Effect of acetylation modification on the structure and properties of windmill palm fiber

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Abstract

Windmill palm fiber, a high-quality cellulose resource, can be extracted from discarded palm sheath. However, the application of this abundant biomass in composite reinforcement is limited due to its hydrophilicity. In this study, the effects of chemical modifications, such as treatment with alkali, acetyl chloride, and acetic anhydride, on the micro-morphology, water repellence, sound absorption, thermal conductivity, and thermal stability of windmill palm fiber have been investigated. The results showed the surface of windmill palm fiber treated with alkali to be relatively long, thick, and smooth. In comparison, acetylation treatment destroyed the cell walls, leaving micro-holes in the surface. Acetylation modification significantly improved the water repellence of the fibers, increasing the water static contact angle to more than 145\textdegree. Acetylation treatment also improved the sound absorption performance of a fiber mat, giving an average sound absorption coefficient of 0.47, maintained a low thermal conductivity of under 0.050 W/m/K, and improved the thermal stability, raising the initial decomposition temperature to above 350°C.

Keywords

Windmill palm fiber, hydrophobic, acetylation modification, thermal property, surface morphology

Natural fibers from renewable green resources, such as crop straw, vegetables, bamboo, hemp, wood, and so on, are characterized by excellent thermal and mechanical properties, low cost, sustainability, and environmental friendliness.\textsuperscript{1} High cost-effectiveness and environmental protection endow natural fibers with the potential to serve as biodegradable alternatives to synthetic fibers.\textsuperscript{2} Among these green fibers, windmill palm fiber extracted from windmill palm sheath meshes has unrivalled toughness. It offers the highest elongation at break and excellent energy absorption ability,\textsuperscript{3–6} acoustic insulation,\textsuperscript{7,8} as well as ultraviolet resistance.\textsuperscript{9} Currently, the windmill palm tree represents an essential resource for economic and social life in China.

Windmill palm fiber extracted from palm sheath is a cellulose fiber with a thin fiber cell wall and a significant central lumen. The length:diameter ratio is more than 60.\textsuperscript{5} Short cellulose fibers with a large length:diameter ratio have been used as reinforcements in composites to improve their properties, especially mechanical strength.\textsuperscript{10} At the molecular level, cellulose chains bear regular equatorially arranged hydrophilic hydroxy (OH) groups,\textsuperscript{11} leading to poor dispersibility and incompatibility with other nonpolar media and matrices.\textsuperscript{12} The blending of relatively hydrophobic polymers with hydrophilic short cellulose fibers often results in poor interfacial adhesion between the materials, leading to poor mechanical properties.\textsuperscript{10,13}

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Alkali treatment, esterification, and etherification are standard methods for hydrophobic modification of cellulose fibers. Nevertheless, alkali treatment does not improve water resistance because abundant hydroxyl groups render the cellulose fibers hydrophilic. Acetylation modification is a more practical modification method for the surfaces of fibers, whereby acetyl groups (CH₃CO-) react with the surface OH of cellulose, making the surface less hydrophilic. Gan et al. increased the tensile strength by 43.3% and the elongation at break by 12.6% through surface acetylation of a cellulose nanocrystal-reinforced composite. Sofla et al. developed a new method for creating high-quality cellulose nanofibers by high-speed blending with acetylation as a pretreatment. Acetylation with acetic anhydride has been widely used to improve thermal stability.

In this study, windmill palm fiber extracted by an alkali–oxygen method has been acetylated with acetyl chloride and acetic anhydride. The structure and properties of the product have been systematically analyzed. Conversion of the hydrophilic raw material into hydrophobic fibers dramatically improved the bulkiness, making the product more suitable for use as a filling material. The hydrophobic modification increased the adhesion between fiber and polymer, increasing the mechanical properties of fiber-reinforced composites. It is hoped that this research will promote the further development and utilization of windmill palm fiber.

