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Thermal and physicochemical properties of sugar palm fibre treated with borax

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Abstract. Natural fibres are biodegradable, low-cost, and low-density materials found in many parts of the world in large quantities. The comparative properties and competitive advantages make it be a preferred material in many engineering applications. The major challenges associated with natural fibres are its low thermal stability, lack of interfacial adhesion, poor resistance to moisture and inferior mechanical properties when compared with synthetic fibres. The solution is to treat it with either alkali, silane, or other treatment methods. Specifically, alkaline treatment comes with an additional problem such as fibre degradation. Therefore, this work employed the use of borax as a treatment chemical rather than its usage as a fire retardant. This could give a treated natural fibre with mild degradation and acceptable properties. Therefore, in this research sugar palm fibre was treated with 5 w/w% borax, washed several times and dried in an oven. The treated and untreated fibres were characterized based on X-Ray Diffraction, thermal stability, and morphological analysis. The experimental results showed that the crystallinity index increased from 44.02 % to 49.56 %, while the thermal stability increased from 246.76 °C to 255.5 °C with borax treatment. The morphological investigation revealed that the surface of the treated fibre is rough with no silica deposit when compared with the untreated fibre. In conclusion, the investigation proved that treatment with the borax has a significant effect on the physicochemical properties of sugar palm fibre.

1. Introduction

The increase in stringent regulation on the level of carbon footprint and other environmental concerns like global warming, disposal of components at the end of their useful life, necessitates the use of environmentally friendly materials in industries. In the recent years, industries like furniture, automobile, packaging, aerospace, building construction are all in search of new materials that will replace the conventional glass fibre and other synthetic fibres through research and development. The most promising and adopted materials nowadays are natural fibre reinforced polymer composites, they are evolving as the realistic alternatives to glass reinforced polymer composites in many applications since the 1990s [1]. These natural fibres like sisal, hemp, kenaf, jute, coir and sugar palm fibre among others offer numerous advantages when compared with synthetic fibres. Some of these advantages are light in weight, biodegradability, non-toxic, low-cost, non-abrasive, availability, less pollutant emission and low environmental impact [1, 2]. Unlike natural fibre, glass fibres can cause acute irritation to the skin, eyes, and upper respiratory tract. The fibrous glass and other synthetic vitreous fibres, when disturbed, release fibres that can become airborne, inhaled and retained in the respiratory tract [3].

Just like other natural fibres, sugar palm fibre that is abundantly found in South East Asia is of research interest in the field of material science. The sugar palm fibre is obtained from sugar palm tree, mostly from its four parts, namely trunk, frond, bunch, and trunk's surface [4]. It has been characterized in terms of physical, mechanical, chemical, thermal, and its morphology in previous research [5-8]. A research was done by Ticoalu, et al. [6] and revealed that sugar palm fibre diameter is within the range of 81 to 313 μm. This shows that the diameter of sugar palm fibre is within the range of natural fibres as compared with sisal 100 to 500 μm [9] and kenaf 40 to 200 μm [10]. In terms of moisture absorption, the bagasse, coir, sisal, and jute have a moisture absorption of 8.8, 10, 11, and 12% respectively [11]. This is much high than sugar palm fibre with 5.36 to 8.7% as reported in the literature by Ishak, et al. [8]. In this case, natural fibres are the obvious choice to substitute glass fibre which will normally improve the environmental performance of the components.

Despite the comparative properties of sugar palm fibre with other natural fibres, this fibre is also having some drawbacks. These drawbacks include but not limited to interfacial adhesion, low thermal stability, low durability, quality variation and poor resistance to environment among others [12]. One of the drawbacks of the natural fibres is moisture absorption, sugar palm fibre is not an exception. Moisture absorption test on sugar palm fibre reinforced epoxy conducted by Leman, et al. [13]. The findings showed that composite plate with 20% fibre loading has the highest moisture content (0.93%) prior to the absorption test. It is shown that a sample with high fibre content possesses the highest rate of moisture absorption. The moisture absorption of a fibre makes it degrade faster and will result in poor adhesion between fibre-matrix interfaces, this phenomenon is as a result of hydroxyl group presents in the fibre. Steps were taken by researchers to treat the fibre surface either by chemical, biological or plasma treatments. This problem was looked into by Bachtiar, et al. [14], in order to reduce the level of water uptake, an alkaline treatment was carried out to the fibre prior to the composite fabrication. The results showed an improvement in both strength and modulus after treatment with NaOH, but the effect is more pronounced for the modulus. These researchers obtained inconsistent results at high concentration and long soaking time and this was presumed to be attributed to the damage of the fibre. In another effort to reduce the moisture absorption, a cost-effective and environmentally friendly method was adopted by Leman, et al. [3]. In this research, the fibres were treated with a seawater and fresh water. The tensile results indicated that the fibre treated with seawater for 30 days had the highest stress value of 23.04 MPa i.e. improvement by 67.26% when compared with untreated fibre. While an improvement of 54.37% was recorded for fresh water treatment. Another experiment by Ishak, et al. [15], shows that with a 30 days seawater treatment the outer layer of hemicellulose and pectin were removed thereby improving interfacial bonding between fibre and matrix. In their results, both impact and flexural results had a higher value than untreated sugar palm fibre.

