

The Effects of Heating Treatment on the Tensile Properties of Palm Petioles Fiber

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Palm petioles fibers (PPF) are used widely in China to make mattresses. The changes of the properties of PPF after heat treatment is an important factor that should be considered. This study focuses on the fiber tensile test after heat treatment under different temperatures and at different times. As the temperature of treatment was increased, the Young's modulus of PPF increased, while the breaking strength and elongation declined. The turning point of the tensile properties was 160 °C. The heating time also had a significant influence on the tensile properties. As the heating time increased, the Young's modulus grew, while the breaking strength and elongation declined.

Keywords: Heat treatment; Palm petioles fiber; Tensile properties

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INTRODUCTION

Palm fibers (PFs) are stripped from windmill palm trees, which are widely distributed in southern China, especially in the Yungui Plateau. Palm trees are tall and straight, with a beautiful shape. As evergreens, palms are used as ornamental trees (Fig. 1 (a)). From an economic perspective, PFs are important because the trunk can be made into appliances, leaves can be made into fans or hats, and roots can be made into medicine (Li *et al.* 2013).

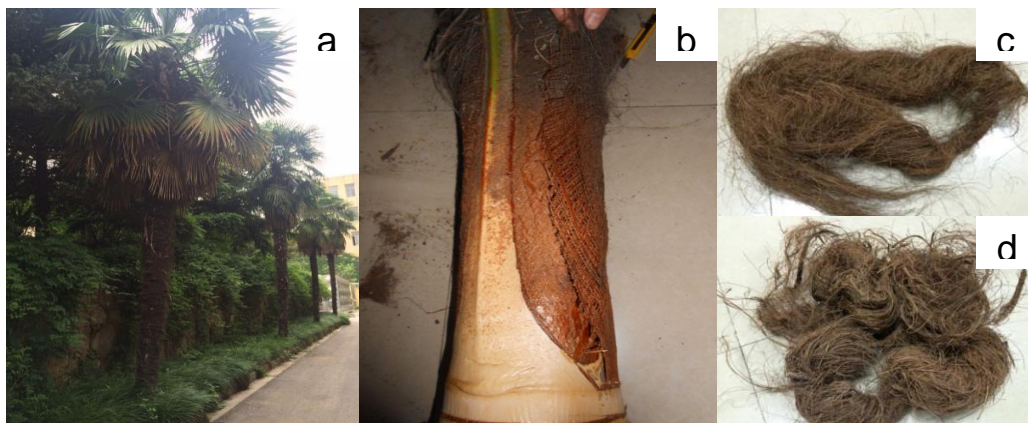


Fig. 1. (a) Palm tree, (b) sheath (palm sheet and petioles), (c) palm leaf sheath fiber, and (d) palm petioles fiber

Palm fibers are divided into palm leaf sheath fibers (PLSF) and palm petioles fibers (PPF), depending on the position of the extraction. The PLSF are extracted from the leaf sheath sheet, while the PPF are extracted from palm petioles (Li *et al.* 2013; Liu *et al.* 2014).

The windmill palm trunk stands upright with cylindrical shape, and the lower part of the trunk is packed by leaf sheath fiber with cross-mesh arrangements (Fig. 1 (b)). The PLSF (Fig. 1 (c)) is dark brown with a smooth and shiny surface. Its surface is supple and elastic, similar to hair. The PLSF does not contain sugar and tannin, and it is the traditional palm fiber. However, the output of PLSF is small, so the price is high. Palm petioles fibers are light brown, and the material feels rough and similar to hay (Fig. 1 (d)). The output of PPF is high, which makes the price low. As a result, processed PPFs are often used to replace PLSFs (Liu *et al.* 2015).

The PPF is composed of cellulose (38.9%), hemicellulose (13.8%), lignin (28.8%), extractives (0.96%), and ash (2.56%) (Liu 2016). The PLSF has a similar composition, but the lignin content is 10% higher than in PPF. PPF is flexible, slender, durable, and has good elasticity because it uses adhesive glue to form a reticular structure. This makes PPF a high-quality material to make fiber composite material (Lu *et al.* 2005; Zhang 2010).