**Materials and methods**

**Materials**

Windmill palm fiber. Windmill palm sheath meshes were obtained from Huangshan, Anhui Province, China. The windmill palm fiber was pulled from the palm sheaths, washed under running water, and then allowed to dry naturally. It was subsequently treated with 4 wt.% hydrogen peroxide and 2 wt.% sodium hydroxide solution in a 1:100 fiber-to-extractant ratio (g/mL) at 70°C for 6 h. The residue was repeatedly washed with running water. The residue was then dispersed into individual short fibers by means of a blender to prepare the alkalized windmill palm fiber.

Acetylated modified palm fiber

Preparation of acetyl chloride-treated windmill palm fiber. Windmill palm fiber (2 g) was placed in a 250 mL conical flask with N,N-dimethylformamide (DMF; 50 mL), and the flask was placed in a constant temperature water bath. The mixture was stirred for 15 min to ensure even soaking. A certain amount of acetyl chloride and a catalytic amount of pyridine were then sequentially added. After treatment at a specific temperature for a predetermined time, as shown in Table 1, the modified solids were repeatedly washed with ethanol and running water. The effects of acetyl chloride concentration, pyridine concentration, and reaction time on the hydrophobicity of the modified windmill palm fiber were evaluated. Each factor was varied over four levels.

Preparation of acetic anhydride-treated windmill palm fiber. An orthogonal experiment was designed to optimize the preparation process of acetic anhydride-treated windmill palm fiber. The influencing factors and their levels for each sample are shown in Table 2. The fiber was placed in a conical flask containing a certain amount of acetic anhydride and acetic acid. After a few minutes, a catalytic amount of aluminum perchlorate was added. The washing and drying process was as described for the acetyl chloride-treated windmill palm fiber.

**Methods**

**Surface morphology.** The micromorphology of the windmill palm fiber was analyzed by means of an electron microscope (TM3030; Hitachi, Tokyo, Japan). Samples dissected along the longitudinal direction were fixed on an electron microscope stage. Nano-gold particles were applied by ion sputtering (E-1045; Hitachi, Tokyo, Japan).

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**Table 1. Factors and levels of the orthogonal design for anhydride treatment**

| Levels | A (Acetyl chloride, wt.%) | B (Pyridine, wt.%) | C (Temperature, °C) | D (Time, h) |
|--------|--------------------------|-------------------|---------------------|-------------|
| 1      | 17.5                     | 5.0               | 40                  | 4           |
| 2      | 12.5                     | 2.5               | 50                  | 5           |
| 3      | 7.5                      | 7.5               | 45                  | 6           |
| 4      | 2.5                      | 10.0              | 55                  | 3           |

**Table 2. Factors and levels of the orthogonal design for acetic anhydride treatment**

| Levels | A (Acetic anhydride, wt.%) | B (Aluminum perchlorate, wt.%) | C (Temperature, °C) | D (Time, h) |
|--------|---------------------------|-------------------------------|---------------------|-------------|
| 1      | 56                        | 0.08                          | 70                  | 6           |
| 2      | 44                        | 0.12                          | 90                  | 8           |
| 3      | 32                        | 0.04                          | 80                  | 4           |
| 4      | 68                        | 0.16                          | 60                  | 2           |
Japan) to increase the conductivity of the samples. The acceleration voltage was 5 kV during testing. The diameter and the length of the windmill palm single fiber with different treatments had been tested by the Imagpro software based on the scanning electron microscopy (SEM) pictures. At least 50 samples had been tested for each sample.

Fourier transform infrared spectrum. Fourier transform infrared spectrum (FTIR; Nicolet 5700, USA) was used to analyze all the windmill palm fiber samples. The FTIR spectra were recorded in the range of 400–4000 cm⁻¹, with 16 scans at a resolution of 8 cm⁻¹.

Static contact angle. The static wettability of the windmill palm fiber was observed using a DSA100 drop shape analyzer instrument (Krüss GmbH, Hamburg, Germany) equipped with a high-speed frame camera (360 fps). The fibers were dispersed in water, then collected by a filter screen and dried to make uniform sheet materials for testing. Water droplets of volume 4 μL were loaded on the surfaces of the respective samples. The static contact angle was measured (three times per sample) within 60 s.

