Effect of Alkaline Treatment on Decomposition Temperature of Fibres from Empty Oil Palm Bunch

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\begin{abstract}
This article reports an investigation of the decomposition temperature of fibres from empty oil palm bunch to achieve improved understanding of the processing temperature limitation of this fibre as reinforcement for thermoplastic composite manufacturing. The effect of alkaline treatments on their chemical decomposition and sample weight changes were performed, using Fourier transform infrared spectrometry (FTIR) and thermogravimetry (TG) techniques. The results show that the hemicellulose removal of fibres after treatment is a factor that provides an advantage on the thermal stability of the fibres. Both initial decomposition temperature ($T_d$) and maximum decomposition temperature ($T_{\text{max}}$) are minor increase. This could reduce the detrimental effect of thermal degradation on the single fibre performance and leads to shift its consolidation temperature ceiling from below 221°C (untreated fibres) to 243°C (5\% NaOH treated fibres) during composite consolidation.
\end{abstract}

\textbf{Index Terms}— alkaline treatment, natural fibres

\section{I. INTRODUCTION}

The plant fibres, such as oil palm bunch, cotton and flax, are renewable, low density and biodegradable materials, and can be used as reinforcement in the manufacturing of thermoplastic composites [1]. In addition, these eco-friendly materials have provided attractive replacement of glass fibres in terms of specific properties, although currently there are several disadvantages of using plant fibres as reinforcement materials due to high process temperature of melted thermoplastic matrix [2]. The fibres from empty oil palm bunch, like other plant fibres, have various physical constituents, which result in poor composite properties due to their thermal degradation at high consolidation temperature [3, 4]. The chemical composition of oil palm bunch is similar to that of wood.

In this work, the focus is on the decomposition temperature of fibres studies of oil palm bunch fibre to improve understanding of the influence of temperature on its thermal stability under alkaline treatment for process determination of the thermoplastic composite manufacturing. Fourier transform infrared spectrometry techniques and thermogravimetry were used to investigate the chemical composition and weight change of the oil palm bunch fibres, respectively.

\section{II. MATERIALS AND EXPERIMENTAL METHOD}

Before the decomposition temperature of fibres studies, the fibres were examined to define fibre density and fibre hydration levels.

\textbf{A. Materials}

The untreated fibre material from empty oil palm bunch was supported from Suksomboon Palm Oil co., Ltd., Chonburi province, Thailand. The fibres were heated at 120°C for 12 hours, then keep in a storage room.

\textbf{B. Treatment}

Fibres were dipped in NaOH solution (1, 2, 3, 4, 5\% by weight) for 20 minutes at room temperature. Accordingly, these fibres were washed in running water several times and dried 80°C for 12 hours.

\textbf{C. Fourier transform infrared spectrometry (FTIR)}

FTIR technique was used to investigate the chemical composition changes of treated and untreated fibres. The spectrum of the fibres was performed using a Shimadzu IRPrestige-21 spectrometer, between 700 and 3,700 cm\textsuperscript{-1} frequency range.

\textbf{D. Thermogravimetric analysis (TGA)}

The decomposition temperature of treated and untreated oil palm fibres was examined with TGA technique. The fibres were weighted of 10 mg and tested under argon atmosphere. Thermogravimetric (TG) and differential thermogravimetric (DTG) curves were recorded. These experiments were examined using a Setaram Labsys evo TG/DTA, TG/DSC thermal analiser. An argon flow rate of 20 ml/min was used between 25 and 600°C at heating rates of 10°C/min with an argon pre-heating process for 15 minutes.

\section{III. RESULTS AND DISCUSSION}

\textbf{A. Fibre density and moisture content}

Untreated fibres from empty oil palm bunch were found to have a fibre density of 0.99 g/cm\textsuperscript{3}. This result was in a range of values reported for oil palm fibres in other studies which were generally in the range 0.70 – 1.60 g/cm\textsuperscript{3} [5].
This is useful value for calculating fibre volume fraction and specific properties of the composite.

The fibres are hydrophilic material and can absorb moisture from atmosphere. Fig. 1 presents the drying rate of moist fibres during the drying process of 140°C. The average initial moisture content was 10.04% for arbitrary storage RH. It can be seen that a drying time of 60 minutes is an appropriate choice. Furthermore, this drying process will be a fibre preparation process before forming, reducing voids in the composites from water evaporation entrapment during layup.

\[ 	ext{Fig. 1. Moisture content as a function of time during a drying process at 140°C} \]

**B. Effect of alkaline treatment on the chemical decompositions of fibres.**

The transmittance bands of fibres from empty oil palm bunch in expression of their functional group wave number were shown in Fig. 2. Spectrums acquaint with the transmittance bands of chemical group characteristics of water, lignin, pectin, hemicellulose and cellulose substances of material without treatment.