In this research, the sugar palm fibre was treated with borax. The treated and untreated fibres were characterized in terms of thermal stability (TG), crystallinity (XRD), physical (density and moisture absorption), and morphological analysis. This study was conducted to study the effect of the borax treatments for effective utilisation of this fibre in many fields.

2. Experimental Procedure

2.1. Materials
The sugar palm fibre used in this research was obtained from sugar palm tree in Jempol, Negeri Sembilan, Malaysia. It was collected from the surface of the trunk and it has a cellulose and hemicellulose contents of 43.87% and 5.57% respectively [16]. The treatment chemical used in this research is di-sodium tetraborate ($Na_2B_4O_7.10H_2O$) obtained from R & M chemicals Malaysia.

2.2. Chemical Treatment
The treatment was carried out at room temperature with an aqueous solution of di-sodium tetraborate at 5 % (w/v) [17]. The fibre was soaked for 24 hours and was thoroughly washed to remove excess

borax. It was later dried in an oven at 60 °C for 24 hours and kept in a zip bag to avoid moisture ingress.

2.3. Density Measurement
Low density is one of the important properties of natural fibres that make them more attractive to substitute synthetic fibres. To verify the effect of the borax treatment on sugar palm fibre, helium pycnometer was used for the density measurement. This test was done in accordance with ASTM D3800M-10 [18] which is similar except that helium gas is used in placed of liquid. The measurement was done at room temperature and was repeated 10 times to ensure accurate results. To obtain the final value of the density, the weight of the fibre was measured with Mettler Toledo weighing balance while the volume was measured by the pycnometer.

2.4. XRD
The XRD analysis help to obtain important parameters that will verify the effectiveness of the borax treatment on sugar palm fibre. Most importantly the amorphous and crystalline nature of treated and untreated fibre can be determined through this analysis. This analysis was done in the XRD laboratory at the faculty of science, University Putra Malaysia. The machine used is PANalytical (Philips) X'Pert Pro PW3050/60 with scanning done at angle 2θ within the range of 5° to 40°. The tube was operated at 40kV and 20mA with Cu Kα X-ray source.

2.5. Moisture Absorption
To determine the moisture absorption of the samples, both untreated and borax treated sugar palm were allowed to absorbed moisture in humidity chamber as described by Ridzuan, et al. [19]. The humidity chamber was set at 70% and 90% relative humidity at a temperature of 23 °C. The samples were left inside the chamber for a period of 8 days for each of the conditions. Prior to the moisture absorption test, the samples were dried at 60 °C for 24 hours. The weight of the samples were measured before and after exposure to the humidity chamber using Mettler Toledo analytical weighing balance. While the moisture absorbed by the samples was computed using equation 1 as follows:

$$Moisture\ Absorption = \frac{W_{wf} - W_{df}}{W_{df}} \times 100 \qquad (1)$$

Where W_{mf} is the weight of the moist fibre and W_{df} is the weight of the dry fibre

2.6. Morphological Analysis
Micrographs of the untreated and borax treated fibres were obtained in a scanning electron microscope SEM (S-3400N SEM HITACHI). The longitudinal image of the fibre was captured at 1000 X magnification. The aim of the analysis was to examine the surface of the fibre for a better understanding of the effectiveness of the borax treatment on the sugar palm fibre.

2.7. Thermal Analysis
TGA analysis of the untreated and borax treated sugar palm fibre were performed to study the degradation behaviour of the sugar palm fibre with an increase in temperature. Thermo-Gravimetric Analyser (TGA/DSC 1 Mettler Toledo) was used to obtain the TG curves for both treated and control sugar palm fibre. The analysis was done at a heating rate of 10 K/min under nitrogen atmosphere.

