

## Acoustical, thermal, and morphological properties of zein reinforced oil palm empty fruit bunch fiber bio-composites

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**ABSTRACT:** In this research, biodegradable composites were prepared with zein as a polymer matrix and oil palm empty fruit bunch (OPEFB) as fiber reinforcement. The fibers were treated with sodium hydroxide (NaOH). The effects of sodium hydroxide treatment on sound absorption, thermal stability, and fiber-polymer matrix interaction in composites were examined. The acoustical sound absorption coefficients of the composites were evaluated using two-microphone transfer function impedance tube method. The spectral, thermal, and morphological studies of the composites were analyzed and characterized using scanning electron microscope (SEM), thermogravimetric analysis (TGA), and Fourier transform infrared (FTIR) spectroscopy. It was found that in all the biodegradable composites, the sound absorption coefficients increased as the frequency increased. Increases in fiber loading caused sound absorption coefficients of the composites to increase. The sodium hydroxide treatment showed a better interface adhesion on fiber and zein matrix. It was also found that this treatment increased the sound absorption coefficients. This was supported by qualitative analysis on the SEM micrographs and FTIR spectrum. © 2016 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2016**, *133*, 44164.

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### INTRODUCTION

Applying sound absorbing materials as acoustic absorbers is a known technique for sound proofing. A wide range of sound absorbing materials exist, their sound absorption properties are dependent upon the frequency, composition, thickness, surface refinement, mounting method, and design. Acoustic panels are usually made from synthetic fibers, which are quite expensive for trivial needs. Besides that, it is hazardous to human health and the environment as well. Thus, some researchers showed keen involvement in putting diligent efforts to create alternative sound absorber from recycled fabrics, such as textile, foam, rubber, or plastic.<sup>1</sup> Commercially available sound absorption materials, which are used for dealing with acoustic sound absorption, are usually made from glass or mineral fiber materials. Wang and Torng<sup>2</sup> had investigated some fibrous porous materials made from rock wool and glass. They showed that the rock wool sound absorption characteristic is comparable to those of glass fiber. However, by considering the health and safety aspect, these types of synthetic fibers can harmfully affected living life and the environment. Thus, this cause an increase in the exploration on the use of organic materials and

opportunity to look for substitute materials made from organic fibers, especially those extracted from agricultural plant waste to be used as sound absorption materials. Organic fiber materials fabricated or used for sound absorber had various benefits, such as renewable, non-abrasive, cheaper, abundance, harmful, and safety. Various researchers<sup>3,4</sup> have succeeded in developing particle composite boards using agricultural wastes. Moreover, Yang *et al.*<sup>5</sup> managed to produce urea formaldehyde reinforced rice straw-wood particle composites as insulation boards. An oil palm agricultural industry generated huge quantities of oil palm biomass or fiber waste such as oil palm frond (OPF), oil palm empty fruit bunch (OPEFB), and oil palm trunk (OPT). The OPT and OPF usually obtained from oil palm agricultural plantation, while the OPEFB obtained from oil palm processing industrial plant.<sup>6</sup> Researchers specified a large amount of oil palm residues harvested plant can be utilized byproducts, and it used to reduce environmental hazards.<sup>7</sup> In examples, OPEFB fibers depicted a great potential in use as a reinforcing material in a polymer matrix. Malaysia and its surrounding South East Asian countries were also known to generate a large amount of OPEFB fiber as waste. In some countries, these wastes were

thrown into the river or open burning, which create air and water pollution that cause a big problem not only to the country but to its surrounding region every year. Nonetheless, these fibers show specific properties that can be used by reinforcing them with polymer matrix to develop bio-composite materials.<sup>8</sup>

Besides, several studies also showed that OPEFB fibers had the potential to be an effective reinforcement in thermoplastic and thermosetting polymers.<sup>9</sup> Over the few past decades, the OPEFB fibers were studied for composite materials manufacturing using different synthetic polymers, for example polyester,<sup>10</sup> polyurethane, polypropylene,<sup>11</sup> phenol formaldehyde,<sup>12</sup> and polyvinylchloride.<sup>13</sup> Basically, in their research works, the polymer resins were used as binder. These researches have focused in their studies on different investigations such as mechanical and physical properties, water absorption, thermal stability, and the effect of surface treatment on the OPEFB fibers as a reinforcing material in a polymer matrix. However, very limited studies have been reported in the literature on the use of OPEFB fiber as reinforcement in natural resins or bio-resins for sound absorption property testing. In order to produce fully renewable and biodegradable composites, both of the polymer matrix and the fiber reinforcement material must be derived from renewable resources.<sup>14</sup> Normally, these are produced by plants that grow during a period less than a year.<sup>14</sup>

Therefore, this work was focused on the property of composites made of zein as the polymer matrix and OPEFB fiber as reinforcement material and water as fluid plasticizer for zein. The primary aims of these studies were to examine the effects of fiber loading and chemical treatment of the natural fiber composites on sound absorption. Thermal and morphological properties of the composites using fibers, with and without chemical treatment had also been investigated. The interactions between fiber and polymer, and the changes in the functional groups, before and after treatment of fibers were analyzed and characterized by using infrared spectral analysis. These properties were studied to evaluate the composites properties as engineering materials.

