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Morphological and Thermal Analysis of Retted Rattan Waste Fibers

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ABSTRACT. The aim of this paper is to study the effects of retting processes on rattan waste fibers, specifically on its morphological and thermal properties. The fibers were prepared for water and chemical retting processes, and were labelled as RF, WRF and CRF for untreated rattan, water-retted rattan and chemical-retted rattan, respectively. Then, these fibers were mixed and hot-pressed into composite boards, which were analyzed for their thermal, strength and morphology properties. Comparatively from Thermogravimetric Analysis (TGA), the WRF has higher thermal stability at lower decomposition temperature, yet the CRF has slightly lower thermal stability compared to RF. It also shows that the retting processes have reduced moisture content inside rattan waste fibers. All the fibers underwent an endothermic process in Differential Scanning Calorimetry (DSC) analysis, where it also displayed that the melting point of RWF, WRF and CRF were 177.80 °C, 183.43 °C, 187.27 °C, respectively. Tensile tests showed that the water retting process had improved the strength properties of WRF/PLA composite board compared other boards. It is supported through the morphological structures of boards. The RF/PLA and WRF/PLA displayed smooth board surfaces indicating the better bonding between fiber and matrix. The removal of certain components such as hemicellulose, lignin and other impurities, obviously can be seen from the morphology of WRF and CRF. It resulted in coarser and rougher fiber surfaces that facilitated good bonding between fibers and matrix as well as reasonable thermal stability. Thus, it is recommended to apply the water retting process before proceeding with fabrication of composite boards.

Keywords: *Retting, Rattan fibers, Morphology, Thermal;*

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1. INTRODUCTION

High demand of green products has motivated researchers to actively explore the potential of utilizing natural fibers. Natural fibers have good properties of being nontoxic, biodegradable, lightweight, superior performance with the addition of easy availability and is cheaper [1]. Thus, it is believed that there is vital need to exploit the agricultural wastes of sisal, banana, kenaf and many more to produce value-added composites for the construction and automotive industries. The exploration of rattan waste usage as filler or reinforcement in bio-composites has not been extensively studied yet. The structure of rattan fibers comprises of cellulose, hemicelluloses and lignin; featuring good properties such as low density, renewability, flexibility, versatility and low cost [2], to be upright substitution as a reinforcing agent in natural fiber composites. The notable drawback of natural fibers is incompatibility between hydrophilic fibers and hydrophobic matrices in the composite world [3], due to impurities in fibers which can result in poor interfacial bonding adhesion of composite, and poor resistance to moisture absorption. This has limited their potential and wide-ranging use. It is important to comprehend that this interfacial bonding plays a vital role in determining the mechanical and physical properties of composites.

Based on these factors, the performance of produced composites can be improved by surface modification of fibers, specifically through the retting process. The retting process dissolves or washes out the impurities of cellular tissues on fiber bundle surfaces such as pectin, lignin, cellulose and wax, by employing water or chemical action [1,4]. Water retting process commonly performed in aqueous environment, where the fibers are soaked in water for a certain period in between 7 to 14 days [5]. The soluble substances come out from the plants and promote the growth of microorganisms that produced specific enzymes as retting agent which eventually degrade the complex structure of fiber to simpler compound. This process generates more uniform and high-quality fibers, yet it changes the chemical composition and pH of water during retting process. While, chemical retting process are affected by few parameters such as type of alkali, chemical concentrations, temperature, solution acidity (pH) and duration of treatment, for its effectiveness. Previous studies [5-7] stated that chemical retting process is more efficient as it produces smoother fiber surfaces within short duration of retting that also improved the mechanical properties of fibers, compared to other retting processes. However, this process has changed the retted fibers into darker color and it consumes high cost of processing. Both these processes improve the stiffness, strength and dynamic flexural moduli of composites, resulting in an increase in interfacial bond strength and adhesion between matrix and fibers. The factors considered for the retting process amongst others are cost-effectiveness, efficiency, labor-friendly, environment-friendly, low cost, simple and easy handling, requiring less skill [5]. Hence, the objective of this paper is to study the effects of water and chemical retting process onto rattan fibers, in terms of its morphology and thermal analysis.

