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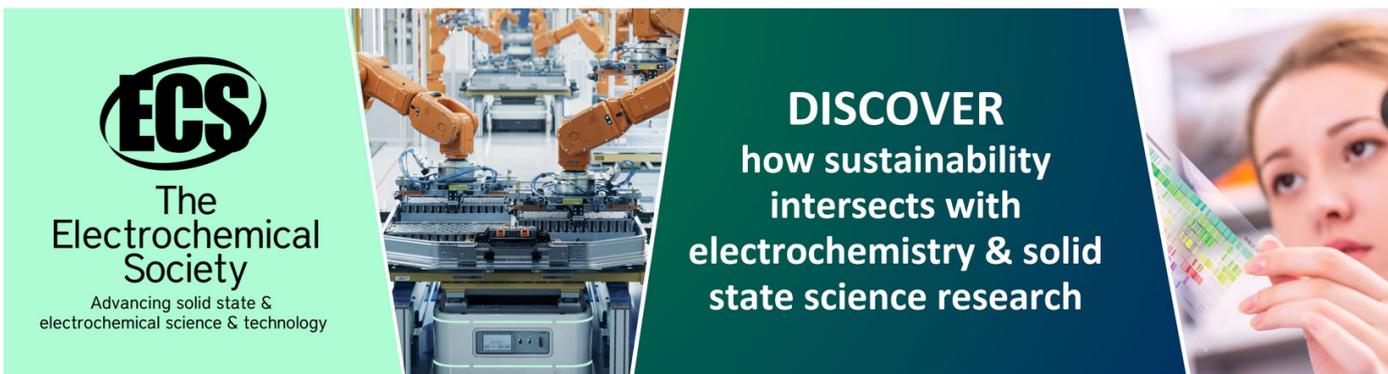
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The influence of alkaline treatment on thermal stability of flax fibres

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Abstract. The paper pursued the hypothesis that alkaline treatment removed hemicellulose and pectin from the flax fibres, the fibre thermal stability will be improved. The use of plant fibres as reinforcement in thermoplastic composites comes with the detrimental effect of thermal degradation on the tensile performance of fibres and composites, particularly during long consolidation times at high temperature. SEM was used to observe the treated fibre surface. The micrographs show that the treatment with a higher concentration of NaOH solution results in a more obviously rough fibre surface. Moreover, FTIR and TGA were used to examine the chemical decomposition and thermal stability, respectively. The spectra of treated materials indicate that both hemicellulose and pectin were dissolved from the fibre surface following treatment. Finally, TGA results revealed that the loss of mass belonging to hemicellulose and pectin in treated fibres results in a shift of the main degradation temperature to higher temperature.

1. Introduction

There has been a high sustainability agenda toward industrial products made from biodegradable or renewable materials. Consequently, it has been shown that there is a growing interest in the application of plant fibre thermoplastic composites [1-3]. Furthermore, research and development (R&D) in this area has resulted in the improvement of the performance of plant fibre composites in industries such as the automotive sector. Natural fibre composites offer several advantages such as eco-friendliness, safety from hazards and light weighting [4, 5].

Nevertheless, the low thermal stability of plant fibres is a limiting factor affecting the composite performance. Plant fibres are combustible materials. The natural properties and the various constituents of plant fibres can generate poor composite performance due to thermal degradation [6-8]. Thermal degradation of the plant fibres at high processing temperatures, typical of thermoplastic systems, is an important issue. The thermal degradation of plant fibre depends on a number of factors namely, chemical composition of materials, temperature and heating rate. The tenacity decreases and the degree of depolymerisation increases with an increase in temperature and/or dwell time at high temperature due to chain scission [7]. For example, the flax fibre retains its strength at 170°C for 120 minutes and at 200°C for 30 minutes, while it shows a 50% drop at 210°C for 120 minutes [7, 9]. Furthermore, the thermal decomposition temperature of fibres limits the choice of polymer as a matrix material due to the melted temperature of the polymer could be less than the thermal decomposition temperature of fibres. Low processing temperatures leads to low impregnation with high viscosity thermoplastic resins. In addition, volatile products from their chemical constituents may be generated



during heating and settle in the laminates. These lead to low impregnation and to the formation of voids during moulding processes [10].

