Effect of Acetylation Modification on 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, its application in textile filling and composite reinforcement is limited due to its hydrophilic solid. In this paper, effects of chemical treatment such as alkali, acetyl chloride, and acetic anhydride treatment on the micro-morphology, water repellency, sound absorption, thermal conductivity and thermal stability of the windmill palm fiber were investigated. The results showed that the surface of windmill palm fiber treated by alkali was relatively smooth and compact. In comparison, the acetylation treatment destroyed the cell wall with micro-holes on the surface. Acetylation modification can significantly improve the water repellency of fibers. Furthermore, the water static contact angle can be achieved 148°. Acetylation treatment improved the sound absorption performance of the fiber mat with an average sound absorption coefficient of 0.47, kept low thermal conductivity of under 0.050 W/(m°C), and improved the thermal stability with the initial decomposition temperature 356°C.

Introduce

Natural fibers from renewable green resources such as crop straw, vegetable, bamboo, hemp, wood and so on, have excellent thermal and mechanical properties, low cost, sustainability, and environmentally friendly (Mahdi et al., 2021). High-cost performance and environmental protection offer natural fiber the potential to act as a biodegradable material alternative for the use of synthetic fibers (Jawaid et al., 2013). Among these green fibers, windmill palm fiber extracted from windmill palm sheath meshes is unique for its toughness. It has the highest elongation at break and excellent energy absorption ability (Chen et al., 2017a; Chen et al., 2017b; Wang et al., 2014; Zhu et al., 2019), acoustic insulation (Chen et al., 2018; Chen et al.), as well as UV resistance (Wang et al., 2016). Now, the windmill palm tree represents an essential source 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 lumen in the middle. The length/diameter ratio is more than 60 (Chen et al., 2017b). Cellulose short fibers with a large length/diameter ratio have been used as reinforcement in composites to improve their behaviours, especially mechanical properties (Taheri et al., 2021). At the molecular level, cellulose chains have regular equatorially arranged hydrophilic hydroxy (OH) groups (Ishida et al., 2021), leads to poor dispersibility and incompatibility with other nonpolar media and matrix (Wu et al., 2018). The blending of relatively hydrophobic polymers and hydrophilic cellulose short fibers often results in poor interfacial adhesion between the materials, resulting in poor mechanical properties (Taheri et al., 2021; Trinh et al., 2021).

Alkali treatment, esterification and etherification are standard methods for hydrophobic modification of cellulose fibers. Nevertheless, alkali treatment still has poor water resistance because large hydroxyl groups make the cellulose fiber hydrophilic (Yang et al., 2021). Among them, acetylation modification is a practical modification method on the surface of the fiber, in which acetyl groups (CH₃CO-) react with the surface hydroxyl groups (OH) of cellulose, making its surface less hydrophilic (Quintana et al., 2018). Lin

Gan increased the tensile strength and elongation at the break of surface acetylation cellulose nanocrystals reinforced composite by 43.3 and 12.6%(Gan et al., 2017). Rainey develops a new method for creating high-quality CNF by high-speed blending with acetylation as a pretreatment(Sofla et al., 2019). Acetylation with acetic anhydride (AA) has been widely used to improve thermal stabilities(Jhu et al., 2019).

In this paper, the windmill palm fiber extracted by the alkali-oxygen method was acetylated by acetyl chloride and acetic anhydride. The structure and properties were studied and analyzed systematically. The hydrophilic fiber converted into hydrophobic raw material dramatically improves the bulkiness for its using as a filling material. The hydrophobic modification increased the adhesion between fiber and polymer, increasing the mechanical properties of fiber-reinforced composites. The 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. Windmill palm fiber was pulled out from the palm sheath, washed under running water, and then dried naturally. The windmill palm fiber was treated with 4 wt.% hydrogen peroxide (H_2O_2) and 2 wt.% sodium hydroxide solution (NaOH) with a 1:100 fiber-to-extractant ratio (g/mL) at 70°C for 6h. The residue was washed with running water several times. Then the residue was dispersed into individual short fibers with a blender to prepare the alkalized windmill palm fiber.

Acetylated Modified Palm Fiber

Preparation of acetyl chloride treated windmill palm fiber

2 g windmill palm fiber was put into a 250 mL conical flask with 50 ml of N, N- dimethylformamide (DMF) under a constant temperature water bath pot. The materials were stirred for 15 min until it is soaked evenly. A certain amount of acetyl chloride and catalyst pyridine had been added in turn. Samples were washed with alcohol and running water several times after being treated under a specific temperature for a predetermined time, as shown in Table 1. The effects of acetyl chloride concentration, pyridine concentration and reaction time on hydrophobic properties of windmill palm fiber were discussed. Each factor has four levels.