2. MATERIALS AND METHODS

The rattan fibers were sieved and oven-dried for 24 hours. The prepared fibers underwent the water retting and chemical retting process [5]. For water retting process, the rattan fibers were immersed in a beaker with tap water for 8 days at the room temperature, before being taken out. Meanwhile, for chemical treatment, the rattan fibers were placed in a continuously stirred beaker with 1% solution of sodium hydroxide (NaOH), at 25 ºC for 60 minutes. Then, the fibers were washed several times with tap water. The fibers used in this study were oven-dried at 35 °C for 48 hours and were labelled as RF, WRF and CRF for untreated rattan, water-retted rattan and chemical-retted rattan, respectively.

After that, the composite boards were manufactured by using an internal mixer (Haake Polylab System Thermo) machine at fiber:matrix ratio of 30:70. The parameters used were heating temperature of 165 ºC, rotor speed of 50 rpm and mixing time of 12 minute, to produce the compound materials which were labelled as RF/PLA, WRF/PLA, CRF/PLA. These compound materials were crushed and used in hot-pressing process at pressing parameters of 160 °C, 3 min and 147.5 kPa.

2.1 Thermogravimetric Analysis test. The thermogravimetric analysis (TGA) was conducted using a Perkin Elmer Thermogravimetric analyzer. The fiber samples were prepared in powder form, placed in a sample pan and inserted into the thermogravimetric furnace, which was heated at a heating rate of 20 °C min^{-1} from room temperature up to a final temperature of 800 °C, under nitrogen flow.

2.2 Differential Scanning Calorimetry test. The differential scanning calorimetry (DSC) analysis was performed using a TA Instruments DSC analyzer. The fiber samples were placed into an aluminum pan covered with a lid and the sample cell was heated under a nitrogen purge at a heating rate of 10 °C min^{-1}, from room temperature up to a final temperature of 500 °C.

2.3 Tensile Strength test. The samples were prepared for tensile strength test according to ASTM D 638, using the Universal Machine (Autograph AGS-X), at loading load of 10 kN and loading speed of 50 mm/min.

2.4 Morphology test. The morphological structures of rattan fibers and composite boards was investigated using a scanning electron microscope (SEM) model Hitachi SU1510, operated at 15 kV at 250x and 1000x magnification, respectively. The samples were coated with a thin layer of gold to avoid the samples' charging as these samples were non-conductive.

3. RESULTS AND DISCUSSION

The thermal analysis, tensile strength and morphology tests are significantly important to determine the necessity of doing retting process on rattan fibers, before proceeding into further steps to produce composite boards in larger scale. The analysis was done to see if the retting process could make any noteworthy differences on the boards' properties.

3.1 Thermogravimetric Analysis. The differences of TGA curves for RF, WRF and CRF were demonstrated in Fig. 1, to observe specific process of weight loss of weight loss of rattan fibers. The first derivative peak of these fibers occurred in the same range of temperature between 50 ºC to 135 °C due to the reduction of moisture content in rattan fibers. The retted fibers which were WRF and CRF retained the same weight loss of 5%, while RF experienced higher weight loss of 8%. Thus, sit showed that the retting process had reduced the moisture content inside rattan fibers [8]. The second derivative peak fell in the temperature range of 135 ºC to 257 °C, attributable to thermal degradation of cellulose and hemicellulose [9]. The RF has 62.5% of weight loss ratio, while WRF and CRF had weight loss ratio of 61.9% and 65.4%, respectively. The WRF had a higher decomposition temperature of 278.45 ºC indicating that the water retting process had improved the thermal stability of the rattan fibers. However, the low weight loss of WRF indicated a low degradation of cellulose and hemicellulose. Based on this, it shows that the water retting process had improved the thermal stability of the rattan fibers at a lower decomposition temperature compared to RF. At the same time, the low thermal stability after the chemical retting process was due to degradation of the cell walls of fibers which may reduce the thermal protection of the treated fibers. Moreover, the exposure of cellulose to direct heat from the cell walls and hemicelluloses may contribute to this lack in thermal stability [10].