3. Result and Discussion

3.1. Density
The densities of the untreated and borax treated fibre were determined with the aid of helium pycnometer. The values were found to be 1.3304 g/cm^3 and 1.3220 g/cm^3 for untreated and borax treated sugar palm fibre respectively. It can be seen from the values that the borax treatment has a slight effect on the density of the treated sugar palm fibre. The density of treated fibre (1.3220 g/cm^3) is slightly lower than the density of the untreated sugar palm fibre (1.3304 g/cm^3). This density of

sugar palm fibre is similar and in some cases lower than other natural fibres like coir 1.25 g/cm^3, cotton 1.51 g/cm^3, flax 1.4 g/cm^3, hemp 1.48 g/cm^3, jute 1.46 g/cm^3, ramie 1.5 g/cm^3, and sisal 1.33 g/cm^3 [20]. This change in density can be associated with the partial removal of hemicellulose and lignin from the fibre. A similar reduction in density was reported by Birnin-Yauri, et al. [21] due to borax treatment.

3.2. XRD

The results of the XRD analysis are shown in figure 1(a) and 1(b) for untreated and borax treated sugar palm fibre respectively. Both XRD of the untreated and borax treated patterns appeared with four (4) major peaks as expected. These peaks were at 15.4°, 16.2°, 22.5, and 34.6° 2θ, they correspond to Miller indices 1-10, 110, 200, and 004 respectively. These patterns are associated to cellulose found in natural fibres [22]. The crystallinity index of both treated and the treated fibres was computed using empirical formula proposed by Segal, et al. [23] given in equation 2.

$$CrI = (I_{200} - I_{am}) / I_{200} \times 100 \tag{2}$$

Based on the formula, the untreated sugar palm fibre has a crystallinity index of 44.02%, while the borax treated fibre has an index of 49.56%. It's clear that the treatment had improved the crystallinity index which is linked to improved mechanical properties. This index is low compared to other natural fibres like flax (72%) [24] and hemp (66%) [25], but it is still within the range of natural fibres.

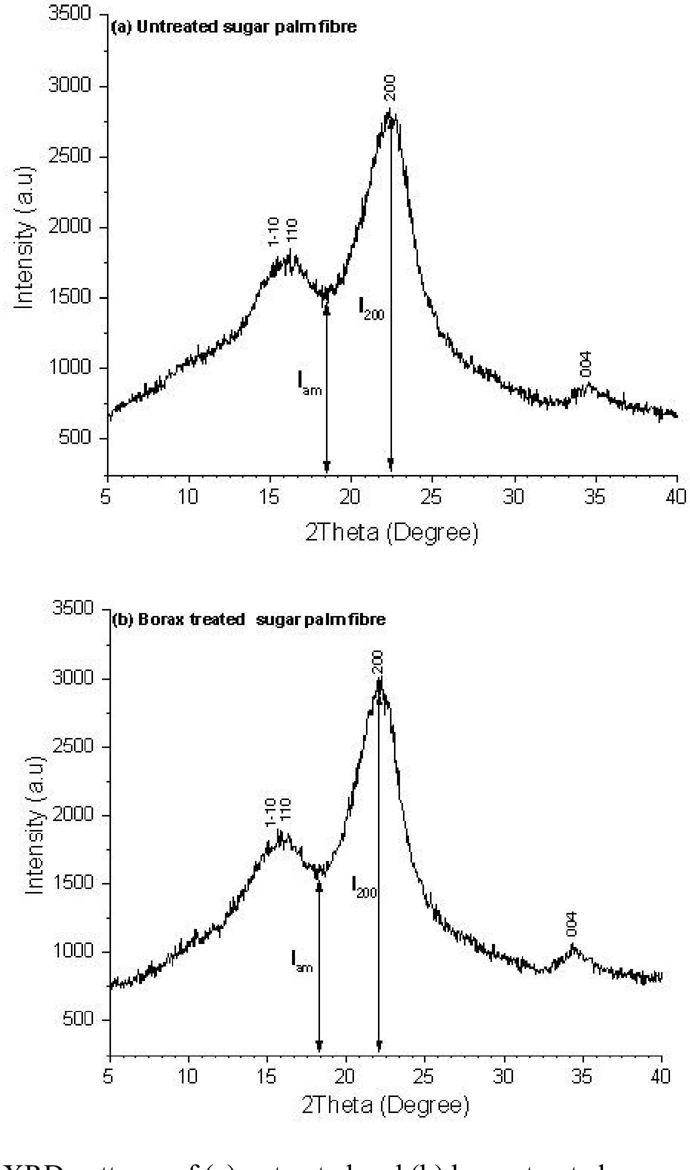


Figure 1. XRD patterns of (a) untreated and (b) borax treated sugar palm fibre.