The chemical composition of plant fibres comprises different compounds with various concentrations. For instance, flax fibre consists of 64.1 – 71.9 (%wt) of cellulose, 16.7 – 20.6 (%wt) of hemicellulose, 2.0 – 20.6 (%wt) of lignin, 8 – 12 (%wt) of moisture content, 1.8 - 2.3 (%wt) of pectin and 1.5- 1.7 (%wt) of wax [6, 11-13]. Hemicellulose and pectin seem to dominate the degraded fibre property due to these material's low thermal stability and low tensile strength. Hemicellulose in the fibres degraded with higher dwelling time at high temperature. Therefore, a decrease of hemicellulose and pectin content of the fibres should enhance their mechanical properties [14].

An attempt is to be made to improve the thermal stability of the fibres and consequently improve composite performance by using alkaline treatment methods. Alkaline treatment is a chemical treatment commonly used to prepare good quality fibres [15]. The removal of hemicellulose and pectin after the mercerisation process also improves the fibre strength and stiffness [10, 16, 17]. Moreover, this treatment increases the fibre rough surface, resulting in improved interfacial adhesion and mechanical performance of the composites.

2. Materials and experimental method

2.1. Materials

The flax fibres were soaked in NaOH solution of 1, 2, 3, 4, and 5% by weight of concentrations for 20 minutes at room temperature. Subsequently, these fibres were then followed by washing in running water several times to clean the fibre surface. The fibres were then dried in a hot air oven at 80°C for 8 hours.

2.2. Fourier transform infrared spectrometry

The Fourier Transform Infrared spectrometry (FTIR) technique was used to establish the chemical composition of treated and untreated flax fibres. In each measurement, five repeats of the treatments were tested at room temperature. The spectrum of the fibres was measured using a Bruker FTIR Tensor 27 spectrometer in attenuated total reflectance (ATR) mode, between 600 and 4000 cm^{-1} frequency range. Alcohol was used for cleaning the spectrometry after every test.

2.3. Thermogravimetric analysis

Thermal stability of treated and untreated flax fibres was studied by thermogravimetric analysis with three repeats for each material. All materials were stored in a plastic zip lock bag before testing. The fibres were cut to weights between 5 and 7 mg and were tested in a platinum pan. Thermogravimetric (TG) and differential thermogravimetric (DTG) curves were obtained. The analysis was conducted using a thermogravimetric analyzer/DSC TA SDT Q600, under nitrogen atmosphere. Nitrogen is commonly used as an inert gas which does not react with plant fibres. A nitrogen flow rate of 150 ml/min was used with a pre-heating process for 15 minutes and temperature range from room temperature to 600°C at heating rates of 10°C/min.

3. Results and discussion

3.1. Effect of alkaline treatment on the fibre surface

Fig.1(a) shows scanning electron microscopy (SEM) images of untreated flax fibre surface. It can be seen that the fibres are not uniform in shape and the diameter varies along the fibre. Figs. 1(b)-(f) show fibre treated by the concentrations of NaOH solution of 1, 2, 3, 4 and 5% by weight, respectively. These micrographs indicate that the treatment with a higher concentration of NaOH solution results in a more obviously rough fibre surface. The SEM micrographs reveal that alkaline treatment changes the fibre surface characteristics due to the treated fibres having lost some of their constituents, with a

relatively high roughness, particularly apparent in the treated fibres with 4 and 5%_wt NaOH solution, as shown in Figs. 1(e) and 1(f), respectively.

Furthermore, it is postulated that the rough surface of treated fibres could enhance mechanical keying at the interface region of the fibres and the matrix material in the composite. The advantages of improved fibre-matrix interfacial adhesion and improved consolidation resulting in the improved tensile properties of the composite could be achieved.

3.2. Effect of alkaline treatment on the chemical compositions of fibres

The chemical composition changes of the fibre surface after treatment were measured by FTIR. Infrared spectra of treated fibres of various treatment conditions are shown in Fig. 2. The spectra present the absorption bands of chemical group characteristics namely, cellulose, hemicellulose, pectin, lignin and water components. The absorption bands of treated and untreated fibres show both similar and different peaks. The presence of similar peaks observed confirms the existence of both hemicellulose and pectin before and after treatment. More important, very small peaks at 1243 and 1731 cm^{-1} , which exist in the untreated fibre (see the arrows in Fig. 2), weakened in the treated fibre. These peaks indicate that both hemicellulose and pectin were dissolved, leaving cellulose and lignin components after treatment.