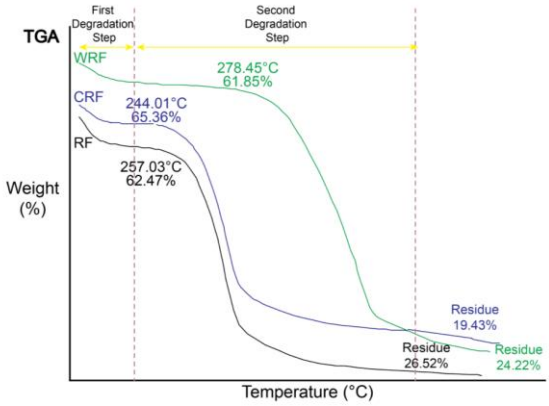

Fig. 1 TGA graphs of rattan fibers

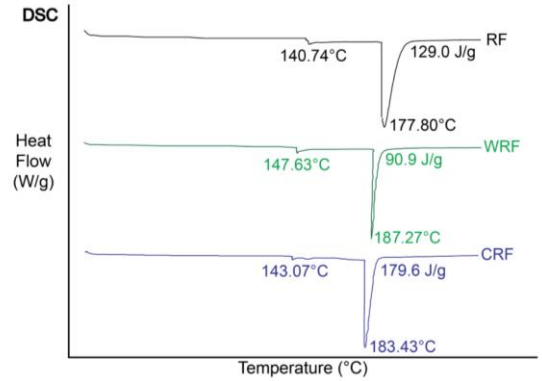

Fig. 2 DSC graphs of rattan fibers

3.2 Differential Scanning Calorimetry. The DSC curves of RF, WRF and CRF are illustrated in Fig. 2, to identify the chemical activity when heat is applied to rattan fibers. The first peak indicates an endothermic process where heat was absorbed by rattan fiber sample due to heat flow needed to reach equivalent temperature of the reference pan. The second peak shows the melting point at which it changes from solid state into liquid state at atmospheric temperature. Based on the curves, the melting point of RF is at 177.80 °C, with energy required of 129.0 J/g. Meanwhile, the melting point of WRF is 187.27 ºC and temperature differences of 9.47 °C from RF. The energy needs to melt for WRF is 90.9 J/g, which is much lower as it involved the biochemical reaction through specific enzymes that are produced as retting agent to decompose. The melting point of CRF is increased to 183.43 ºC with temperature differences of 5.63 °C from RF. In contradict, the CRF needs high energy of 179.6 J/g compared to RF and WRF, as alkaline treatment resulted in increase of cellulose crystallization.

The temperature difference between WRF and CRF is 3.84 °C, indicating an increase of 2.09%, due to higher content of moisture after water retting [8]. The retting processes have improved the thermal stability of the rattan fibers [1], and this data is important for subsequent incorporation into various purposes [10].

3.3 Tensile Strength. Fig. 3 shows the tensile behaviors and Fig. 4 demonstrated the morphological of failure structure of composite boards, which are the RWF/PLA, WRWF/PLA and CRWF/PLA. Generally, it is observed that the neat PLA nominated the highest tensile strength of 69.5 MPa. The addition 30% of rattan fibers content has dropped the tensile strength drastically about 53% into 32.5 MPa. This is due to incorporation between rattan fiber and PLA matrix, with some fiber pull out that can be seen in Fig. 4(a). Water retting process shown the positive effect on the tensile strength of WRF/PLA composite with increasing about 28% that give the value of 41.6 MPa. The morphological structure in Fig. 4(b) had supported this increment value, which showed that the small void occurrence as well as improved adhesion of PLA and rattan fibers that leads to better stress transfer. The chemical retting process, however, had caused reduction in tensile strength values to 40.6% compared to RF/PLA, gave the tensile strength value of 19.3 MPa for CRF/PLA composite. The big micro-void presence during the production of CRF/PLA composite, attributed to poor interfacial adhesion of fiber and matrix, as illustrated in Fig. 4(c).