3.3. Moisture Absorption

The moisture uptake of the untreated and borax treated sugar palm fibre when exposed to humidity at 70% and 90% RH is shown in figure 2. The moisture absorption test was conducted for a period of 8 days, i.e. not up to equilibrium stage. The untreated sugar palm fibre showed high moisture sensitivity when compared with modified sugar palm fibre with borax. This means that the hydrophilic nature of the sugar palm fibre was decreased with the treatment. The results showed that at 70% relative humidity, the untreated fibre absorbed 1.15% of moisture as compared to 0.86% for the borax treated sugar palm fibre. Furthermore, the moisture absorption rate increased with the increase in the percentage of the relative humidity. This result was in agreement with the previous study on moisture absorption for alkaline treated abaca fibre [26]. The hydroxyl group in the fibre which is responsible for the moisture absorption had been partially removed by treating the fibre with borax. This analysis has further confirmed the effectiveness of this treatment as found in the earlier XRD analysis.

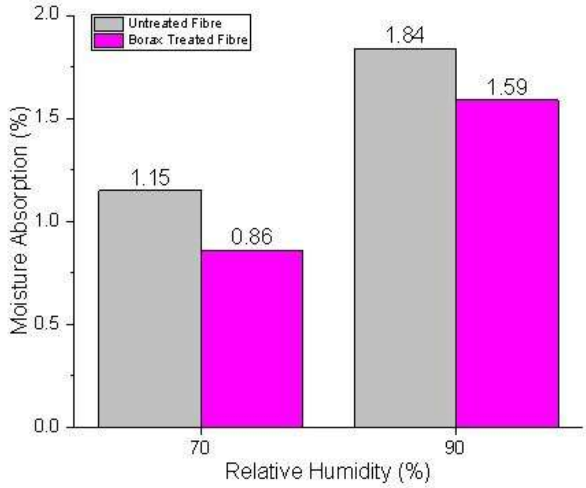
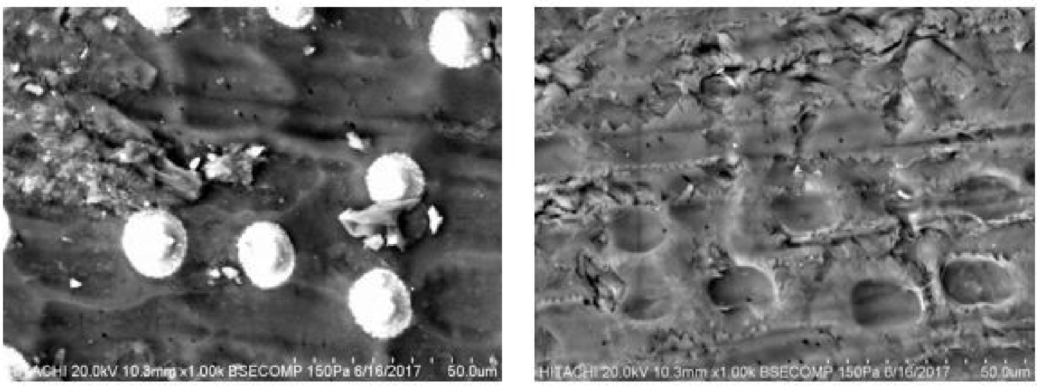


Figure 2. Moisture absorption of the untreated and borax treated sugar palm fibre.

3.4. Morphological Analysis

Untreated and borax treated sugar palm fibres were analysed under scanning electron microscope SEM in order to reveal the changes undergone by the surfaces due to the borax treatment. The morphological surfaces are shown in figure 3(a) and (b) with clear differences between the two samples. The borax treated sugar palm fibre is comparatively rough with clear pits due to the removal of silica deposit as seen in figure 3(b). There are many silica deposits and hemicellulose on the surface of the untreated which seriously affect the compatibility of the natural fibres with the matrix. But with this treatment, improved compatibility is guaranteed.



(a) Untreated sugar palm fibre **(b) Borax treated sugar palm fibre**

Figure 3. SEM images of (a) untreated and (b) borax treated sugar palm fibre.

3.5. Thermal analysis
The curves of weight loss (TG) and its derivatives (DTG) for untreated and borax treated sugar palm fibres are shown in figure 4 and 5 respectively. The curves are characterized with three (3) main stages as shown in the figures. In all the samples, these stages are associated with water loss, degradation of hemicellulose and cellulose, and lastly degradation of lignin. The untreated fibre has a thermal stability of 246.76 °C which is lower than the borax treated fibre with 255.5 °C as its thermal stability. This showed that the thermal stability increased with this treatment. The residue left for treated fibre was high at 31.90% as compared with untreated which is 28.08 %. This further confirmed the high thermal stability of borax treated sugar palm fibre.

