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1 **Interfacial properties of windmill palm fiber reinforced laminated veneer lumber (LVL)**
2 **under high voltage electrostatic field (HVEF)**

3

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11

12 **Abstract:** The acid / alkali pretreatment of windmill palm fiber (WPF) and the application of
13 high voltage electrostatic force (HVEF) technology were used to prepare WPF-reinforced
14 laminated veneer lumber (LVL). The effects of acid / alkali pretreatment on the surface
15 morphology, chemical composition, crystallinity and mechanical properties of WPF were
16 investigated. Moreover, the effects of intensity of HVEF on chemical composition and
17 wettability of WPF were analyzed. Based on the mechanical experiment of WPF reinforced
18 LVL composites, the mechanism of acid / alkali pretreatment and HVEF for the reinforcement
19 of the composite was systematically clarified. The results showed alkali treatment efficiently
20 removed silica particles and cuticles on the fiber surface and produced windmill palm fiber

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21 (ALWPF) with relatively high Young's modulus and tensile strength (increased by 167.07%
22 and 26.52%, respectively). After HVEF treatment, the increase of surface-active groups (- OH
23 and - CHO) led to the increase of surface oxidation degree and water contact angle of WPF.
24 Under the synergistic effect of acid / alkali pretreatment and HVEF, the interfacial properties
25 between the adhesive and WPF were improved, which led to the effective improvement of the
26 mechanical properties of WPF reinforced LVL. This study showed that the synergistic effect
27 of acid / alkali pretreatment and HVEF not only broaden the application scope of WPF, but
28 also provide insights into the engineering of LVL production.

29

30 **Keywords:** Windmill palm fiber, Acid / Alkali pretreatment, High voltage electrostatic field,
31 Interfacial properties, Laminated veneer lumber composites

32

33 **1.Introduction**

34 In recent years, laminated veneer lumber (LVL) produced from fast-growing species for
35 building materials has attracted increasing attention and research [1]. LVL not only has most
36 of the properties of natural wood, but also has many excellent properties including high
37 strength and toughness and good stability [2]. LVL has a wide range of applications, in the
38 fields of non-structural bearing (including carriage board, furniture, floor, packaging materials)
39 and structural bearing (including timber structure keel, room beam) [3]. However, the
40 mechanical properties of LVL based on fast-growing wood are limited by recurring
41 limitations such as low strength, poor toughness and dimensional stability [4]. Therefore, the
42 production of LVLs from fast-growing woods has become a research hot spot in the wood

43 industry in order to expand their applicability in the field of load-bearing structures [5].

44 The reinforcement based on wood has become an important way to improve the
45 comprehensive utilization rate of wood resources and the quality grade of wood products [6].

46 Currently, the most widely used composite materials are fiber-reinforced composites, which
47 have excellent bending resistance and shear resistance, especially in the vicinity of the
48 bending load [7]. Natural fiber is very attractive to structural materials, such as panel or wall
49 materials, because natural fiber has low density, environment-friendly, low cost and easy
50 availability, which can reduce construction time and environment pollution [8]. The natural
51 fiber reinforcement materials include hemp, flax, bamboo fiber, coconut fiber, etc [9]. Flax
52 fiber was widely used in car seats and door trims. Compared with the door trim made of
53 traditional long fiber reinforced thermoplastic composites (LFT) and acrylonitrile butadiene
54 styrene (ABS) plastics, the weight of the door trim made of natural fiber reinforced
55 composites can be reduced by more than 20% [10]. The composites of flax and hemp with
56 acrylated epoxidized soybean oil (AESO) can meet the strength requirements of housing
57 applications, such as roof / wall panels and construction wood [11]. The ceiling made of hemp
58 fiber and semi-rigid polyurethane elastomer has the advantages of simple process, higher
59 strength and weight reduction of more than 10% [12]. The properties of composites depend on
60 the properties of components and the interfacial compatibility between components [13].

61 Although natural fiber has good advantages, its main disadvantage is the poor interfacial
62 performance between natural fiber and matrix, which leads to the insufficient interfacial force
63 between the fiber and resin, the poor bonding interface performance, and greatly reduces the
64 overall performance of fiber reinforced composites [14]. Therefore, it is urgent to explore new

65 fiber reinforced materials and efficient surface modification to improve the interfacial
66 bonding properties between fiber and wood, so as to improve the mechanical properties of
67 fiber reinforced wood composites.