A number of presence peaks observed confirms their existence of both cellulose and lignin before and after treatment. The spectrum range 3600 – 3100 cm\(^{-1}\) were attributed to the OH stretching of methanol and cellulose. A peak at 2916 cm\(^{-1}\) was attributed to the alkyl, aliphatic and aromatic in cellulose. A peak at 22851 cm\(^{-1}\) was attributed to the C-Hn stretching of cellulose. A peak of 1608 cm\(^{-1}\) was attributed to the water from moist fibres. Wave numbers of 1512, 1423, 1327 and 1161 cm\(^{-1}\) peak were attributed to the C=C stretching of lignin, C-Hn bending of aromatic rings in polysaccharides in cellulose and anti-symmetrical deformation of the C-O-C in lignin, respectively.

More important, a number of invisible peaks observed infers to their fibres chemical composition removal after treatment. A peak at 1724 cm\(^{-1}\) was attributed to the C=O stretching in hemicellulose and Methyl ester and Carboxylic acid in pectin. A peak at 1238 cm\(^{-1}\) was attributed to the C-H bending in hemicellulose. This analogism could be concluded that both hemicellulose and pectin were removed after alkaline treatment, particularly higher concentration of alkaline.

**C. Effect of alkaline treatment on the decomposition temperature of fibres.**

Fig. 3. shows weight loss (TG), its derivative (DTG) and second derivative (D\(^2\)TG) curves of fibres heating from 25°C to 600°C at 10°C/min heating rate. Table I shows the decomposition temperature of the fibre from Empty Oil Palm Bunch determined from these tests. DTG curve reveals to water vaporization from moist fibres between the temperature of 25 and 140°C.

Their drying rate were much lower than fibre moisture content as described in section 3.1, possibly due to a pre-heating process for 15 minutes of air releasing in thermogravimetric analyzer. Argon was used to remove the air inside the fibre chamber and could dehydrate the fibres. After dehydration, a shoulder peak between 220 and 320°C distributed to is the temperature of the thermal depolymerisation of glycosidic linkages of cellulose, pectin and hemicellulose [6-10]. Finally, the main peak (a range of 275 to 400°C) attributed to cellulose decomposition with high weight loss [11-14].

The initial decomposition temperature (T\(_0\)) and the maximum decomposition temperature (T\(_{\text{max}}\)) were calculated from the DTG and second time derivatives (D\(^2\)TG) curves, as described in Yao et al.’s report [15] and were shown in Table I. The results from this study reveal that the untreated fibres would be stable until 221.89°C and start to degrade rapidly. Nevertheless, fibre treatment could shift its T\(_0\) and T\(_{\text{max}}\) to higher temperature. For instance, 221.89°C and 243.56°C of T\(_0\) for no treated and 5% of NaOH treatment, respectively, and 300.05°C and 356.98°C of T\(_{\text{max}}\) for no treated and 5% of NaOH treatment, respectively.

From an engineering perspective, many steps processing of composite manufacturing are required, prior to treatment, in order to fulfill higher mechanical performance of single fibres and consequently reinforced composites. A small increase of composite processing temperature (only 17.67°C increased from 221.89°C of untreated fibres to 239.56°C) could not be useful due to most thermoplastics have their melting point lower than 220°C.

**TABLE 1**

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Initial decomposition temperature (T(_0)) [°C]</th>
<th>Maximum decomposition temperature (T(_{\text{max}})) [°C]</th>
</tr>
</thead>
<tbody>
<tr>
<td>No Treatment</td>
<td>221.89</td>
<td>300.05</td>
</tr>
<tr>
<td>Treated NaOH 1%</td>
<td>239.56</td>
<td>360.28</td>
</tr>
<tr>
<td>Treated NaOH 2%</td>
<td>239.62</td>
<td>353.88</td>
</tr>
<tr>
<td>Treated NaOH 3%</td>
<td>238.78</td>
<td>357.83</td>
</tr>
<tr>
<td>Treated NaOH 4%</td>
<td>244.50</td>
<td>359.82</td>
</tr>
<tr>
<td>Treated NaOH 5%</td>
<td>243.56</td>
<td>356.98</td>
</tr>
</tbody>
</table>
Fig. 2. FTIR spectrums of treated and untreated fibres from empty oil palm bunch.
Fig. 3. The thermogravimetric analysis curves of fibres; (a) No treatment, (b) NaOH 1%, (c) NaOH 2%, (d) NaOH 3%, (e) NaOH 4%, (f) NaOH 5%

Furthermore, more alkaline and processing requirement consume operation time and cost. On the other hand, a process with its consolidation temperature lower than $T_0$ (lower than 221.89°C) of composite processing, the fibre from Empty Oil Palm Bunch performance would be still not changed. High performance of plant fibre reinforced thermoplastic composites would actually achieve the same advantage composites.

IV. CONCLUSION

This article reports on the influence of alkaline treatment on decomposition temperature of fibres from empty oil palm bunch for investigating the maximum consolidation temperature of a forming process of fibre reinforced thermoplastic composites. The results can be concluded as follows:

A reduction of hemicellulose and pectin from the fibre after treatment.

The alkaline treatment results in a shift of the main degradation temperature of fibre materials to higher temperature.

The forming process of consolidation temperature lower than 221.89°C is recommended for untreated fibres from empty oil palm bunch material.
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REFERENCES