Sound absorption performance. Windmill palm fibers of 5 g, 10 g, 15 g and 18 g were dispersed in water, then collected by a filter screen and dried to make palm mats with different surface density. The samples were cut into circles of radius of 5 cm and 1.5 cm, respectively. The sound absorption coefficients of windmill palm fiber nonwoven mats were measured by the transfer function method using an impedance tube (SW463; Reputation, China). Each sample was tested three times, and the average value was taken. The test results of three acoustic frequency bands (80–400 Hz, 400–1600 Hz, 1600–6300 Hz) were fitted to a curve. The sound absorption coefficient was taken as the average value over six octaves of 125, 250, 500, 1000, 2000, and 4000 Hz.

Thermal conductivity. Windmill palm fiber, cotton, and wool were placed in 10 cm × 10 cm polyester mesh bags. The moisture contents of these samples with a bulk density of 300 g/m³ were allowed to equilibrate for 24 h under standard atmospheric conditions. The thermal conductivities of windmill palm fiber samples after different modification treatments were tested with a KES-F7 apparatus (THERMO LABO II, Japan).

Thermogravimetric analysis. Thermogravimetric analysis was carried out using a thermogravimetric analyzer (5700; TA, USA). Thermograms were acquired between 35°C and 600°C at a heating rate of 10°C/min, with nitrogen as the purge gas at a flow rate of 50 mL/min.

Results and discussion

Acetylation treatment and the resultant hydrophobicity

The effect of acetyl chloride modification on the hydrophobicity of windmill palm fiber was discussed according to an orthogonal experimental design. The static water contact angle was taken as the evaluation index. The effects of acetyl chloride and pyridine concentrations and of treatment temperature and time on hydrophobic modification of the fibers were analyzed. The results obtained are shown in Table 3. All of the tested samples showed a static water contact angle of more than 90°. The maximum value attained was 148°. The optimal hydrophobicity of acetylated windmill palm fiber can contribute to its oil adsorption ability.19 The higher the R value, the more significant the influence of the corresponding factor on the static contact angle. The blank row of R_E was bigger than R_C and R_D in the control group. The lower values of R in the columns headed C and D compared to that in the column headed E indicated that the treatment temperature and time had no significant effect on the static contact angle of palm fiber in the ranges 40–55°C and

| Table 3. Hydrophobic characteristics of acetyl chloride-modified fibers treated under different conditions |
|---|---|---|---|---|---|---|
| Samples | Factors | A | B | C | D | E_{Blank} | CA/° |
| 1 | 1 | 1 | 1 | 1 | 1 | 1 | 128 |
| 2 | 1 | 2 | 2 | 2 | 2 | 130 |
| 3 | 1 | 3 | 3 | 3 | 3 | 136 |
| 4 | 1 | 4 | 4 | 4 | 4 | 120 |
| 5 | 2 | 1 | 2 | 3 | 4 | 125 |
| 6 | 2 | 2 | 1 | 4 | 3 | 127 |
| 7 | 2 | 3 | 4 | 1 | 2 | 148 |
| 8 | 2 | 4 | 3 | 2 | 1 | 116 |
| 9 | 3 | 1 | 3 | 4 | 2 | 124 |
| 10 | 3 | 2 | 4 | 3 | 1 | 117 |
| 11 | 3 | 3 | 1 | 2 | 4 | 122 |
| 12 | 3 | 4 | 2 | 1 | 3 | 120 |
| 13 | 4 | 1 | 4 | 2 | 3 | 97 |
| 14 | 4 | 2 | 3 | 1 | 4 | 98 |
| 15 | 4 | 3 | 2 | 4 | 1 | 112 |
| 16 | 4 | 4 | 1 | 3 | 2 | 109 |
| K_1 | 514 | 474 | 486 | 494 | 473 |
| K_2 | 516 | 472 | 487 | 465 | 511 |
| K_3 | 483 | 518 | 474 | 487 | 480 |
| K_4 | 416 | 465 | 482 | 483 | 465 |
| R | 100 | 53 | 13 | 29 | 46 |
3–6 h, respectively. The R values for the other two factors indicate that A (concentration of acetyl chloride) had a more significant influence on the results than B (concentration of pyridine). The appropriate process level can be chosen as that yielding the highest value of \( k \). Therefore, \( A_2B_3 \) was identified as the best process for modification with acetyl chloride, that is, with 12.5 wt.% acetyl chloride and 7.5 wt.% pyridine.