68 Researchers have also been interested in wind palm fiber (WPF) in recent years [15]. The
69 study on the mechanical properties of WPF shows showed that the breaking strength of WPF
70 is higher, the fiber is longer, the linear density is much smaller than that of sisal fiber, the
71 elongation at break is larger, and the initial modulus is close to that of pineapple leaf fiber
72 [16]. In a single palm fiber, the fiber cells are closely arranged, the non-fiber cell content was
73 relatively low [17]. WPF has good mechanical properties, and has been used as reinforcement
74 in the fiber reinforced composites [18]. Surface morphology affects the adhesion between
75 fiber and polymer, and surface modification can improve the application range of materials
76 [19]. The mechanical properties of composites are largely determined by the adhesion
77 between resin and fiber. In most cases, there is a regular arrangement of silica on the surface
78 of the palm fibers. Tomlinson [20] proposed that silica in *Palmae* can be divided into two
79 types: one is conical or cap shaped with parenchymatous basal cells, the other is rough or
80 spiny spheroid with thick cell wall. A large number of hydrophobic substances are also
81 present on the fiber surface, such as cerolipoid [21]. This complex surface composition of
82 WPF improves their thermal and chemical stability but also reduces the adhesion between the
83 fiber and the resin and limits their broad use. In the literature, it was found that the silica on
84 the surface of jujube palm fibers could be peeled off by low concentration of alkali and acid
85 solution, leaving annular holes [22]. Shanmugasundaram, N. et al. [23] showed that the
86 impurities on the palm petiole fiber surface were gradually removed using an increasing

87 NaOH concentration of NaOH. For a concentration of 15%, the cell wall of the fiber surface
88 was damaged and micro holes appeared, which increased the surface roughness and the
89 adhesion between the fiber and the polymer. The above surface modification methods have a
90 positive effect on the interfacial adhesion.

91 High voltage electric field (HVEF, > 1 kV) is used in the process of material preparation
92 and modification as a treatment method which has a unique impact on the physical and
93 chemical properties of materials [24]. Under the action of electric field, the excited free
94 electrons collide with the chemical groups on the material surface, resulting in the formation
95 of broken chemical bonds and new free electrons [25]. As a result, the polarization degree and
96 surface free energy of the material surface are significantly improved. Kilic [26] measured the
97 surface potential of the fiber material in the electric field. The results show that the surface
98 energy of the fiber material increases and the resistivity decreases after the electric field
99 treatment. The physical and chemical properties of biomass materials, adhesives and their
100 bonding interface will be affected by HVEF. It is found that biomass materials can produce
101 weak ionization by the action of electric field, which is caused by the movement of ions [27,
102 28]. In direct current (DC) electric field, the polarization phenomenon of biomass materials
103 has the typical characteristics of ion ionization phenomenon [29]. Therefore, the application
104 of HVEF in the hot-pressing process of the composite can increase the number of free
105 electrons and chemical bonds between the material and the adhesive, which provides more
106 chemical reaction sites to increase the conditions for the reaction between the material and the
107 adhesive.

108 In this study, a combination of acid / alkali pretreatment and HVEF was used to improve

109 the surface properties of windmill palm fiber (WPF). The surface morphology, composition
110 and properties of the treated WPF were examined in detail. The bond strength test of
111 WPF-reinforced phenol-formaldehyde glue-based laminated veneer wood composites verified
112 the enhancing effect of the treatment. The mechanical test of the composite was further
113 carried out.

114

115 **2. Materials and Experiments**

116 2.1 Materials

117 The dimensions of 200 mm × 200 mm × 2.5 mm masson pine (*Pinus massoniana Lamb.*)
118 veneers and phenol formaldehyde (PF) adhesive were provided from Lianyungang Nanfang
119 Wood Industry Co., Ltd. (Jiangsu Province, P.R. China). In order to improve the bonding
120 properties of wood veneer composites, the moisture content of masson pine veneers was
121 adjusted at $7 \pm 2\%$ by oven-dried at 50 °C for 24 h, the average crack degree of masson pine
122 veneer was 35%, and the density was 0.475g / cm³. The PF adhesive of formaldehyde (F) /
123 phenol (P) ratio was 2, the solid content of PF adhesive was $45 \pm 2\%$, the viscosity was 132
124 mPa·s at room temperature, the density was 1.205 g / ml, and the pH was 13.5. Commercial
125 WPF (*Trachycarpus fortunei*) fabric (1500 mm × 800 mm) was provided by Rugao Shuangma
126 Chemical Co., Ltd. (Jiangsu Province, P.R. China). The consumption of woven WPF was 500
127 g/m², and it was woven using the "plain-weave" type of knitting. Sulfuric acid (98% solution),
128 nitric acid (98% solution), sodium hydroxide, benzene and ethanol (95% solution) were
129 produced by Nanjing Chemical Reagent Co., Ltd. (Jiangsu Province, P.R. China). The
130 aluminum plates (300 mm × 300 mm × 1.5 mm) as electrode plates were provided from

131 Zhengguang metal material Co., Ltd. (Shandong Province, P.R. China.). A high voltage
132 electrostatic generator (DISK-120K) was produced by Zhaomao coating Accessories Co., Ltd.
133 (Guangdong Province, P. R. China).