## EXPERIMENTAL

### Materials

Commercially available zein with the product code of 'Z3625' was purchased from Sigma-Aldrich Corporation, United States was used as a polymer matrix. OPEFB are waste materials that produced in very large quantities by oil palm mills. Oil palm belongs to the species of *Elaeis guineensis* under the family of *Palmaceae*.<sup>15</sup> OPEFB fibers were obtained from Federal Land Consolidation and Rehabilitation Authority (FELCRA) oil palm mill plant located in Kota Samarahan, Sarawak, Malaysia. The fibers have the diameter of 8–300  $\mu\text{m}$ , length 0.89–142 mm, lumen width 8  $\mu\text{m}$ , density 0.7–1.55  $\text{g}/\text{cm}^3$ , and fibril angle 46°. It constituted about 45–50% of cellulose, 25–35% of hemicellulose, and 19–29% of lignin.<sup>16</sup> Sodium hydroxide (NaOH) with product code 'S/4920/API' was supplied by Fisher Scientific, UK. The pellet form of sodium hydroxide is highly soluble in water, highly alkaline, and odorless.

### Methods

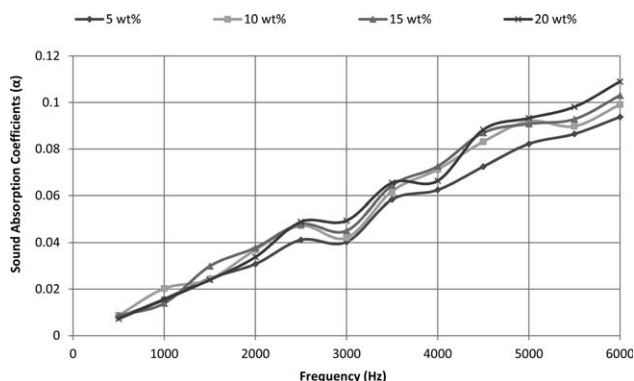
**Specimen Preparation.** The OPEFB fibers were washed with a detergent solution to remove dust and other impurities. It was done at room temperature before being rinsed with water to remove the detergent. The fibers were immersed for 2 hours at room temperature. NaOH solution was prepared by dissolving 5 wt % NaOH pellets in distilled water. After treatment, OPEFB fibers were washed with distilled water until it neutralized to pH 7. The OPEFB fibers were then kept for 5 hours at 80 °C in a hot air oven with natural air convection.

Zein polymer matrix acts as a binder in the composites was reinforced with 5 wt %, 10 wt %, 15 wt %, and 20 wt % of untreated and treated OPEFB fibers. It was prepared by using a hot compression molding technique, which is a common method currently used in the wood based panel industry using 30 Ton Hydraulic Hot Press with the product code of 'LS-22071', Lotus Scientific (M) Sdn. Bhd., Malaysia. Distilled water was added without any chemicals into the mix of zein and OPEFB fibers, which act as a plasticizer that ignites the bonding between zein matrix and OPEFB fibers. The required amount of zein and fibers were measured depending upon the weight ratio of the fiber to zein before evenly sprayed on the mold cavity. Distilled water, equivalent to twice the total weight of fibers and zein, was sprayed into the mixture of fibers and zein. The mold was placed between two plates of compression molding machine at a predetermined temperature of 160 °C and for a particular time of 20 minutes at a pressure of 7 MPa.<sup>17</sup> After compression molding, the press was cooled down with cold running water before removing the composites. The composites obtained were conditioned in accordance to ASTM E41-92<sup>18</sup> standards;  $24 \pm 3$  °C in temperature, 65% in relative humidity, and 101 kPa in pressure for 24 hours before testing.

**Characterizations of the Composites. Sound absorption test.** Sound absorption properties were measured using impedance tube according to ASTM E1050-12<sup>19</sup> standards. These setups were employed in extent for different acoustical parameters measurement in the range of 500 to 6,000 Hz. The method of measurement only required plane wave propagation that/to occur in the tube.

**Thermal stability test.** Thermogravimetric analysis (TGA) was conducted to analyze both spectrums for untreated and treated fiber composites. The TGA was performed on the TA-60 WS workstation analyzer supplied by Shimadzu Corporation, Japan. The specimens were heated at a heating rate of 10 °C/min and examined under flowing nitrogen of 80 mL/min over a temperature range of between 30 °C and 700 °C. The test was done according to ASTM E1868-10.<sup>20</sup>

**Infrared spectral test.** The functional groups of both untreated and treated fibers were understood based on the infrared spectrum obtained from the Fourier transform infrared (FTIR) spectroscopy, in the range of 4,000  $\text{cm}^{-1}$  to 400  $\text{cm}^{-1}$ , by using a FTIR-8101 spectrometer supplied by Shimadzu Corporation, Japan. Sample pellets for FTIR spectroscopy were fabricated with a mixture of small agate pestle of 0.5 mg powdered sample mixed with approximately 100 mg of dried powdered sample bromide. The mixture was taken into a specific die dimensions,



**Figure 1.** Sound absorption coefficients of zein/treated OPEFB fiber composites with various fiber loading.

whereas the pellets were made by applying it using vacuum pressure. All the infrared spectrum information obtained through 'IRsolution' software was generated in transmittance (T %) versus wavenumber ( $\text{cm}^{-1}$ ). The quantitative and qualitative of infrared analysis were done according to ASTM E168-06<sup>21</sup> and ASTM E1252-98<sup>22</sup> standards.