The optimum conditions for acetic anhydride modification of windmill palm fiber were likewise identified through orthogonal experimental design. The results are shown in Table 4. As with acetyl chloride modification, the water contact angles of all samples were above 90°. The maximum static contact angle following acetic anhydride modification was 143°, slightly lower than that for the optimal acetyl chloride-modified sample.

The extreme value of each factor was more significant than the comparison column \( R_e \), which indicated that all factors significantly influenced the results. According to the R values, the influences of various factors on the hydrophobicity of the fibers decreased in the order: \( B \) (concentration of aluminum perchlorate) > \( D \) (treatment time) > \( A \) (concentration of acetic anhydride) > \( C \) (treatment temperature). The optimum level of each factor in the modification process, giving the highest \( k \) value, was \( A_4B_2C_1D_3 \). When the concentration of acetic anhydride was 68 wt.%(acetic acid concentration 32 wt.%) and the concentration of aluminum perchlorate was 0.12 wt.%, the most hydrophobic palm fiber velvet was obtained after treatment at 70°C for 4 h.

The best processes were then applied to treat windmill palm fibers by acetyl chloride modification and acetic anhydride modification. The static water contact angles were 148° and 145°, respectively, showing excellent hydrophobicity.

### Microstructural characteristics

Figure 1 shows the surface morphologies of windmill palm short fiber before and after acetylation modification at different enlargements. Windmill palm short fibers extracted by alkali treatment showed smoother and cleaner surfaces due to the light damage to the fiber cell walls. The surfaces of the acetylated windmill palm fibers contained many small holes and showed partially broken cell walls. Windmill palm fiber is mainly composed of cellulose, hemicellulose, and lignin connected by hydrogen bonding. Acetylation replaces hydrogen bonds with acetyl groups, destroying the compact cell structure; causing damage to the cell wall, in turn holes appear. The windmill palm short fibers modified by acetyl chloride were twisted along the axial direction, as shown in Figure 1(e). The spiral-like fibers were therefore shortened by the modification. The fiber surface was not smooth, with irregular granular adhesions. After acetic anhydride modification, the fiber surface showed an extensive fibrillation phenomenon. Also, many more pores appeared in the cell walls compared with acetyl chloride treatment. The fibrillation as well as the micro pores increased the surface roughness of the windmill palm fiber, which increased the hydrophobic property. The SEM image in Figure 1(i) reveals that the pores in the fiber cell walls were open and discontinuous. Acetylation modification greatly influenced the surface morphology of the fibers, loosening the initially dense structure.

#### Table 4. Hydrophobic characteristics of acetic anhydride-modified fibers treated under different conditions

| Samples | A | B | C | D | EBlank | CA° |
|---------|---|---|---|---|--------|-----|
| 1       | 1 | 1 | 1 | 1 | 143    |
| 2       | 1 | 2 | 2 | 2 | 123    |
| 3       | 1 | 3 | 3 | 3 | 134    |
| 4       | 1 | 4 | 4 | 4 | 107    |
| 5       | 2 | 1 | 2 | 3 | 123    |
| 6       | 2 | 2 | 1 | 4 | 127    |
| 7       | 2 | 3 | 4 | 1 | 108    |
| 8       | 2 | 4 | 3 | 2 | 91     |
| 9       | 3 | 1 | 3 | 4 | 126    |
| 10      | 3 | 2 | 4 | 3 | 137    |
| 11      | 3 | 3 | 1 | 2 | 114    |
| 12      | 3 | 4 | 2 | 1 | 121    |
| 13      | 4 | 1 | 4 | 2 | 130    |
| 14      | 4 | 2 | 3 | 1 | 136    |
| 15      | 4 | 3 | 2 | 4 | 114    |
| 16      | 4 | 4 | 1 | 3 | 132    |
| \( K_1 \) | 507 | 522 | 516 | 508 | 485 |
| \( K_2 \) | 449 | 523 | 481 | 458 | 489 |
| \( K_3 \) | 498 | 470 | 487 | 526 | 512 |
| \( K_4 \) | 512 | 451 | 482 | 474 | 480 |
| R       | 63 | 72 | 35 | 68 | 32     |