134

135 2.2 Windmill palm fiber (WPF) pretreatment

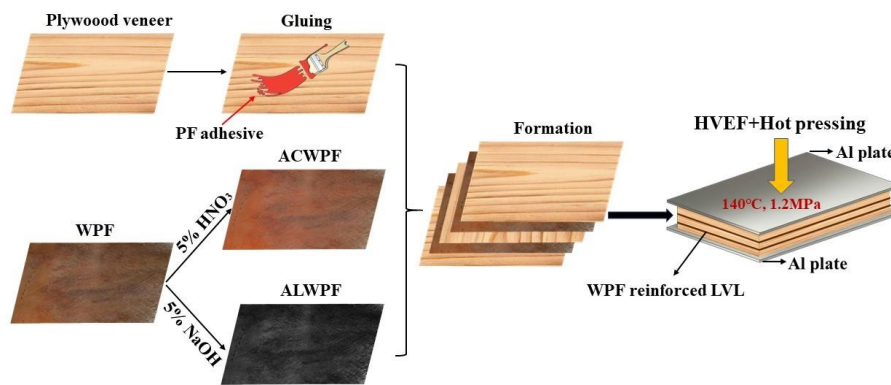
136 In this experiment, alkali treatment and acid treatment were used to pretreat the windmill
137 palm fibers. Two portions of windmill palm fibers with the same weight were put them in a
138 500ml beaker, and then added with 5 % sodium hydroxide solution and 5 % nitric acid
139 solution respectively. The beakers were treated in a 70 °C water bath for 2 hours. After water
140 bath treatment, the pretreated samples were taken out, cooled down, washed to neutral with
141 distilled water, dried in oven at 70 °C for 12 hours. At last, the alkali treated windmill palm
142 fiber (ALWPF) and acid treated windmill palm fiber (ACWPF) were prepared.

143

144 2.3 Preparation of windmill palm fiber reinforced laminated veneer lumber composites

145 The WPF reinforced plywood composites were prepared under different treatments of WPF
146 and different high voltage electrostatic field (HVEF) strength to study the effect of treatment
147 methods on the interfacial properties of plywood. The three-layer WPF reinforced composites
148 were assembled by double-sided gluing process, and the gluing amount of PF adhesive was
149 150g / m². The composites were put into a hot press with high-voltage electrostatic device for
150 hot pressing. The high voltage electrostatic device contained two aluminum electrode plates,
151 one of which was connected to the high voltage electrostatic generator, the other was
152 connected to the ground, and the composites were placed between the two plates. The

153 hot-pressing parameters of plywood were as follows: the temperature was 140 °C, the
 154 pressure was 1.2 MPa, the hot-pressing time was 1.5min/mm, HVEF treatment time was the
 155 same as hot pressing time, and the electrostatic generator was stopped immediately after
 156 reaching the treatment time. The plywood assembly method and hot-pressing process with
 157 HVEF were shown in **Fig. 1**. The reinforced materials contained raw windmill palm fiber
 158 (RWPF), ALWPF and ACWPF. The parameters of HVEF included 0 kv, 20 kV, 40 kV, 60 kV.
 159 Each experiment was repeated 10 times.



160
 161 **Figure 1** Schematic diagram of preparation of WPF reinforced LVL
 162

163 2.4 Characterization methods

164 The structure of the material was analyzed by Fourier transform infrared spectroscopy
 165 (FTIR, TENSOR-27, Bruker, Germany) in this experiment. The samples were ground into
 166 powder and screened with 100 screens. Then, the powder was mixed with potassium bromide
 167 (KBr) and pressed tablets. The wavenumber scanning range was 4000 - 400cm⁻¹, and the
 168 resolution was 1cm⁻¹. Environmental scanning electron microscopy (ESEM, JSM-7800F,
 169 JEOL, Japan) was used to observe the surface morphology of the samples. The samples were
 170 fixed on the stage with conductive adhesive and plated with gold. Then, the samples were