**Morphological test.** The morphological studies of the specimens were observed under TM3030, Hitachi Tabletop Scanning Electron Microscope (SEM) supplied by Hitachi Limited, Japan. It had a field emission gun and an accelerating voltage of 15 kV, to collect images. The test specimens were sliced and mounted on aluminum stubs with double sided adhesive tape and sputter coated with metal (such as gold) for 5 min to a certain thickness approximately 10 nm under 0.1 torr and 18 mA to make the sample conductive. The spurs coated JFC-1600 machine use was equipped by JEOL Limited, Japan. The test preparations were done according to ASTM E2015-04.<sup>23</sup>

## RESULTS AND DISCUSSION

### Sound Absorption of Zein/OPEFB Fiber Composites

Regardless of the fiber loading, both the treated and untreated OPEFB fiber reinforced zein composites demonstrated an increasing trend of sound absorption coefficient with increasing the excitation frequency as shown in Figures 1 and 2. The sensitivity of fiber loading to the sound absorption coefficient of the composites is not noticeable at the excitation frequency below 1,500 Hz, whereas, this effect is comparatively obvious at the frequency above 1,500 Hz. In general, the absorption at low excitation frequency is low due to the long wavelength of sound wave.<sup>24</sup> The increasing trend of the sound absorption coefficient with increasing frequency is consistent with the results observed in other researches of natural fiber reinforced polymer composites with different matrix and fibers.<sup>25–28</sup>

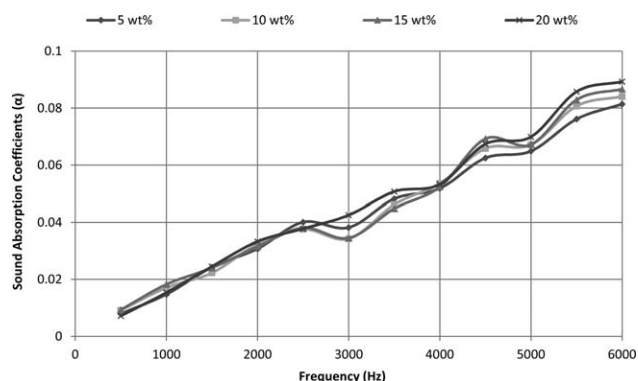
The factor causing the higher sound absorption coefficient at higher OPEFB fiber loading is the larger volume of fiber with unique porous structure, which impedes the vibration of the sound. OPEFB fibers are porous fiber materials, which contain many connected air cavities, and those air cavities might be the major contributors of sound energy absorption. The physical characteristics of the fiber such as the size, shape, and types of pores may influence the effectiveness of the fiber as sound absorbent. The

mechanism by which OPEFB fibers absorb sound energy mainly involves the conversion of kinetic energy of the sound to heat energy when the sound strikes the fibers. Hence, the sound energy will be dissipated after striking the composites via conversion into heat.

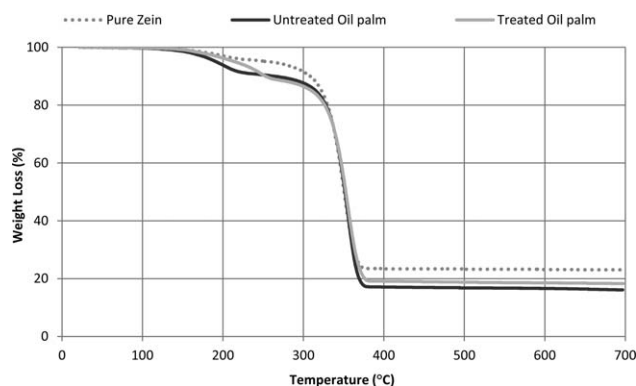
Comparing Figures 1 and 2, it can be observed that the sound absorption coefficients of the treated OPEFB fiber reinforced zein composites are higher than the untreated counterparts. The difference in the sound absorption behavior is attributed to the modification to the fiber physical characteristics after the chemical treatment, thereby altering the effectiveness of the fiber as sound absorbent. The chemical treatment is known to alter the lignin and hemicellulose structure thus produces a more serrated and rough surface.<sup>27</sup> For untreated fiber, lignocellulosic contents such as lignin, pectin, and hemicellulose, and low molecular weight materials can form a dense layer on fiber surface, which caused sound reflection.<sup>29</sup> Thus, alkaline treatments were used to partially remove pectin, hemicellulose, and lignin content from the fibers. This produced more porous structure on the fiber surface, which lowered the reflection. This could attribute to the facts that rough fiber surface area increased the friction between the fibers and sound wave that resulted in high sound absorption. Fatima and Mohanty<sup>30</sup> confirmed that the alkaline treatment enhanced the mechanical and acoustical properties of the fiber composites due to the removal of impurities from the fiber surface and separation of fiber strands. Thus, it is speculated that this increased surface roughness of treated OPEFB fiber has increased its effectiveness for sound absorption.