With a certain strength and flexibility, brown fiber composite materials can be used as a mattress material, cushioning material, and packaging material. Hot press forming and high temperature curing processes are used to make composite materials from PPF; molding and injection molding are the most commonly used technologies. According to the different types of adhesives, the heating temperature ranges from 100 °C to 170 °C. Palm fiber composite material is light and has a certain impact resistance. It is an alternative for car components, especially the exterior part of the car, which reduces the dependence on man-made glass fiber material and, consequently, decreases pollution and carbon emissions. Typical compression molding and injection molding occurs between 170 °C and 180 °C. High temperature stability and flammability must be considered when using brown fiber composite materials for applications. In this article, the tensile properties of PPF were tested after high temperature treatment. These experiments provided basic data for applications of PPF.

EXPERIMENTAL

Materials

Raw PPF was provided by DaZiRan Science & Technology Co., Ltd. (Guiyang, China). The initial moisture content of the material was 12% to 13%. The selected PPF had no joints or defects. The fineness was 100 to 120 tex, and the length was over 150 mm.

Methods

To make palm fiber composites, the heating temperature was set between 100 °C and 200 °C at 20 °C intervals, using six settings. The heating time was 3 h, 6 h, or 9 h (Du *et al.* 2008). The material was stored for over 24 h at a constant temperature (25 °C) and humidity (50%) to reach the average moisture content so as to reduce the amount of error.

Tensile Test

Specimens were tested on a Material university testing machine (SHIMADZU AGS-X20KN, Kyoto, Japan). The area of fracture of the tested specimen was between the

grippers and at least 5 mm away from fixture. It was ensured that the part of fracture on the test specimen was between the grippers; otherwise, the data was judged to be invalid. Thirty tested specimens were prepared, with the aim of obtaining 10 valid data points. Latex was put on the clamp surfaces to avoid slipping. Each fiber specimen was fixed on the central line of the clamping device at a distance of 50 mm. The speed of machine was 20 mm/min.

Thermogravimetric Analysis

The specimen was broken into pieces and dried at 103 °C (Liu *et al.* 2016). Samples were tested on a thermogravimetric machine, also known as a microcomputer thermal balance machine (NETZSCH TG209F3, Bavaria, Germany). Nitrogen was used as the protective gas, and alumina was used as reference material. The method of dynamic temperature was also used in the test. The test started at room temperature, and the temperature increased at 20 °C/min to a maximum temperature of 600 °C. (Liu. 2010)

RESULTS AND DISCUSSION

Changes in the Number and Color of the Fibers After Heat Treatment

Table 1 shows that with the increase of heating temperature and time, there was more loss on the number of PPF specimen. To obtain 10 valid data points, more PPF specimens were needed to finish the tensile test. When the heating temperature was over 180 °C, the fiber specimen was easily broken. Over 200 °C, the PPF was utterly carbonized, and it immediately broke when touched.

The color of fiber changed after heating treatment, especially as the temperature rose. From 120 °C to 140 °C, the color was almost the same as the original. The color was darker when the sample reached 160 °C, and the surface of part fiber appeared nut-brown. At 180 °C, the majority of the surface of the tested fiber was nut-brown. When the temperature was 200 °C, the fiber became sepia, and it broke as soon as it was pressed. When the heating temperature was constant, the color became darker with increased heating time.

Table 1. Number of Tensile Test PPF Specimens and Equilibrium Moisture Content

Temperature (°C)	Time (h)					
	3		6		9	
	Number of Specimens	Moisture Content (%)	Number of Specimens	Moisture Content (%)	Number of Specimens	Moisture Content (%)
100	11	9.77	14	9.07	20	9.76
120	15	9.55	16	9.63	21	9.15
140	15	8.72	17	8.54	19	8.23
160	16	8.35	16	7.91	20	7.58
180	17	7.09	19	7.44	25	7.41
200	-	7.53	-	7.96	-	6.87

Changes in Average Moisture Content of the Fiber Specimen after Heating Treatment