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

Levels	Factors			
	A	B	C	D
	Acetyl chloride (vt.%)	Pyridine (vt.%)	Temperature (°C)	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

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 levels of each sample were shown in Table 2. The fiber was added in to a conical flask containing a certain amount of acetic anhydride and acetic acid. Catalyst aluminium perchlorate had been added after a few minutes. The washing and drying process is just like the acetyl chloride treated windmill palm fiber.

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

Levels	Factors			
	A	B	C	D
	Acetic anhydride (vt.%)	Aluminum perchlorate (Vt.%)	Temperature (°C)	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

Methods

Surface morphology

The micromorphology of windmill palm fiber was analyzed by an electron microscope (TM3030, Hitachi, Tokyo, Japan). Samples dissected along the longitudinal directions were fixed on an electron microscope stage. Nano-gold particles were coated by ion sputtering (E-1045, Hitachi, Tokyo, Japan) to increase the conductivity of the samples. The acceleration voltage was 5 kV during testing.

Static Contact Angle

The static wettability of the windmill palm fiber was observed using a drop shape analyzer DSA100 instrument (Krüss GmbH, Hamburg, Germany) equipped with a high-speed frame camera (360 fps). A water droplet with a volume of 4 μL was loaded on the surface of the different samples. The static contact angle was measured (three times per sample) within 60 s.

Sound Absorption Performance

The sound absorption coefficient of windmill palm fiber nonwoven mats was measured using the transfer function method by impedance tube (SW463, Reputation, China). Different samples were cut into circles with diameters of 10 cm and 3 cm, respectively. Each sample was tested three times to take the average value. The test results of three acoustic frequency bands (80 Hz ~ 500 Hz, 400 Hz~1600 Hz, 1600 Hz ~ 6300 Hz) were fitted into a curve. The average sound absorption coefficient was taken as the average sound absorption coefficient of six octaves of 125, 250, 500, 1000, 2000 and 4000 Hz.

Thermal Conductivity

Windmill palm fiber, cotton and wool were filled into a 10 cm×10 cm size polyester mesh bag. The moisture regains of these samples with a bulk density of 300 g/m³ were balanced for 24 h under standard atmospheric conditions. The thermal conductivity of windmill palm fibers with different modification treatments was tested by KES-F7(THERMO LABO, Japan).

Thermogravimetric Analysis

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

Results And Discussion

Acetylated treatment and the hydrophobic property

The effect of acetyl chloride modification on the hydrophobic properties of windmill palm fiber was discussed according to the orthogonal experimental design. The static contact angle was treated as the evaluation index. The effects of acetyl chloride and pyridine concentration, treatment temperature and time on hydrophobic modification of fiber were analyzed. The specific results were shown in Table 3. All the samples under different conditions had a static contact angle of more than 90 °. The maximum

reached 148 °. The perfect hydrophobic property of acetylated windmill palm fiber may gift oil absorption ability(Do et al., 2020).

The higher the R value was, the more significant the influence of the corresponding factors on the static contact angle will have. The blank row of R_E bigger than R_C and R_D in the control group indicated that the treatment temperature and time had no significant effect on the static contact angle of palm fiber in the range of 40 ~ 55 °C and 3 ~ 6 h, respectively. The other two factors judge that A (concentration of acetyl chloride) had a more significant influence on the results than B (concentration of pyridine) according to the value of R. The proper process level can be chosen when the value of k was the largest. Therefore, A_2B_3 was the best process for the modification of acetyl chloride. The results showed that the concentration of acetyl chloride was 12.5 wt.%, and the concentration of pyridine was 7.5 wt.% was the best solution.

Table 3
Hydrophobic characteristics of acetyl chloride modified fibers
treated by different conditions

Samples	Factors					CA/ °
	A	B	C	D	EBlank	
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	

The optimum design scheme of acetic anhydride modified windmill palm fiber was discussed through the orthogonal experimental design. The results were shown in Table 4. As with acetyl chloride modification, the contact angles of all samples were above 90 °. The maximum static contact angle of acetic anhydride modification was 143 °, slightly lower than the acetyl chloride modified sample.

