study the thermal stability of PPFs, thermogravimetric analysis was carried out and high-temperature degradation of this fibre was studied using TG curves, as portrayed in Fig. 4. The degradation occurred in three stages with initial weight loss of around 4–8% observed below 100 °C. The first stage of thermal degradation was visible at the temperature range 50–190 °C (weight loss of around 15%) and represented the elimination of volatile content, moisture and some waxy substances from the fibres [37–39]. The second stage of degradation was observed at the temperature range from 230 to 350 °C (weight loss of around 50% for raw and

crystallinity in treated fibres [35]. Furthermore, CS was also determined by using Eq. [2] and the values are tabulated in Table 3. The increased CS values in all modified fibres indicate the improvement in moisture and chemical resistivity properties of fibres. The large CS in modified fibres represents condensed surface areas due to lower chemical and water absorption behaviour in these fibres [36].

Table 2 – Peak positions and assignments of chemical groups in raw and modified PPFs.

<table>
<thead>
<tr>
<th>Raw PPF (Ca.)</th>
<th>NaOH (Ca.)</th>
<th>SA (Ca.)</th>
<th>BP (Ca.)</th>
<th>PP (Ca.)</th>
<th>Possible allocations</th>
</tr>
</thead>
<tbody>
<tr>
<td>3454</td>
<td>3454</td>
<td>3454</td>
<td>3454</td>
<td>3454</td>
<td>OH-stretching</td>
</tr>
<tr>
<td>2928</td>
<td>2928</td>
<td>2928</td>
<td>2928</td>
<td>2928</td>
<td>CH-stretching</td>
</tr>
<tr>
<td>2347</td>
<td></td>
<td></td>
<td>-</td>
<td>-</td>
<td>C=C-stretching</td>
</tr>
<tr>
<td>1760</td>
<td>1760</td>
<td>1760</td>
<td>1760</td>
<td>1760</td>
<td>C=O-stretching (Amide)</td>
</tr>
<tr>
<td>1566</td>
<td>1566</td>
<td>1566</td>
<td>1566</td>
<td>1566</td>
<td>C=O-stretching</td>
</tr>
<tr>
<td>1028</td>
<td>1028</td>
<td>1028</td>
<td>1028</td>
<td>1028</td>
<td>C—OH-stretching</td>
</tr>
</tbody>
</table>

Table 3 – Crystalline characteristics of the raw and modified PPFs.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Crystalline index (%)</th>
<th>Crystallite size CS(0 0 2) (nm) (Ca.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw PPF</td>
<td>42.6</td>
<td>7.98</td>
</tr>
<tr>
<td>NaOH</td>
<td>46.6</td>
<td>9.09</td>
</tr>
<tr>
<td>SA</td>
<td>50.66</td>
<td>13.11</td>
</tr>
<tr>
<td>BP</td>
<td>55.76</td>
<td>15.97</td>
</tr>
<tr>
<td>PP</td>
<td>58.78</td>
<td>16.77</td>
</tr>
</tbody>
</table>

Table 4 – TGA summary of raw and optimally treated PPFs.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$T_{30%}$ (°C)</th>
<th>$T_{max}$ (°C)</th>
<th>$Y_{2}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw PPF</td>
<td>182</td>
<td>217</td>
<td>14.1</td>
</tr>
<tr>
<td>NaOH</td>
<td>186</td>
<td>219</td>
<td>13.2</td>
</tr>
<tr>
<td>SA</td>
<td>232</td>
<td>268</td>
<td>6.1</td>
</tr>
<tr>
<td>BP</td>
<td>216</td>
<td>255</td>
<td>8.8</td>
</tr>
<tr>
<td>PP</td>
<td>210</td>
<td>252</td>
<td>9.6</td>
</tr>
</tbody>
</table>

Table 5 – Tensile properties of raw and optimally treated PPFs.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Raw PPF</th>
<th>APPF</th>
<th>SPPF</th>
<th>BPPF</th>
<th>PPPF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter(µm)</td>
<td>40</td>
<td>35.43</td>
<td>34.75</td>
<td>36.76</td>
<td>35.85</td>
</tr>
<tr>
<td>Tensile strength (MPa)</td>
<td>660 ± 7.5</td>
<td>740 ± 5.54</td>
<td>756 ± 5.65</td>
<td>752 ± 3.87</td>
<td>742 ± 3.87</td>
</tr>
<tr>
<td>Young’s modulus (GPa)</td>
<td>4.76 ± 0.6</td>
<td>5.81 ± 0.8</td>
<td>6.93 ± 0.4</td>
<td>6.90 ± 0.1</td>
<td>5.85 ± 0.1</td>
</tr>
<tr>
<td>Elongation at break (%)</td>
<td>0.44 ± 0.11</td>
<td>2.43 ± 0.05</td>
<td>3.36 ± 0.07</td>
<td>2.67 ± 1.12</td>
<td>1.65 ± 0.75</td>
</tr>
</tbody>
</table>