Another factor which contributed to the improved sound absorption property of the treated OPEFB fiber reinforced zein composites is the reduction of fiber diameter and thereby the increase of the aspect ratio (length/diameter) after chemical treatment. The increase in sound absorption was observed with decrease in fiber diameter. This is due to thinner fibers can oscillate more easily when exposed to sound wave. The interpretation of results presented here is in agreement with Seddeq<sup>26</sup> who showed that fiber size plays a major role on sound absorptive of composite. It is also shown that sound absorption increases as fiber diameter decreases.

Apart from the effect of fiber characteristic, the choice of zein as the matrix polymer to form the composite plays a role in affecting the resultant sound absorption coefficient of the



**Figure 2.** Sound absorption coefficients of zein/untreated OPEFB fiber composites with various fibers loading.

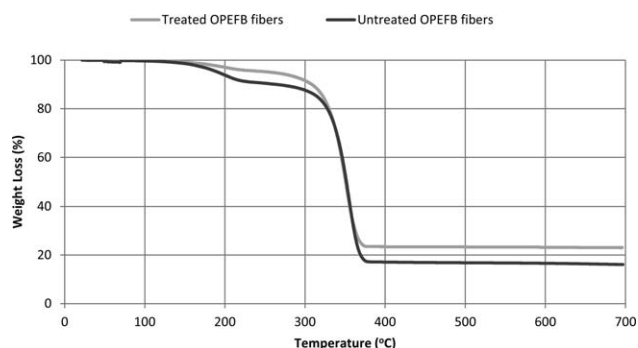


**Figure 3.** Thermal gravimetric curves of zein/treated OPEFB fibers composites, zein/untreated OPEFB fibers composites, and pure zein composites.

composites, due to the void formation during the processing stage, which assisted sound absorption. The fabrication of composite with zein as matrix polymer involves utilizing water as plasticizer. The molding temperature of 160 °C induces cross linkages within the zein protein and also results in the formation voids. It is the vaporization of water at this temperature, which resulted in the formation of voids. It is believed that this microstructure is favorable in improving the sound absorption property. The extent of crosslinkages and voids is dependent on the temperature and processing time of the zein. This suggests that it is feasible to control the processing temperature and time to control the number, size, and type of voids form within the zein matrix polymer, which affects the sound absorption coefficient. Although, the effect of processing temperature and time on the resultant coefficient of sound absorption is outside the scope of current study, it should be mentioned that it can be investigated as an extension of the current work.

#### Thermal Stability of Zein/OPEFB Fiber Composites

Figure 3 showed the thermal degradation of 15 wt % of OPEFB untreated and OPEFB alkaline treated fibers reinforced zein composites were investigated using TGA. Figure 3 showed that the initial weight loss started at around 160 °C due to the volatilization of moisture and other thermally reactive products that present in the fiber composites. The thermal decomposition of the zein/treated OPEFB fiber composites started at comparatively higher temperatures than that of the zein/untreated OPEFB fiber composites. Figure 3 showed that zein/treated OPEFB fiber composites had higher thermal stability than a zein/untreated OPEFB fiber composites. The decomposition started at 180 °C and complete at 700 °C. The fiber composites started losing weight



**Figure 4.** Thermal gravimetric curves of untreated and alkaline treated OPEFB fibers.

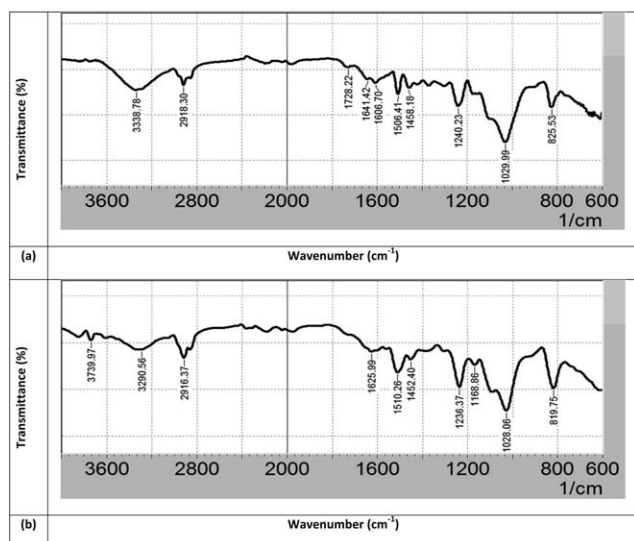
drastically in the range of 260 to 360 °C. As shown in Figure 3, it showed that the composites have two-step degradation. The first step is due to the degradation of the hemicelluloses at 250 °C. Meanwhile, the second step is due to the degradation of lignin and cellulose at 380 °C.<sup>31</sup> Thermal degradation of pure zein showed a one-step process that started at 260 °C and ended at 390 °C. The TGA results confirmed that the sodium hydroxide treatment of natural fibers increases the thermal stability of the composites. A vital aspect that caused the high thermal stability of treated fiber composites is due to the enhancement on the fiber and polymer matrix interactions, which create additional intermolecular bonding between fiber and polymer matrix in the composites.<sup>31</sup> Table I shows the weight loss percentages at different temperatures of pure zein and its composites.