After heat treatment, the fibers were stored for over 24 h under a constant temperature (25 °C) and constant humidity (50%). The results are shown in Table 1. When the temperature was 200 °C, the fiber was carbonized, and it broke as well as it was pressed. As a result, when it was 200 °C, the number of specimens was zero. The moisture content was different for each heating temperature and time. In general, the average moisture content declined with increasing heating temperature and time. However, there were some exceptions. The most important component of PPF was hemicellulose, which was the most sensitive and most easily influenced by environmental conditions. In high temperatures, hemicellulose aggregates into water-insoluble polymers, which reduces the fiber hygroscopicity.

The mechanical properties of PPF were influenced by changes in moisture content. When the heating temperature was under 140 °C, the moisture content of fiber was above 8%; when the heating temperature was over 160 °C, the moisture content of fiber was under 8%. This result was consistent with the color changes. Fiber was darker and part of the fiber surface became light brown or nut-brown at 160 °C. Also, the moisture absorption ability of hemicellulose in palm fiber decreased apparently.

Thermogravimetric Analysis of PPF

In the thermal weight curve of the PPF (Fig. 2), when the heating temperature of increased, the weight gradually declined in four stages.

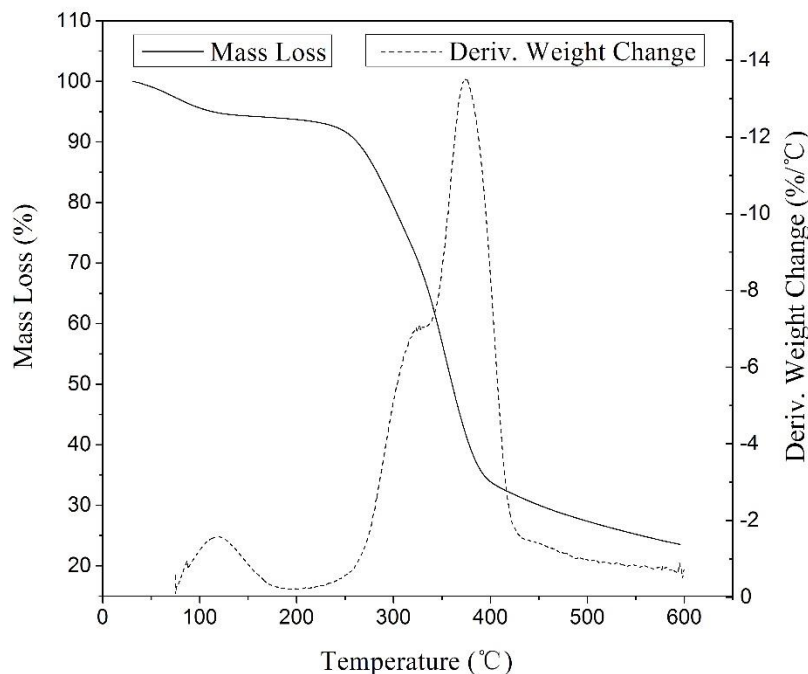


Fig. 2. TG curve of palm petioles fiber

First, from room temperature to 100 °C, the weight declined, and fiber lost its moisture. Second, from 100 °C to 250 °C, the weight declined by a small amount. Third, when the temperature was 250 °C to 350 °C, the weight of the fiber specimen declined

greatly. At high temperature, the main components of the fiber, lignin and hemicellulose, were pyrolyzed. After that, the weight declined gradually. The weight loss rate of PPF was at its largest under 75 °C, and then it recovered. Its rate suddenly increased from 250 °C to 350 °C; the peak value was 14 %/min. This was also the same as the stage of fiber's quick loss of weight. After that, the rate increased, and when the temperature was over 380 °C, the data stayed constant.

Test Results of Tensile Properties of Fiber Specimens after Heat Treatment

As shown in Table 2, changes in heating temperature and heating time resulted in changes in the tensile properties of PPF. In general, with increasing heating temperature, Young's modulus was larger, while the tensile strength and elongation were smaller. As shown in Table 3, both heating temperature and heating time played an influential role in every index, but heating temperature had a large influence on the indexes.