3.3. Effect of alkaline treatment on the thermal stability of fibres

The effect of alkaline treatment on the differential thermogravimetric (DTG) peak position is shown in Fig. 3. An initial low peak, in the range of 40°C to 150°C is attributed to a mass loss of water from the moist fibres. After this peak, the DTG curve of untreated fibre shows a small shoulder peak between 230°C and 260°C, which is attributed to thermal degradation of the hemicellulose and pectin. Moreover, the curve also shows the main peak at 330°C, which is attributed to cellulose degradation. It can be seen from Fig. 3 that the alkaline treatment changes the DTG main peak position, shifting it to higher temperature, compared with untreated fibres. This shifting depends significantly on the concentration of NaOH solution. The main degradation temperature increases from 330°C for untreated fibre to 355°C for 5% NaOH mercerized fibres.

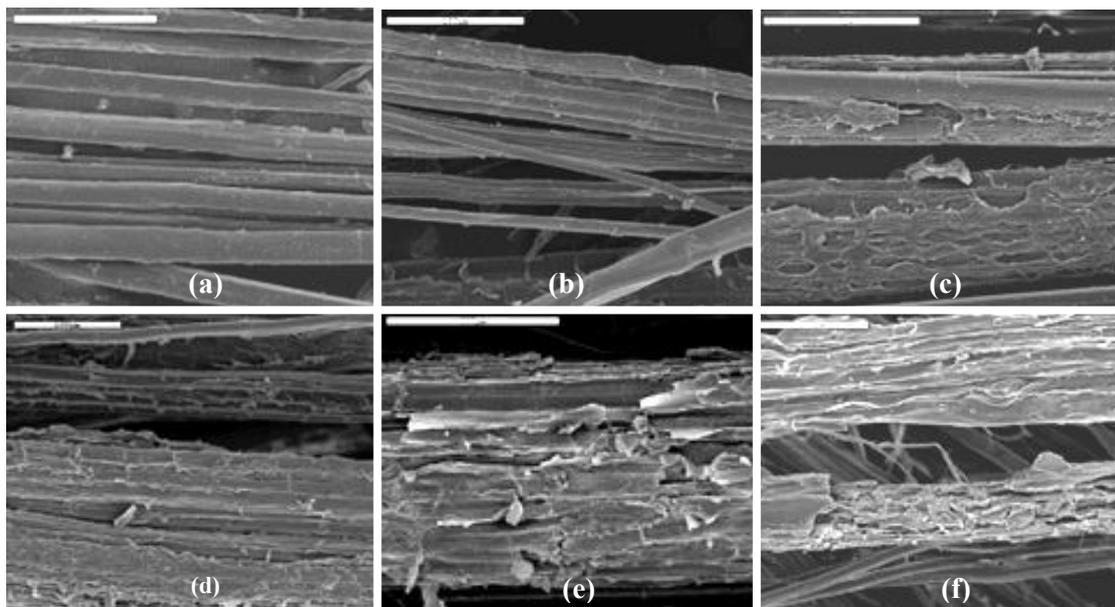


Figure 1. (a) SEM micrograph of untreated flax fibres (b) flax fibres treated with 1%_wt NaOH (c) flax fibres treated with 2%_wt NaOH (d) flax fibres treated with 3%_wt NaOH (e) flax fibres treated with 4%_wt NaOH (f) flax fibres treated with 5%_wt NaOH.

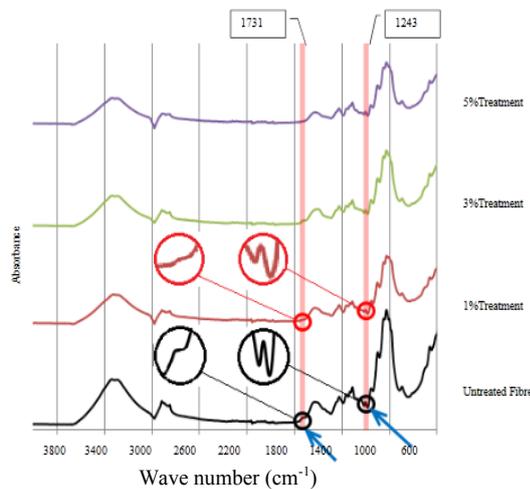


Figure 2. FTIR spectra of treated and untreated flax fibres.