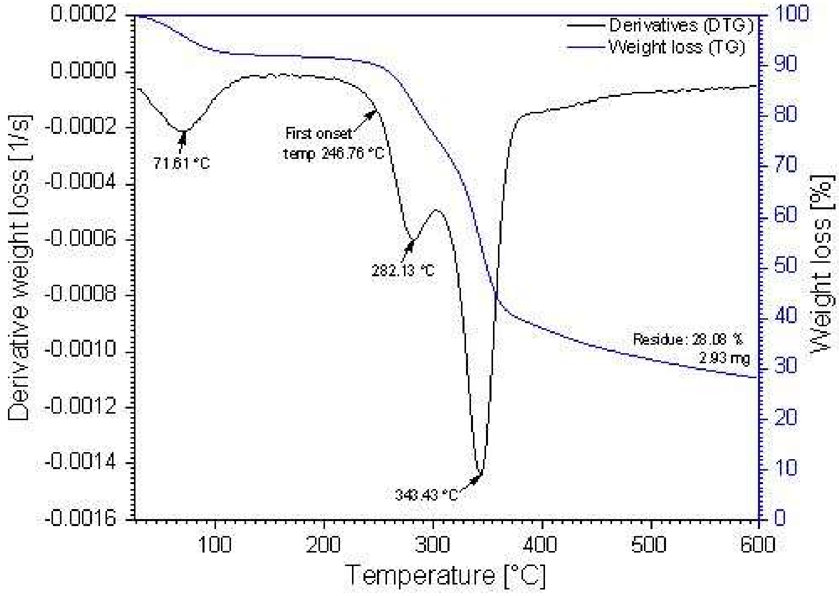


Figure 4. Thermogravimetric and its derivative curve for untreated sugar palm fibre.

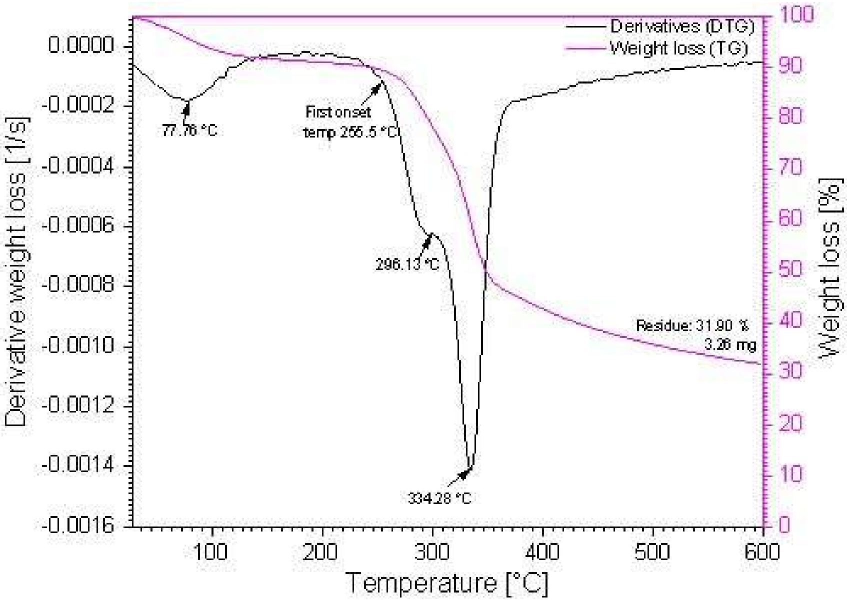


Figure 5. Thermogravimetric and its derivative curve for borax treated sugar palm fibre.

4. Conclusion
In this research, density, XRD, moisture absorption, morphological and thermal analysis were used to characterize untreated and borax treated sugar palm fibre. The sugar palm fibre was treated with 5% (w/v) of borax for the purpose of improving the adhesiveness between the fibre and the matrix. With this modification, the crystallinity index was increased from 44.02% to 49.56% and also reduced moisture absorption was noticed when compared with the untreated sugar palm fibre. The borax

treatment had proved that a thermally stable lignocellulosic sugar palm fibre with an onset temperature of 255.5 °C can be obtained. As clearly revealed by SEM analysis the silica deposit had been removed leaving only the pits which are term as the roughness of the fibre. It is concluded in this research that borax can effectively treat sugar palm fibre and other natural fibres not necessarily as fire retardant additives as shown in many research.

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