Table 2. Tensile Properties of Palm Petioles Fiber after Heating

Temperature (°C)	Time (h)	Young's Modulus		Breaking Strength		Elongation	
		Average Value (MPa)	COV (%)	Average Value (MPa)	COV (%)	Average Value (%)	COV (%)
100	3	1030.45	33.05	73.28	35.29	8.33	19.36
	6	1071.32	55.27	79.8	17.08	9.33	25.02
	9	828.76	20.28	64.18	27.02	9.92	31
120	3	377.68	29.77	79.11	18.44	8.42	24.49
	6	990.62	40	76.31	26.49	8.5	22.82
	9	905.39	38.13	78.95	19.38	8.08	19.36
140	3	1089.17	23.59	76.15	22.48	8.08	35.16
	6	988.61	31.64	69.23	13.11	8.33	17.08
	9	1674.56	18.69	62.88	32.71	7.33	24.6
160	3	2438.57	34.22	91.69	37.8	5.5	29.55
	6	1987.68	20.28	48.88	21.68	2.75	31.47
	9	1269.32	16.86	60.38	31	5.67	24.49
180	3	997.01	22.11	33.3	18.44	3.26	24.81
	6	2179.6	18.71	29.83	20.08	2	25.02
	9	1063.28	27.02	22.38	17.93	2	29.02
200	3	-	-	-	-	-	-
	6	-	-	-	-	-	-
	9	-	-	-	-	-	-

Table 3. ANOVA for Effects of Heating Temperature and Time

Influence Factor	P value		
	Young's Modulus (MPa)	Breaking Strength (MPa)	Elongation (%)
Heating temperature	4.36446E-20	2.59E-07	0.0003
Heating time	0.0101	0.0001	0.0028

Note: 5% is the significance level, $p < 0.05$, shows the influence of factors affecting significant differences in index, $p > 0.05$, it indicates that factors influencing the difference indicator value was not significant

Effect of Heating Temperature on Tensile Properties of Palm Fiber

Effect on Young's modulus

With increasing heating temperature, the Young's modulus of PPF generally increased (Fig. 3). When the temperature was between 100 °C to 160 °C, there were no dramatic changes. However, when the temperature was between 160 °C to 180 °C, the modulus of elasticity suddenly changed. The change of unit temperature of Young's modulus changed most when the temperature was at this period. Especially when heated for six hours, Young's modulus increased by 260%, which was at its mutation maximum point.

Young's modulus is a physical quantity that describes the ability of a solid material to resist deformation. When Young's modulus suddenly increases, it indicates that the deformation resistance ability of PPF is greatly increased. At a high temperature, the cellulose of the PPF had a poor ability to resist heat. Lignin also began softening.

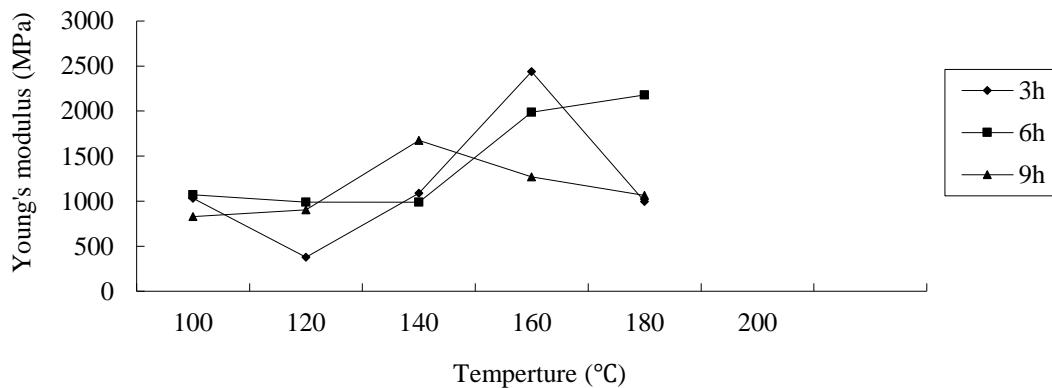


Fig. 3. Heating temperature and Young's modulus of palm petioles fiber

Effect on tensile strength

As the heating temperature was increased, the tensile strength of PPF decreased (Fig. 4). However, the changes in breaking strength were not the same in different heating times. There was a great change in breaking strength above 160 °C.