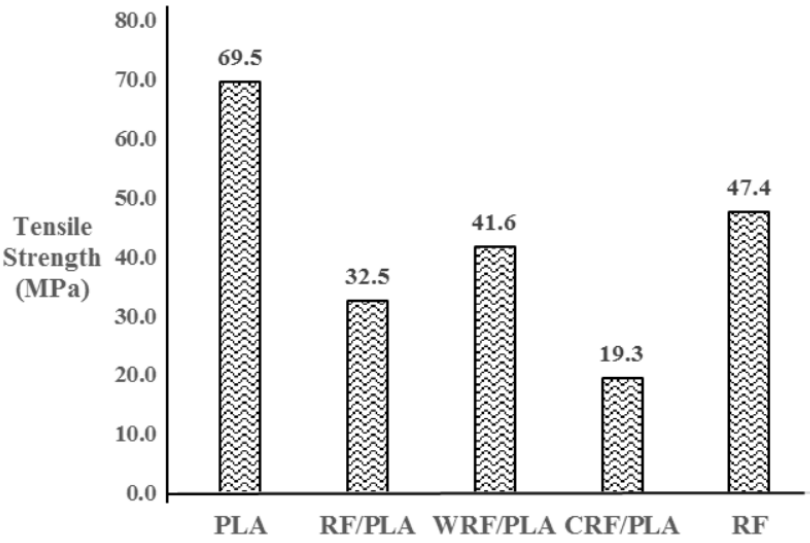

Fig. 3 The graph of tensile strength for PLA, composite boards and rattan fibers

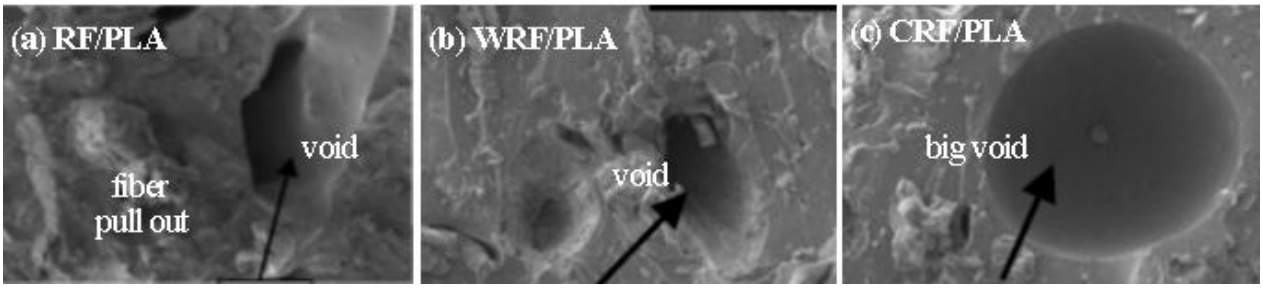

Fig. 4 Morphological of failure structures of the composite boards, (a) RF/PLA, (b) WRF/PLA and (c) CRF/PLA