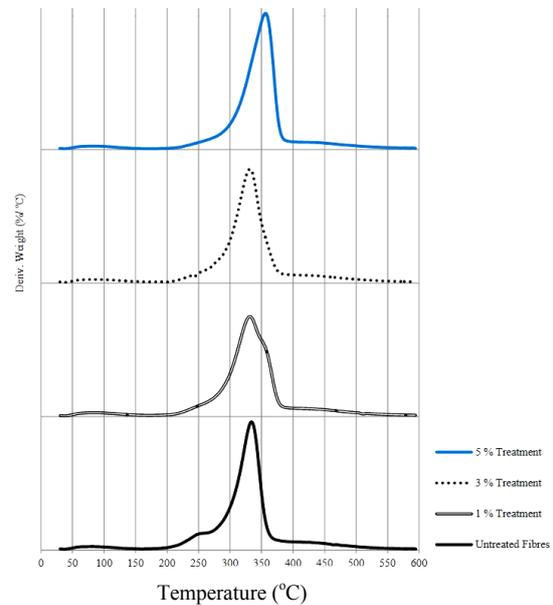


Figure 3. The differential thermogravimetric (DTG) curves of treated and untreated flax fibres.

The shoulder peak of the DTG curve, at about 230°C to 260°C, disappears after treatment with a higher concentration of NaOH solution, supporting the hemicellulose and pectin loss. Hemicellulose and pectin have low thermal stability, are removed from the fibre surface by the alkali. The long chain polymers, i.e., cellulose and lignin, are retained. Thus, the hypothesis is that treated fibres have prolonged thermal stability at higher temperature due to removal of the layers susceptible at lower temperatures.

3.4. The void content and the fractured failure surface of composites

Fig 4 shows the void content of 3%_wt NaOH treated flax fibre composites with the fibre volume fraction (V_f) of 30% manufactured at 200°C for 30, 60 and 90 minutes of consolidation time in an isothermal vacuum forming process [error bars are 95% confidence limit]. The consolidation time affects the void content in a similar way to that of the untreated composite [14]. The void content of 0.96% is achieved with 90 minutes of consolidation time.

The tensile fractured surfaces of 3%_wt NaOH treated fibre composites, manufactured at 200°C for 30 and 90 minutes, are shown in Fig. 5. Voidage presence in Fig. 5(a) reveals the poor consolidation of the composites due to insufficient consolidation time. The fibres were easily pulled out from their bundle and the matrix, leaving gaps and holes on their fractured surfaces. By contrast, the fractured surface of the composite, manufactured with 90 minutes consolidation time, shown in the micrographs of Fig. 5(b). It can be seen that the improved bonding is due to the enhanced surface roughness of the fibres as a result of treatment. The contribution of high fibre surface roughness resulted in good mechanical interlocking mechanism of the fibres and the matrix.

4. Summary

The treatment with a higher concentration of NaOH solution results in a more obviously rough fibre surface.

The treatments reduce the hemicellulose and pectin from the fibre surface following treatment.

The loss of mass belonging to hemicellulose and pectin in treated fibres results in a shift of the main degradation temperature to higher temperature.

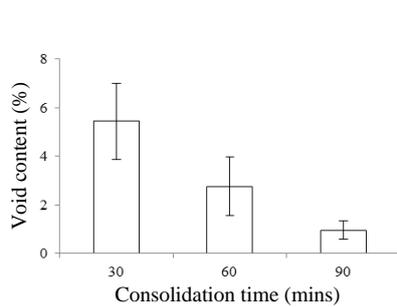


Figure 4. Void content of treated fibre composites (V_f % of 30%) [error bars are 95% confidence limit].

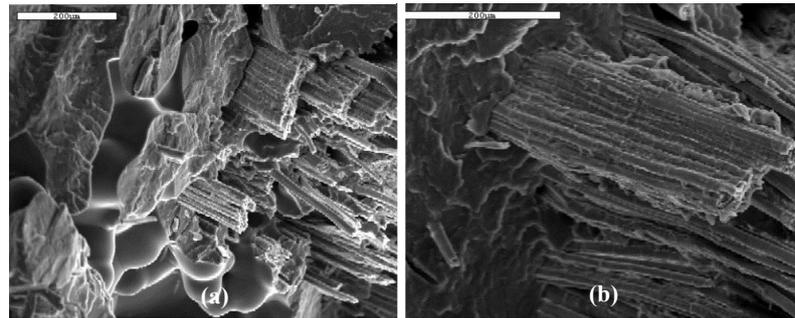


Figure 5. SEM images of tensile fractured surfaces of 3%_{wt} NaOH treated flax/PP composite, manufactured at 200°C (a) a consolidation time of 30 minutes (b) a consolidation time of 90 minutes.

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