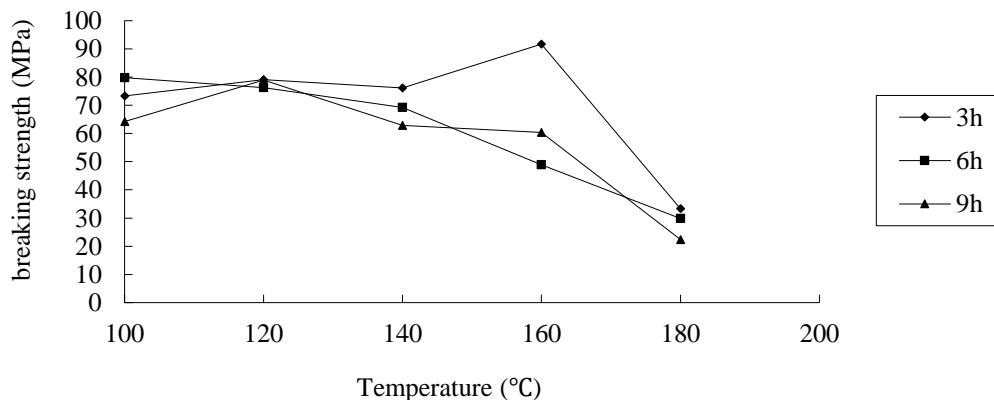


Fig. 4. Heating temperature and breaking strength of palm petioles fiber

Heating temperature had some impacts on the pyrolysis of cellulose. When the temperature was 100 °C to 160 °C, the pyrolysis of cellulose was not evident, as the tensile strength was stable. When the temperature was over 160 °C, the molecular chain of cellulose broke. Also, the crystal structure was obviously damaged, which led to the decline in the degree of polymerization and adhesion effect. Lignin began to soften at 160 °C, so the degree of polymerization also declined. When the temperature was over 160 °C, the breaking strength declined greatly.

Effect on elongation

Elongation is displacement of the specimen in the rupture value and original length ratio, which can intuitively show the tensile capability of the fiber. Materials capable of extending the ratio by more than 5% are known as plastic materials, while those that break with less than 5% extension are known as brittle materials. For plastic materials, the tensile strength is almost the same as the compressive strength. For brittle materials, the tensile strength is smaller than the compressive strength.

As shown in Fig. 5, with increasing temperature, the elongation of PPF declined. When the temperature was lower than 160 °C, maybe between 140 °C and 150 °C, PPF reached its critical value and began to change to a brittle material. When the temperature was 180 °C, it had exceeded the softening temperature of lignin. Under such conditions, the fibers were easy to separate, and the effect of hard solid was very small. Meanwhile, the degree of polymerization of cellulose decreased greatly at high temperatures, and the adhesion effect declined. As a result, the elongation of PPF decreased.