Table 4
The hydrophobic characteristics of acetic anhydride modified
treated by different conditions

Samples	Factors					CA / °
	A	B	C	D	E Blank	
1	1	1	1	1	1	143
2	1	2	2	2	2	123
3	1	3	3	3	3	134
4	1	4	4	4	4	107
5	2	1	2	3	4	123
6	2	2	1	4	3	127
7	2	3	4	1	2	108
8	2	4	3	2	1	91
9	3	1	3	4	2	126
10	3	2	4	3	1	137
11	3	3	1	2	4	114
12	3	4	2	1	3	121
13	4	1	4	2	3	130
14	4	2	3	1	4	136
15	4	3	2	4	1	114
16	4	4	1	3	2	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	

The extreme value of each factor was more significant than the comparison column R_E, which indicated that all factors significantly influenced the results. The influence of various factors on the hydrophobic property of fiber was B (concentration of aluminium perchlorate) > D (treatment time) > A (concentration of acetic anhydride) > C (treatment temperature), according to the value of R. The optimum technological

level of each factor in the modification process was $A_4B_2C_1D_3$, due to the highest k value. When the concentration of acetic anhydride was 68 wt.% (at this time, the concentration of acetic acid is 32 wt.%) and the concentration of aluminium perchlorate was 0.12 wt.%, the best hydrophobic property of palm fiber velvet can be obtained after being treated at 70 °C for 4 h.

The best process then treated windmill palm fibers of acetyl chloride modification and acetic anhydride modification. The water static contact angles were 148 and 145 °, respectively, showing excellent hydrophobic properties.

Microstructural Characteristics

Figure 1 described the surface morphology of windmill palm short fiber before and after acetylation modification under different enlargement times. Windmill palm short fibers extracted by alkali treatment had smoother and cleaner surfaces due to the damage to the fiber cell wall. At the same time, the surfaces of acetylated modification windmill palm fibers contained many small holes and partially broken cell wall. Acetylate treatment destroyed hydrogen bonding and damages fiber cell wall (Chen et al., 2018). The windmill palm short fibers modified by acetyl chloride were twisted along the axial direction showed in Fig. 1e. The spiral-like fiber had a shorter length after modification. The fiber surface was not smooth, with irregular granular adhesions. After acetic anhydride modification, the fiber surface appeared serious fibrillation phenomenon (Gudayu et al., 2020), and the pores on the cell wall increased significantly compared with acetyl chloride treatment. SEM imaging of Fig. 1i revealed that the pores on fiber cell wall are open and discontinuous. Acetylation modification greatly influenced the surface morphology of fibers, which made the dense structure loose, leading to the decrease of mechanical properties.

Microscopic Morphology Of Palm Fiber

Windmill palm fibers will become paper-like thin sheets with hydrophilic properties through natural air dried or oven-dried. These short fibers were connected by hydrogen bonding, which is difficult to disperse. The methylene blue-dyed liquid can be instantly absorbed by freeze-dried windmill palm fiber (Fig. 2a), showing solid hydrophilic properties. The colour of acetylated fiber was light yellow with a hydrophobic surface. The blue water drops were spherical on the surface of fiber pile (Fig. 2b). Acetic anhydride-modified fiber was white and similar to alkali-treated fiber. At the same time, the hydrophilic and hydrophobic properties were quite different. Water droplets can maintain a spherical structure on acetic anhydride modified fiber (Fig. 2c). It had a good water repellency.

Sound Absorption Property

Windmill palm fiber velvet treated with acetyl chloride and acetic anhydride were used as raw materials to prepare the nonwoven mats to study the sound absorption property. The effect of treated methods and surface density on the acoustic property was shown in Fig. 3. The sound absorption coefficient exceeds 0.2 in most frequency bands, indicating the samples all had good sound absorption performance. In contrast, the acetylated windmill palm fiber had the highest sound absorption coefficient. The alkali treated windmill palm fiber nonwoven mat had the highest acoustic property with the sound absorption coefficient of 0.43 when the surface density reached 0.187g/cm^2 . The lumen in the middle of the fiber and the pits in the cell wall becomes continuous pores that made sound waves pass through multiple interstices (Ferreira et al., 2021).

As shown in Fig. 3b, the peak value of the acetyl chloride-treated windmill palm fibre's sound absorption coefficient moves to low frequency with surface density increase. Then the line turned back to high frequency when the surface density reached 0.140g/cm^2 . The average sound absorption coefficients of the four materials have the same trend, which was 0.23, 0.35, 0.47 and 0.42, respectively. When the surface density was 0.047g/cm^2 , the sound absorption coefficient of the material increased uniformly with the increase of frequency. In contrast, the sound absorption coefficient line of the other materials can be divided into three stages. In the first stage, the rising rate was the fastest and reached the peak value. Then the average sound absorption coefficient decreased slightly and remained stable at last. The maximum sound absorption of the samples reached 0.92 when the surface density was in the range of 0.093 to 0.14g/cm^2 , higher than the kapok fiber (Zheng et al., 2021). The d_1 and d_2 were defined as the frequency widths of the slowest or fastest material reaching and always maintaining above 0.5, respectively. There was no d_1 or d_2 for alkali treated windmill palm fiber nonwoven mat for this material with a surface density of 0.047g/cm^2 did not reach 0.5. d_1 was about 1.4 times of d_2 for acetyl chloride treated palm fiber nonwoven mat.