3.4 Morphological Analysis. Fig. 5 shows the morphological of surface structures of composite boards at 250x magnification, along with the rattan fiber structures at1000x magnification (in inserted pictures) under SEM images. The surface of RF/PLA board in Fig. 5(a) is smooth, with compacted fibers bonding that contributed to better strength and dimensional stability of boards. The fiber surface of RF is rather smooth with the presence of impurities and other elements. Fig. 5(b) shows the fine surface of WRF/PLA board, with the rougher fiber surfaces and cleaner vascular bundles of WRF due to removal of certain components compared to RF/PLA. This revealed the tendency of the water retting process to wash out the impurities and cemented materials such as lignin hemicellulose, lignin, waxes, oils and pectin away from the fibers bundle into more structured fibers bundle with greater uniformity and higher quality [5,7]. Similar observations were found in previous studies [1,4,7,9,11,12]. On the other hand, Fig. 5(c) illustrates coarser surface of CRF/PLA board, with more separated fibers and collapsed cell walls compared to RF/PLA and WRF/PLA boards. The difference is due to the effective degradation of natural and artificial impurities [9,13], hence increasing the aspect ratio and improving the dimension of pore size of rattan fiber, and increasing the amount of exposed cellulose [4,8]. It should be noted that the removal of impurities leads to a less dense and less rigid interfibrillar region which allows the fibrils some rearrangement among themselves. The rough surfaces produce after retting process, has facilitates both mechanical interlocking and bonding reaction due to the disruption of hydrogen bonding in the fiber surface and exposure of the hydroxyl groups to the matrix, thereby leading to better incorporation and interfacial adhesion the fiber–matrix bonding [4,5,7]. It also leads to reduction of fiber diameter that increases the aspect ratio.

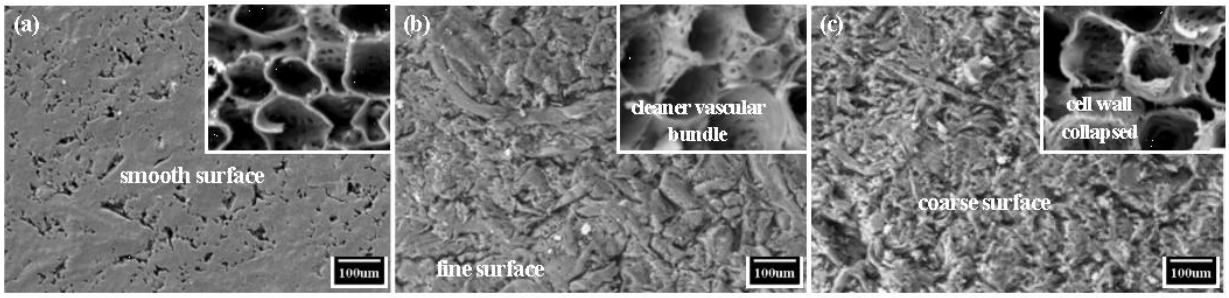

Fig. 5 Morphological structure of surfaces of composite boards, with the fiber structures under 1000x magnification (in inserted pictures). From left: (a) RF/PLA, (b) WRF/PLA, (c) CRF/PLA

Based on both observations in Fig. 5, the structured surface morphology shows that water retting process has improved surface roughness as expected, resulting in better mechanical interlocking as well as increasing the amount of cellulose exposed on,13,14 the fiber surface, thus increasing the number of possible reaction sites during the manufacturing process [8,13,14]. Thus, it resulted in better load stress transfer between the

matrix and the reinforcing fibers during production of the composite [7,11,14], by removing hydrophilic hydroxyl groups and improved moisture resistance property.

4. SUMMARY

Rattan fibers underwent water retting and chemical retting processes; and then were analyzed for thermal analysis, tensile strength properties and morphological structures through TGA, DSC, tensile test and SEM. The thermal analysis, strength values and morphological observation showed that these two retting processes had changed the thermal stability and modify the surfaces of rattan fibers. Coarser and rougher surfaces after the retting process facilitated good interfacial bonding between fibers and matrix, which is vital in producing good quality composite boards with considerable bard strength. In conclusion, the water retting process can be applied on fibers, before proceeding with further steps.

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