Figure 4 showed thermal gravimetric curves of untreated and treated OPEFB fibers. It is known that thermal degradation of OPEFB fibers involves several weight loss steps. Mostly it is due to decomposition of major lignocellulosic constituents such as hemicelluloses, cellulose, and lignin. The weight loss also occurs due to the removal of moisture that present in fibers. Besides lignocellulosic constituents and percentage of moisture, thermal analysis also depends on the degree of crystallinity and structure of OPEFB fibers. Decomposition of fibers continues by losing its absorbed moisture, followed by hemicelluloses then cellulose and lastly lignin. An untreated and alkaline treated fiber shows similar behaviors with small variations in weight loss percentage.

Initial weight loss 100 °C and below is due to absorbed moisture gradual evaporation. The hydrophilic nature of the natural fibers causes difficulty in elimination of water that presents as structural bound. The second weight loss approximately from 150 to 500 °C is due to decomposition of three major

**Table I.** Weight Loss at Different Temperatures of Pure Zein and Zein/OPEFB Fibers Composites Obtained from TGA with Alkali Treated (T) and Untreated (UT)

Samples	Weight loss (%)									
	100 °C		200 °C		300 °C		400 °C		500 °C	
	UT	T	UT	T	UT	T	UT	T	UT	T
Zein/OPEFB Fiber —Composites	0.8	1.2	2	2.6	4.8	5.8	20.1	21.9	100	100
Pure Zein	0.0		0.0		0.4		6.8		99.8	



**Figure 5.** Fourier transform infrared spectrum of (a) zein/untreated OPEFB fiber composites, and (b) zein/treated OPEFB fiber composites.

lignocellulosic constituents of fibers (lignin, hemicellulose, and cellulose) and it is chemically active. Thermo-chemically hemicelluloses decompose between 150 °C and 350 °C, cellulose between 275 °C and 350 °C, and lignin between 250 °C and 500 °C. The untreated fibers had lowest onset of degradation temperature of 272 °C. Nevertheless, alkaline treated fibers degraded from 310 – 320 °C that is at higher temperatures. From the analysis, it concluded that thermal performances of natural fibers improve by surface treatment. Increase of treated fibers decomposition temperature is due to the removal of smaller molecules (and thus an increase in molecular weight) and amorphous from surface fiber.<sup>32</sup> Furthermore, it is highly recommended that the mode of decomposition and thermal degradation during fabrication of natural fibers reinforced polymer matrix composites under influence of heat for optimization process parameters.<sup>33</sup>

#### Infrared Spectral of OPEFB Fibers and Zein/OPEFB Fiber Composites

Infrared spectrum can be divided into three regions; the far-infrared (<400  $\text{cm}^{-1}$ ), mid-infrared (4,000–400  $\text{cm}^{-1}$ ), and the near-infrared (13,000–4,000  $\text{cm}^{-1}$ ).<sup>34</sup> Figure 5(a,b) shows the FTIR spectra of the zein/untreated OPEFB fiber composites and zein/treated OPEFB fiber composites from 4,000 to 400  $\text{cm}^{-1}$ . The spectra offer qualitative and semi-quantitative data signifying the absence and presence of lignocellulosic compounds, and changes of the absorption band intensities after and before treatment.<sup>35</sup> Hydrogen bonds in the natural fiber network structure were disturbed by the sodium hydroxide treatment, therefore, increasing the surface roughness. The sodium hydroxide treatment removed a certain amount of wax, hemicelluloses, lignin from the fiber cell wall, and depolymerized cellulose, and uncovers the short length cellulose fibril. This was the reason behind the increased sound absorption coefficient for the treated fiber composites. A large band at 3,338.78  $\text{cm}^{-1}$  is mainly related to the hydroxyl groups and the bonded O–H stretching vibrations present in carbohydrate.<sup>36</sup> Alcohol group

assigned at 3,338.78  $\text{cm}^{-1}$  and 1,240.23  $\text{cm}^{-1}$  were reduced mainly due to significant reduction of cellulose and hemicellulose that reacted with sodium hydroxide. It is coherent with Suardana,<sup>37</sup> whereas the peak at 1,240  $\text{cm}^{-1}$  reduced when alkali concentration was increased.