NaOH treated PPFs and 55–60% for SA, BP and PP treated PPFs) and shows the degradation in α-cellulose and hemicellulose contents. The third and final degradation stage occurred at 390–510 °C and corresponded to the degradation of lignin and other non-cellulosic substances in the PPFs. The TG curves in Fig. 4 confirm the better thermal stability characteristics of PPFs. Table 4 summarizes the detailed $T_{20\%}$ (Decomposition temperature at 20% weight loss), $T_{\text{max}}$ (Decomposition temperature at the maximum decomposition rate), and $Y_C$ (Residual yield at 600 °C) values.

3.5. DSC analysis

The DSC curve of raw and modified PPFs is shown in Fig. 5. Two endothermic peaks were observed in the temperature range of 330–490 °C and may be attributed to the loss of moisture from the fibres. The peaks seen in the DSC profile are in good agreement with the mass loss seen in this temperature range in TG analysis of common natural fibres [40].

3.6. Tensile testing

The tensile properties of raw and various chemically treated PPFs are tested and tabulated in Table 5. It is observed that all the diameters of chemically treated fibres were reduced simultaneously compared to the raw fibres. The concentration of 6% NaOH was the most effective surface modification among all the treatments. The average of five samples was chosen from raw and various treatments for the tensile test, respectively. The average tensile strength and elongation at break of chemically treated PPFs are higher than those of raw fibres as shown in Table 5. The elimination of hemicellulose by various chemical treatments can be attributed to the tendency of the fibres to compact closely. It was found that the Young’s modulus is higher in raw fibres, while there is not a much significant difference between the chemically treated fibres. The tensile values of PPFs exhibit good properties and tend to be adequate reinforcements in polymer composites [37,38].

3.7. Surface morphological analysis

The surface morphology of raw and chemically treated PPFs was examined by SEM. This technique provides an excellent method to understand the effect of chemical treatments that
can significantly eliminate impurities due to the reduction of hydrophilicity in chemically treated fibres [37]. The corresponding SEM images of the raw and chemically treated PPFs are shown in Fig. 6. The SEM micrograph (Fig. 6(a)) of PPFs shows the longitudinal surfaces of the raw PPFs. It can be clearly seen that PPFs consists of a parallel set of microfibris. In addition, there are waxes, oils and other considerable impurities on the surface of the fibre. In general, it is believed that chemical treatments remove non-cellulose materials and impurities from the surface of the fibre. Fig. 6(b)–(e) shows the micrographs of PPFs treated with sodium hydroxide, benzoyl peroxide, potassium permanganate, and stearic acid, respectively. It can be seen that several chemical treatments caused an increase in the number of pores in the surface of the fibre. It also showed a very clean surface and the absence of impurities was very obvious. In fact, after treatment with stearic acid (Fig. 6(c)), it is observed that waxes, oils, and other impurities are completely removed without damaging the surface of the fibre. In addition, the treatment also damaged the surface of the fibre; These changes have benefit in the manufacture of composite materials when the natural fibres are used as reinforcement [35]. The rougher surface improves the mechanical interlocking adhesion between the fibres and the matrix. However, when the fibres were treated with potassium permanganate (Fig. 6(e)), it was discovered that the surface of the fibre was affected by a high concentration that caused damage to the surface topography [39,41,42].

4. Conclusions

The leaves from P. pusilla plant were directly extracted by a combined mechanical and water retting process, subsequently treated with four different chemical solutions and then distinguished by various means. The microscopy study carried out using scanning electron microscopy confirmed that upon surface modification by various chemical treatments, the surfaces of PPFs become rough mainly due to the elimination of hemicellulose and impurities such as oils and waxes. These microscopic analysis results were upheld by chemical analysis and FTIR studies. X-ray diffraction results confirmed the enhanced crystalline index and crystallite size in modified fibres compared to untreated fibre. The thermal stability of PPFs was found to be improved on various chemical treatments and was validated by thermogravimetric and differential scanning calorimetric studies respectively. The tensile properties of the fibres were found to increase after various treatments due to improved fibre structure. Finally, the characterization results imply the probable usage of PPF fibres for many prospective applications particularly as reinforcement of polymer composites.

**Funding**

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).

**Conflicts of interest**

The authors declare no conflicts of interest.

**Uncited reference**

[42].

**Acknowledgments**

The authors are thankful to Mr. Senthamaraikannan P and Mr. Vijay R who has provided advice and assistance in this research. The authors are also grateful to the National Institute of Technology, Tiruchirappalli for providing their lab facilities.

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