Microscopic morphology of palm fiber

Windmill palm fibers become paper-like thin sheets with hydrophilic properties when naturally air dried or oven dried. These short fibers were connected by hydrogen bonding, making them difficult to disperse. Water dyed with methylene blue was instantly absorbed by oven-dried windmill palm fiber (Figure 2(a), (d)), showing the hydrophilicity of the solid. When water droplets contact the fiber surface in air, they spread out and the contact angles approach 10° in a short time, illustrating the hydrophilicity property of the materials. The chloroacetyl chloride-treated fibers with a hydrophobic surface appeared fluffy.
Figure 1. Windmill palm short fiber with different chemical treatment: (a), (b) and (c) alkali-treated windmill palm fiber; (d), (e) and (f) acetyl chloride-treated windmill palm fiber; (g), (h) and (i) acetic anhydride-treated windmill palm fiber.

Figure 2. Windmill palm fiber before and after acetylation modification: (a) alkali-treated windmill palm fiber with a hydrophilic surface; (b) acetyl chloride-treated windmill palm fiber with a hydrophobic surface; (c) acetic anhydride-treated windmill palm fiber with hydrophobic surface.
after being oven dried for the lack of hydrogen bond (Figure 2(e)). The blue water drops were seen to be spherical on the surface of the fiber pile (Figure 2(b)). Acetic anhydride-modified fibers had an average statistic contact angle of 151 ± 3°. Nevertheless, their hydrophilic and hydrophobic properties were quite different. Water droplets also maintained a spherical structure of the acetic anhydride-modified fibers (Figure 2(c)), with a statistic contact angle of 153 ± 4°, demonstrating good water repellence.

The morphological parameters of windmill palm fibers with different chemical treatments are shown in Figure 3. The single fiber after alkali treatment has the longest length and largest fineness at 698 ± 213 μm and 10.84 ± 2.41 μm, respectively. After acetylation, the length and fineness of the fiber is reduced to varying degrees. The fiber breakage contributes most to the decrease of the fiber length. The partial degradation of the cell wall leads to the decrease of fiber fineness. Among them, the length and fineness of the fiber treated by acetic anhydride are 291 ± 130 μm and 6.20 ± 2.17 μm, respectively. Unlike the decrease of diameter and length, acetylation increases the aspect ratio (length:diameter) of fiber from 48 for alkali treatment to 73 for acetyl chloride treatment and 98 for acetic anhydride treatment. The higher aspect ratio may benefit the mechanical property of the fiber-reinforced composites. In addition, acetylation treatment increases the curl of the fiber, which can be seen in Figure 1. The curl of the fiber is characterized by the ratio of the projected length of the real length of the fiber. The curl degree of the three samples is 0.94, 0.87 and 0.73, respectively.

### Fourier transform infrared spectrum

The chemical composition of windmill palm fiber after different chemical treatments based on infrared are shown in Figure 4. Almost all the samples have two obvious broad peaks at 3400 cm⁻¹ and 2900 cm⁻¹, which are the vibration of OH and CH, respectively. As a lignocellulose fiber, windmill palm fiber is mainly composed of cellulose, hemicellulose, and lignin. The characteristic lignin peak appears at 1500–1600 cm⁻¹, which belongs to the stretching vibration of C=\text{C} in the benzene ring. The characteristic functional group of hemicellulose appears at 1741 cm⁻¹, which belongs to the vibration of C=O or the CH₃CO group. These functional groups were not found in the infrared spectra of the chemically treated single fiber samples, which indicated that the samples were relatively pure cellulose without lignin and hemicellulose. An obvious absorption peak can be seen at about 1640 cm⁻¹ of alkali-treated fiber, which is the vibration of OH contained in cellulose-bound water. The peak at 1380 cm⁻¹ is the vibration of C-CH₃. The palm fiber treated by acetyl chloride or acetic anhydride has strong narrow frequency absorption peaks at 175 cm⁻¹ and 1240 cm⁻¹. The peak at 1757 cm⁻¹ can be attributed to the stretching vibration of the carbonyl group.
(C=O) in the ester bond. The peak at 1240 cm\(^{-1}\) belongs to the extension of C-O of the acetyl (CH\(_3\)CO) group. The obvious existence of these two peaks confirmed that both acetyl chloride and acetic anhydride could acetylate with palm fiber. The increasing hydrophobic groups such as CH\(_3\)CO contribute significantly to the water repellence.