Chemical structural fragments within molecules were known as functional groups. In same wavenumber range, it tends to absorb infrared radiation regardless of the structure of the rest of the molecule that functional group was located.<sup>38</sup> Due to the hemicellulose removal from the natural fiber surface, the carbonyl group stretching at 1,728.22  $\text{cm}^{-1}$  was almost reduced. It was observed that the water molecules from the sodium hydroxide treatment decrease the intensity at 1,606.70  $\text{cm}^{-1}$  due to removal of O–H bonding.<sup>39</sup> A similar peak was also observed in the spectral between the zein/untreated OPEFB fiber composites and zein/treated OPEFB fiber composites that contribute to C–H stretching at 2,918.30  $\text{cm}^{-1}$ . Due to sodium hydroxide, zein/treated OPEFB fiber composites intensity band peak at 1,240.23  $\text{cm}^{-1}$  characterized as the O–H bending plane was reduced due to formation of glycosidic bonding. In general, glycosidic linkages formed cellulose and groups of hydroxyl with a small amount of carboxyl, whilst hemicellulose and lignin are dominated by hemicellulose with ether bonds characterized by a significant amount of carboxyl groups.<sup>40</sup> Absorption band at 1,029.99  $\text{cm}^{-1}$  usually reflects the C–OH stretching vibration of the cellulose that associated with polysaccharide components.<sup>41</sup> The reasons for the higher sound absorption coefficient were due to the reduction of lignocellulose components from the natural fibers.

Figure 6(a,b) showed the FTIR spectra of the untreated and alkaline treated fibers, in the range of 4,000–400  $\text{cm}^{-1}$ . The absorption band at 3,600–3,400  $\text{cm}^{-1}$  may be due to various hydroxyl groups of OH stretching vibration. The reaction had occurred during alkaline treatment between the cellulose and sodium hydroxide that change the degree of cellulose structures and cause swelling. In the cellulose, sodium hydroxide  $\text{Na}^+$  ions had expanded the pores between lattice planes before passing through it. The  $\text{Na}^+$  ions penetration caused the formation of new Na–cellulose–I lattice that creates larger spaces between the cellulose molecules that usually filled up with water. Within new structure, hydroxyl OH– groups of cellulose were converted into O–Na groups temporary as the  $\text{Na}^+$  ions were removed after rinsed with water. The result cause hydrogen bonds of cellulose to be destroyed and released more hydroxyl groups that increased the surface roughness of fibers.<sup>42</sup>

The OH compounds may also include primary aliphatic, secondary alcohols, and absorbed water that found in lignin, hemicellulose, cellulose, extracts, and carboxylic acids in extractives.<sup>43</sup> The absorption band near the OH stretching vibrations, at 2,922.28  $\text{cm}^{-1}$  may relate to CH stretching vibrations. While, the bands at 1,626.06  $\text{cm}^{-1}$  region for cellulose may ascribe to C=O stretching vibration of the alpha-ketone carbonyl.<sup>44</sup> Similar results showed by Sim *et al.*,<sup>45</sup> whereas the changes, reduction or occasional disappearance of typical hemicellulose and lignin in the spectra of fibers was due to alkaline treatment, which is located at the peak of 1,031.00  $\text{cm}^{-1}$ , 1,241.25  $\text{cm}^{-1}$ , 1,368.55  $\text{cm}^{-1}$ , 1,442.82  $\text{cm}^{-1}$ , and 1,478.50  $\text{cm}^{-1}$ . The peak

1,723.47  $\text{cm}^{-1}$  is attributed to acetyl groups of C=O stretching hemicellulose. This peak is not present in the alkali treated fibers. The hemicellulose removal from the surfaces of the fiber causes the peak to disappear. It produced self-arrangeable fibrils in the fiber after alkaline treatment due to the removal of hemicellulose that decreases the rigidity and density of inter-fibril regions.<sup>46</sup> Therefore, insignificant reduction detection band intensity of C=O stretching showed poor modification of hemicellulose. Apart from that, hydroxide ions and sodium cation from NaOH also cause lignin structure to be degraded. These ions catalyzed formation of the six-membered transitions. It was used to cleave the lignin aryl-alkyl bonds.<sup>47</sup> Therefore, after the treatment, the undetected spectra of C—O—C stretching band from alkaline treated fibers evidenced the breakdown of the bonds. Moreover, the sticky materials like pectin and waxes were also removed. It is proven through reduction of the intensities band of C—O—C, O—H, and C—O from treated fiber spectra.<sup>42</sup>

Figure 7 showed the FTIR of pure zein. The FTIR spectrum of zein shows the bands assigned to different C=O stretching vibrations at 1,722.43  $\text{cm}^{-1}$ . The differences are seen in the range 1,039.63  $\text{cm}^{-1}$ –1,259.52  $\text{cm}^{-1}$  that attributed to C—O and C—O—C stretching vibrations. There are bands at 1,379.10  $\text{cm}^{-1}$  and 1,452.40  $\text{cm}^{-1}$  in zein spectra that may attribute to asymmetric and symmetric deformational vibrations of C—H in  $\text{CH}_3$  groups. Other main bands are located at 1,164.71  $\text{cm}^{-1}$  and 1,039.63  $\text{cm}^{-1}$  assigned to C—C chain stretching vibration and CH bending and wagging vibration.<sup>48</sup>