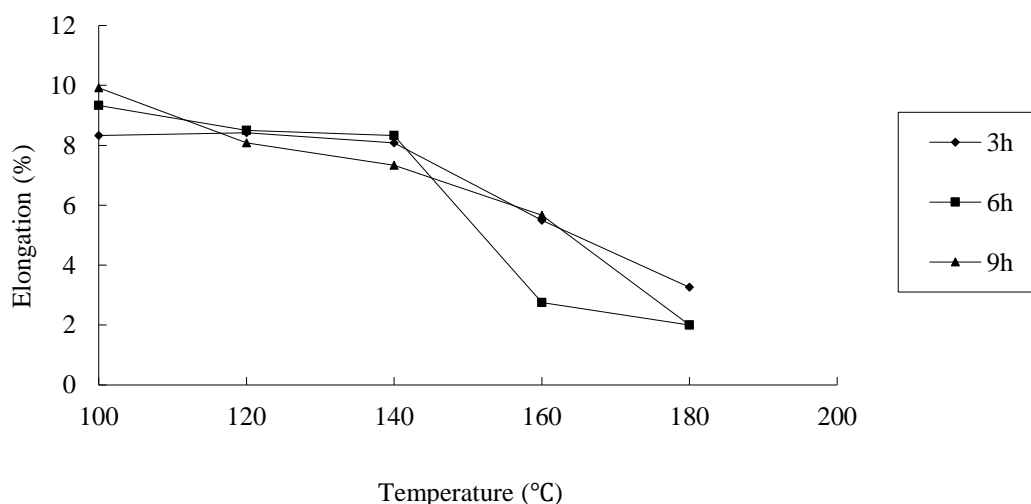


Fig. 5. Heating temperature and elongation after break of palm petioles fiber

Effect of Heating Time on Tensile Properties of PPF

Effect of heating time on elastic modulus of PPF

In general, when the heating time increased, the Young's modulus of PPF increased gradually. When the temperature was under 160 °C, Young's modulus changed a little, and heating time had an irregular effect on PPF's ability to resist deformation when it was under 160 °C. When the temperature was over 160 °C, with the increase of heating time, the

cellulose, hemicellulose, and lignin of PPF were heated by the high temperature, and the change of the interior was gradually changing. When the temperature was under 160 °C, the influence of heating time was not obvious. When the temperature was over 160 °C, the fiber distinctly changed to a darker “carbon-like” color. The longer the heating time was, the weaker the anti-deformation ability was, and it gradually lost its ductility. Also, the Young's modulus was increased greatly.

Effect of heating time on tensile strength of PPF

With changes in time, the tensile strength slightly changed, with an overall decline. After a long time heating under high temperatures, the fibers were easy to separate, and the crystal structure was obviously damaged. The degree of polymerization of cellulose decreased greatly at high temperatures, and adhesion effect declined. As a result, the tensile strength declined.

Effect of heating time on elongation of PPT

With increased heating time, elongation was declining, especially when the temperature was over 160 °C. According to thermal gravimetric analysis, with the increase rate of temperature being 20 °C/min for 100 °C to 200 °C, the weight loss was very small. However, the mechanical properties of the fibers had a relatively large change at a long time and high temperature, which demonstrated that heating time did have a great effect on mechanical properties of fiber.

CONCLUSIONS

1. With the increase of heating temperature, the color of PPF changed gradually. When the heating temperature was 120 °C to 140 °C, the color of PPF was almost the same as in normal temperature. When it was at 160 °C, the color became darker, and part of the fiber appeared dark brown. When it was at 180 °C, most of the fiber appeared dark brown. When the temperature was 200 °C, the fiber was sepia and it broke as well as it was pressed. When the temperature remained the same, the longer the heating time was, the darker the color was.
2. According to the thermogravimetric experiment, when the treatment temperature rose, the weight of PPF decreased. When the temperature rose from room temperature to 100 °C, the weight declined. The weight was stable when the temperature was 100 °C to 250 °C, and when it was 250 °C to 350 °C, the weight decreased dramatically. After 350 °C, the weight decreased slowly.
3. The heating temperature had great impacts on the tensile properties of PPF. With the increase of heating temperature, Young's modulus of PPF increased overall. When the temperature reached 160 °C, Young's modulus suddenly increased. With the increase of heating time, the tensile strength of PPF declined, and 160 °C was the turning point for changes to tensile strength of the palm fiber. With the increase of heating temperature, the elongation of PPF declined. After heating at 160 °C, the critical value was close to 5% and after that, the material started the qualitative change from plastic material to brittle material.

4. Heating time also had great impacts on tensile properties. With increased heating time, Young's modulus increased, while the tensile strength and elongation declined.
5. The turning point of heating temperature for both PPF and PLSF was 160 °C; however in total, the tensile properties of PPF began to change below 160 °C while PLSF changed above 160 °C. The heat resistance of PLSF was greater than that of PPF.

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