**Sound absorption property**

Windmill palm fiber velvet samples treated with acetyl chloride and acetic anhydride were used as raw materials to prepare nonwoven mats to study their sound absorption properties. The effects of treatment methods and surface density on the acoustic properties are shown in Figure 5. The sound absorption coefficient exceeds 0.2 in most frequency bands, indicating that all of the samples had good sound absorption performance. The acetylated windmill palm fiber had the highest sound absorption coefficient. The alkali-treated windmill palm fiber nonwoven mat had the highest acoustic property when the thickness was 10.5 mm, with a sound absorption coefficient of 0.43 at a surface density of 0.187 g/cm\(^2\). The lumen in the middle of the fiber and the pits in the cell walls provided continuous pores, allowing sound waves to pass through multiple interstices that improve the sound absorption property.

As shown in Figure 5(b), the peak value of the sound absorption coefficient of acetyl chloride-treated windmill palm fibers shifted to a lower frequency with increasing surface density. It then reverted to a higher frequency when the surface density reached 0.140 g/cm\(^2\). The average sound absorption coefficients

![Image](image-url)
of the four materials showed the same trend, and amounted to 0.23, 0.35, 0.47, and 0.42, respectively. When the surface density was 0.047 g/cm², the sound absorption coefficient of the material increased uniformly with increasing frequency. In contrast, the sound absorption coefficient plots of the other materials can be divided into three stages. In the first stage, the rate of increase was fastest and the peak value was reached. The average sound absorption coefficient then decreased slightly and finally remained stable. The maximum sound absorption of the samples reached 0.92 when the surface density was in the range 0.093–0.14 g/cm², higher than that of kapok fiber.31

The parameters $d_1$ and $d_2$ were defined as the frequency widths of the slowest or fastest material reaching and always maintained above 0.5, respectively. No $d_1$ or $d_2$ could be determined for an alkali-treated windmill palm fiber nonwoven mat, because this material with a surface density of 0.047 g/cm² did not attain a coefficient of 0.5. The $d_1$ was about 1.4 times higher than $d_2$ for an acetyl chloride-treated palm fiber nonwoven mat.

The peak value of the sound absorption coefficient for materials treated with acetic anhydride gradually shifted to a lower frequency with increasing surface density. The highest average sound absorption coefficient was 0.38, much lower than that of the acetyl chloride-treated windmill palm fiber mats. The decreased thickness shown in Figure 3(d) resulted in the decrease of acoustic properties, the same as reported in the literature.32 What is more, the increase in the pore diameter, which can be seen in Figure 2, decreased the acoustic property.33 The $d_1$ was about 4.1 times higher than $d_2$ for acetic anhydride-treated palm fibers. The results indicated that surface density has a great influence on the sound absorption properties. All of the samples exhibited absorption coefficients greater than 0.5 for frequencies from 2000 Hz to 5000 Hz when the surface density was above 0.093 g/cm². The sound absorption properties of poplar seed, kapok, wool, hemp and cotton have been reported, and the average sound absorption coefficients of these five samples were 0.225, 0.095, 0.273, 0.385 and 0.38,34 much lower than that of the windmill palm fiber with the same thickness. It can be seen in Figure 2(a) and (c) that the sound absorption performances of alkali and acetic anhydride-treated fibers were poor at the surface density of 0.047 g/cm². It is mainly affected by the