#### Morphological of Zein/OPEFB Fiber Composites

Respectively, Figures 8(a,b) show the interaction between fiber and matrix before and after fiber chemical treatment. Meanwhile, Figure 8(d) shows a large lumen and thin cell walls of OPEFB fibers and Figure 8(c) shows a considerable number of small holes on the cross section of the OPEFB fiber reinforced zein

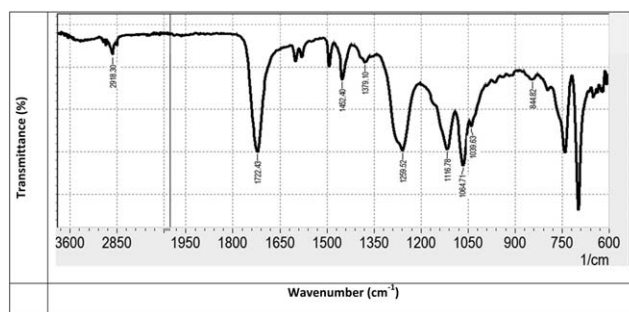


Figure 7. Fourier transform infrared spectrum of pure zein.

composite, which is formed mainly due to the evaporation of water during the composite compression molding process. These small holes could help to trap the sound energy and it is converted into heat and provides better sound absorption to the composites. In fibrous materials, the sound is being absorbed by viscous effect between fiber frame and numerous air cavities that will attenuate part of sound energy and convert it into heat.<sup>49</sup> It can be deduced that the unique lumen structures of OPEFB fibers resulted in lower density and higher porosity of their composites.

The SEM micrograph of both untreated and treated OPEFB fibers were shown in Figure 8(e,f) respectively. From the figures, we can observe the significant changes in the surface morphology as a result of alkaline treatment. Due to the alkaline treatment, the impurities present in the untreated fiber surface were removed and the fiber strands were separated, and the treatment also led to a rougher surface.<sup>37</sup> The untreated fiber surface consists of oil covering and waxy substance called cuticle, which was of aliphatic origin and nonpolar in nature, which rendered it incompatible with the Zein matrix as shown in Figure 8(a).

From Figure 8(b), it can be inferred that fibers were tightly bonded to the Zein matrix, which indicated better interfacial bonding between the fiber and matrix, which was improved by the alkaline treatment of fibers. The important modification achieved with alkali treatment is the disruption of the hydrogen bonding in the network structure, thereby increasing the surface roughness. By observing the microstructures of the OPEFB fibers cross section in Figure 8(d), it can be realized that natural fibers possess a multiscale structure. A single OPEFB fiber is made up of a bundle of hollow sub-fibers. The distributed fibers in the composites create a porous structure that enhanced sound absorption. Therefore, due to chemical modification the acoustical and thermal properties could be strongly influenced.<sup>50</sup> The chemical treatment also disrupted the absorption bands associated with lignocellulosic constituents. The infrared spectra in Figure 6(a,b), of both the untreated and treated OPEFB fibers characterized by FTIR spectroscopy confirmed the effects of chemical reactions on the lignocellulosic constituents (cellulose, hemicellulose, lignin, and pectin) of OPEFB fibers.

#### CONCLUSIONS

Briefly, the sound absorption coefficients of zein/OPEFB fiber composites with various fiber weight percentage (wt %) have been evaluated. The results show that the composites with 20 wt % fiber loading have a higher sound absorption coefficient