The peak value of sound absorption coefficient of materials treated by acetic anhydride gradually moved to low frequency with increased surface density. The average sound absorption coefficients of the four materials are increasing continuously, which are 0.15, 0.26, 0.33 and 0.42, respectively. The d_1 was about 4.1 times of d_2 for acetic anhydride treated palm fiber. It indicated that the surface density had a great influence on the sound absorption properties. All the samples exhibited absorption coefficients greater than 0.5 for frequencies of up to 2000 Hz, when the surface density was above 0.047g/cm^2 . It had the same acoustic property as coir fiber/polymer composites (Cheewawuttipong and Memon, 2021). Thus, the windmill palm fiber nonwoven mats were suitable for use as sound absorption materials.

Warm Performance

Cellulose fibers were in everyday use as thermal insulation for many years (Brzyski et al., 2019). The properties of windmill palm fiber with different treatments should be still investigated. The thermal conductivity of alkalized windmill palm fiber, acetyl chloride treated palm fiber, and acetic anhydride treated palm fiber were similar, which were 0.047 ± 0.015 , 0.048 ± 0.016 and $0.050 \pm 0.013 \text{ W/(m}^\circ\text{C)}$,

respectively. The thermal conductivity of cotton and wool were $0.066 \pm 0.015 \text{ W/(m}^\circ\text{C)}$ and $0.057 \pm 0.011 \text{ W/(m}^\circ\text{C)}$. It much higher than windmill palm fiber samples under the same bulk density. High hollowness can catch a large amount of still air, which made windmill palm fiber have excellent thermal insulation performance and can be used as filler in the textile field.

Thermal Property

The thermal properties of untreated and acetylated palm fiber were shown in Fig. 6 – 5. The degradation of all samples was divided into three stages. In the first stage, the temperature was about 30–100 °C, mainly the evaporate moisture content from the fibers cell wall(Asim et al., 2021). In the second stage, lignin, hemicellulose and cellulose began to degrade by breaking chemical bonds. The third stage was simultaneous carbonization and oxidation(Chen et al., 2016). The initial decomposition temperatures of windmill palm fiber treated by alkali, acetyl chloride, and acetic anhydride were 333 °C, 336 °C and 356 °C, respectively. Acetylation improved the thermal stability of the material slightly. The fastest change of TG curve was the central decomposition temperature of the material, which was 330 °C, 337 °C and 356 °C, respectively. When the temperature was 600 °C, the decomposition of the material tended to end. Furthermore, the residual carbon content was stable. The residual carbon rate was the highest, with 22.4% of the windmill palm fiber treated by alkali. While the residual carbon content of windmill palm fiber treated by acetic anhydride was the lowest of 11.4%.

Conclusions

In this paper, alkali and acetylation treatment was used to solve the defect of strong hydrophilicity of windmill palm fiber to expand its application in the filling and composite filed. Both acetyl chloride and acetic anhydride can prove the hydrophobic property of windmill palm fiber with static contact angles above 145 °. Acetylation treatment produced many pore structures on the fiber cell wall, which may reduce the mechanical properties of the fiber. When used as the reinforcing matrix, it was necessary to weigh the poor adhesion caused by hydrophilicity and the decreased strength caused by the destruction of the cell wall. After acetic anhydride treatment, the thermal stability of the fiber was increased with the initial decomposition temperature increased from 330 to 356 °C. It may be beneficial to improve the mechanical properties of hot-pressed composites. The modified windmill palm fiber can still keep low thermal conductivity, superior to cotton and wool. Windmill palm fiber had better sound absorption performance because of its high hollowness and multi-scale pore structure optimized by acetyl chloride. The average sound absorption coefficient was up to 0.47 for the unique structure dissipating more energy.

Declarations

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Figures

Figure 1

Windmill palm short fiber with different surface treatment: a alkali treated windmill palm fiber, b acetyl chloride treated windmill palm fiber, c 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

Figure 3

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

Figure 4

Thermal insulation performance of different windmill palm fiber velvet

Figure 5

Thermal properties of palm fiber before and after surface modification.