![Figure 5. Sound absorption performance of palm fiber with different surface density: (a) alkali-treated windmill palm fiber; (b) acetyl chloride-treated palm fiber velvet; and (c) acetic anhydride-treated palm fiber velvet; (d) the thickness of three kinds palm fiber nonwoven mats.](image-url)
lower thickness (3.2 mm) for the alkalized sample, while the influence of increasing hydrophobicity and shortening fiber length on the change of pore space was mutually restricted for acetic anhydride-treated fibers, limiting the further improvement of the sound absorption performance of the samples.

**Thermal insulation performance**

Cellulose fibers have long been used for thermal insulation. However, the properties of windmill palm fiber following different treatments have yet to be investigated. Here, the thermal conductivities of alkalized windmill palm fiber, acetyl chloride-treated palm fiber, and acetic anhydride-treated palm fiber were found to be similar at 0.047 ± 0.015, 0.048 ± 0.016, and 0.050 ± 0.013 W/m/K, respectively. The thermal conductivities of cotton and wool are 0.066 ± 0.015 and 0.057 ± 0.011 W/m/K, respectively, higher than those of windmill palm fiber samples at the same bulk density (Figure 6). Each windmill palm fiber has a big lumen in the middle, resulting in a high hollowness range from 28% to 48%. A high degree of hollowness can catch a large amount of still air, which endows windmill palm fibers with excellent thermal insulation performance such that they might be used as a filler in the textile field.

**Thermal properties**

The thermal properties (TG) of alkalized and acetylated palm fiber are shown in Figure 7. The degradation of all samples can be divided into three stages. The first stage in the range 30–100°C mainly involves the evaporation of moisture from the fiber cell walls. In the second stage, lignin, hemicellulose, and cellulose are degraded by the breaking of chemical bonds. The third stage involves simultaneous carbonization and oxidation. The initial decomposition temperatures of windmill palm fiber treated with alkali, acetyl chloride, and acetic anhydride were determined as 333°C, 336°C, and 356°C, respectively. Thus, acetylation slightly improves the thermal stability of the material. The fastest rates of change in the TG traces were around the main decomposition temperatures of the material, that is 330°C, 337°C, and 356°C, respectively. Thus, acetylation slightly improves the thermal stability of the material. When the temperature reached 600°C, the decomposition of the materials was complete, and the residual carbon content was stable. The residual carbon content was highest for the windmill palm fiber treated by alkali, amounting to 22.4%, and the residual carbon content of the windmill palm fiber treated with acetic anhydride was the lowest, amounting to 11.4%.

**Conclusions**

In this study, alkali and acetylation treatments have been used to overcome the shortcoming of the strong hydrophilicity of windmill palm fiber to expand its applicability in the fields of fillings and composites. Both acetyl chloride and acetic anhydride treatments improved the hydrophobicity of windmill palm fiber, affording products with static water contact angles above 145°. Acetylation treatment introduced many pore structures in the fiber cell walls. When using such materials as reinforcing matrices, it is necessary to balance the poor adhesion caused by hydrophilicity
and the decreased strength caused by the destruction of cell walls. After acetic anhydride treatment, the thermal stability of the fibers was increased, with the initial decomposition temperature increasing from 330°C to 356°C. This may serve to improve the mechanical properties of hot-pressed composites. The modified windmill palm fiber retains low thermal conductivity, lower than that of cotton and wool. Windmill palm fiber also shows better sound absorption performance because of its high hollowness and multiscale pore structure imparted by acetyl chloride treatment. The average sound absorption coefficient reached 0.47, because of the unique structure dissipating more energy.

**Author contributions**

Chen Changjie, Xinhou Wang and Wang Guoheng conceived and designed the experiments; Chen Changjie, Jing Tan and Weiguang Zhang performed the experiments; Chen Changjie and Zhong Wang analyzed the data; Zhong Wang contributed reagents/materials/analysis tools; Chen Changjie wrote the paper.

**Declaration of conflicting interests**

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