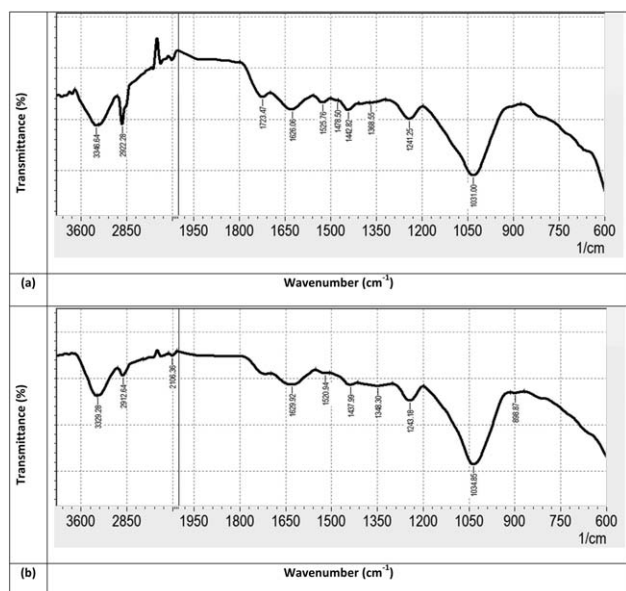
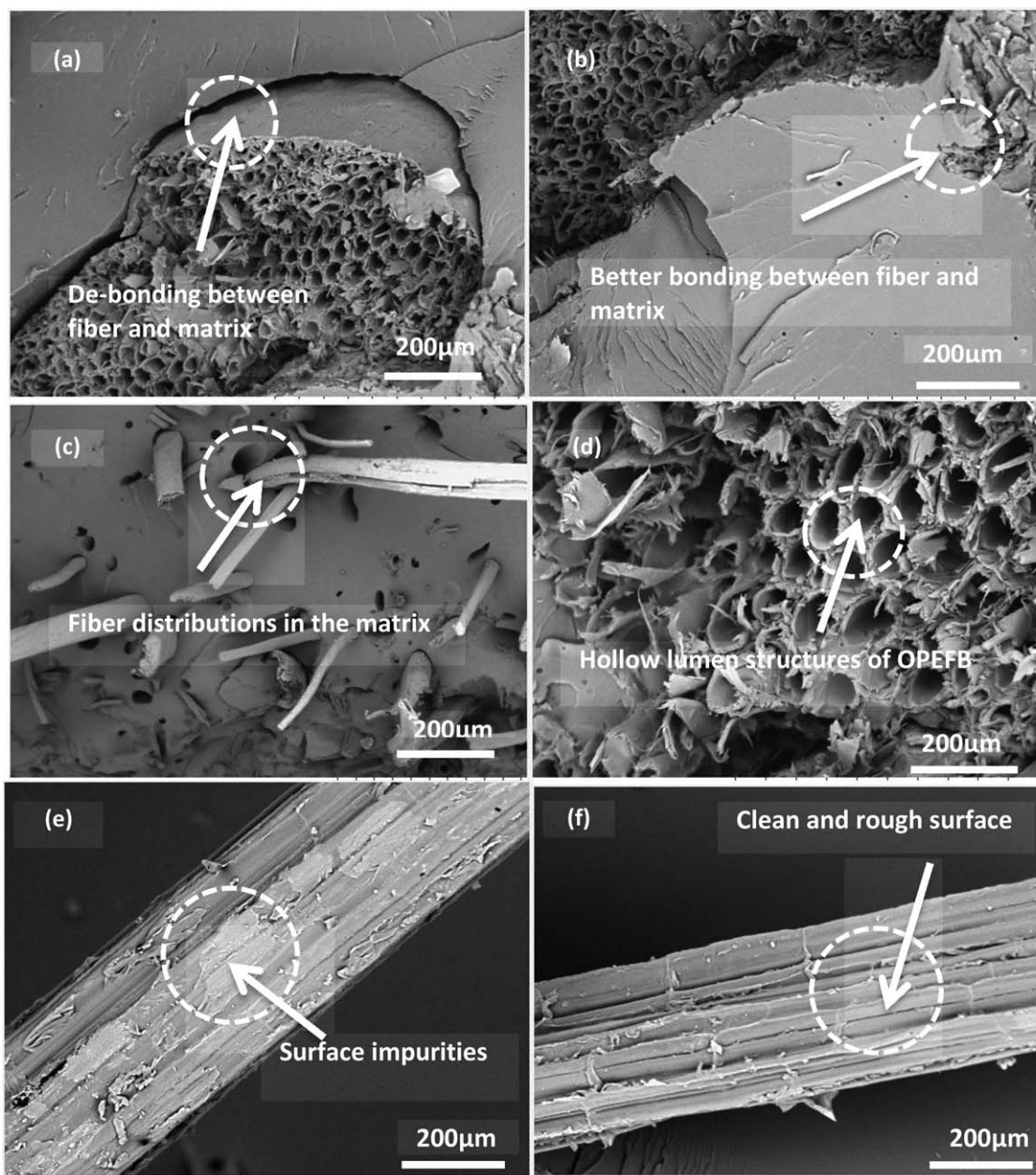


Figure 6. Fourier transform infrared spectrum of (a) untreated OPEFB fiber, and (b) alkaline treated OPEFB fiber.



**Figure 8.** Morphology of (a) zein/untreated OPEFB fiber composites, (b) zein/treated OPEFB fiber composites, (c) cross section of zein/treated OPEFB fiber composites, (d) Hollow lumen structure of OPEFB fiber, (e) Surface of untreated OPEFB fiber, and (f) surface of alkali treated OPEFB fiber.

with exceptions at certain frequencies. In all the zein/untreated OPEFB fiber composites and zein/treated OPEFB fiber composites, the fiber loading at 20 wt % fiber showed higher sound absorption coefficients compared to the composites of 5 wt %, 10 wt %, and 15 wt % fiber loading. Thermal stability of zein/untreated OPEFB fiber composites and zein/treated OPEFB fiber composites was lower than that of pure zein. There was a marginal improvement in the thermal stability of the fibers after alkaline treatment due to the reduction of the hydrophilic nature of OPEFB fibers. The morphological study showed better interfacial adhesion due to the effect of alkaline treatment on the OPEFB fibers. The FTIR analysis showed the

changes in the spectrum and the functional groups, between the untreated and the treated fibers. The properties of the zein/OPEFB fiber composites show that OPEFB could be used as eco-friendly and biodegradable reinforcement for composites. As both the matrix polymer and filler are biodegradable, the zein bio-composites have the advantage of minimizing environmental pollution at its end-of-use in addition to enhanced acoustical and thermal properties. Finally, based upon the results of this study, it is shown that the zein reinforced natural fiber composites are promising alternatives as sustainable, biodegradable, and a renewable sound absorption material.

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