171 placed in the ESEM, and the accelerating voltage was set at 10-15kV. The ESEM was
172 equipped with X-ray energy dispersive spectrometer (EDS, Inca X-Max, Oxford, UK), which
173 can be used for analysis chemical composition of samples. The acceleration voltage of EDS
174 was 20kV. X-ray photoelectron spectroscopy (XPS, AXIS-HSi, Kratos, Japan) was used to
175 analyze the chemical states of the elements in the samples. The pure magnesium $K\alpha$ was used
176 as the X-ray source. The vacuum degree of XPS was lower than 1.33×10^{-10} Pa. The X-ray
177 power was 150W, the narrow sweep energy was 20 eV, the step size was 0.1eV, and the
178 number of scans was 3 times. The internal standard method was used to eliminate the
179 charging effect. The binding energy C1s (284.8ev) of the hydrocarbon was used for energy
180 correction. The samples were analyzed by X-ray powder diffraction (XRD, XD-3, Beijing,
181 China). The experimental conditions were: the pure copper $K\alpha$ radiation (k) was 1.5418 Å, the
182 acceleration voltage was 36 kV, the current was 20 mA, the scanning step size was 0.02 °,
183 scanning ranged from 5 ° to 90 °, the scanning speed was 2 ° / min. The crystallinity of the
184 samples can be determined quickly by the empirical crystallinity index [30].

185

186 2.5 Chemical composition analysis of windmill palm fiber

187 The cellulose, hemicellulose and lignin of raw materials were determined by NREL method
188 [31]. The dried sample (300mg) which had been extracted by benzene alcohol mixture
189 (benzene / ethanol (95%) ratio of 2 / 1, Soxhlet extraction) for 6h was put into a 100mL bottle.
190 Three groups of parallel samples were prepared. 72% concentration of sulfuric acid (3mL)
191 was evenly added to the bottle. Then the bottle was put in a 30 °C water bath for 1h, and kept
192 shaking during the reaction. The bottle was take out, and added 84mL distilled water. The

193 tightly capped bottle was putted into the autoclave at 121 °C for 1 h. After the acid hydrolysis,
194 the mixture was separated by filtration. The absorption value of the diluted liquid was
195 determined by UV spectrophotometer at 205nm, and the absorption value was kept between
196 0.7 and 1.0. At the same time, the pH of the filtrate was adjusted to 1-3, and the contents of
197 glucose, xylose, arabinose and other monosaccharides were determined by HPLC. The solid
198 was washed with distilled water and dried for 24 h at 105 °C. The calculation formulas were
199 as follows:

200 Calculation formula of acid-soluble lignin (ASL) content

$$201 \quad ASL = \frac{Dilution \ factor \times Absorption \ value \times Filtrate \ volume}{Absorption \ coefficient(110) \times Dried \ sample} \times 100\% \quad (1)$$

202 Calculation formula of acid-insoluble lignin (AIL) content

$$203 \quad AIL = \frac{Dried \ residual}{Dried \ sample} \times 100\% \quad (2)$$

204 Calculation formula of cellulose content

$$205 \quad Cellulose = \frac{Glucose \ concentration \times 0.9 \times Filtrate \ volume}{Dried \ sample} \times 100\% \quad (3)$$

206 Calculation formula of hemicellulose content

$$207 \quad Hemicellulose = \frac{Xylose \ arabinose \ concentrations \times 0.88 \times Filtrate \ volume}{Dried \ sample} \times 100\% \quad (4)$$

208

209 2.6 Fineness measurement of windmill palm fiber

210 Fiber fineness referred to the degree of fiber thickness, which was usually measured by
211 linear density and physical thickness. The larger the linear density was, the thicker the fiber
212 was. The fineness of fiber was measured by the cut-middles method. Firstly, the fiber was
213 straightened, the free fiber and the fiber shorter than the test length ($L_c=10$ mm) were

214 removed, then the fiber was cut. Each experiment was repeated 5 times. The calculation
215 formula of fiber linear density was as follows:

$$216 \quad T_t = 10^3 G/nLc \quad (5)$$

217 Where G was the weight of middle fiber, n was the number of fiber, and T_t was the linear
218 density of fiber.

219

220 2.7 The mechanics property analysis of WPF

221 The mechanics property of WPF was measured by ASTM C1557-14 [32]. The stretch
222 length was set to 30 mm in this experiment. The electronic universal strength testing machine
223 (WDW3050, Jilin, China) was used to test the tensile strength of windmill palm fiber samples.
224 The tensile speed was 0.5 mm / min, and each experiment was repeated 15 times. The fiber
225 diameter was measured by optical microscope. Ten test points were taken for each fiber, and
226 the average value was taken as the fiber diameter. The cross-sectional area of the fiber was
227 calculated by using the formula of circle.

228

229 2.8 Electron spin resonance measurement

230 The electron spin resonance (ESR, EMX 10 / 12, Bruker, Germany) were used to measure
231 the content of free radicals in the samples. The sample size was $40\text{mm} \times 350 \pm 20\mu\text{m}$ (length
232 \times diameter). The sample was oven-dried at 103°C for 48h (the moisture content was less than
233 4%). Each experiment was repeated for 10 times.

234

235 2.9 Wettability property of WPF

236 The wettability properties of WPFs were characterized by the contact angle test (the
237 sessile drop method). The moisture content of WPF was controlled at 4%. The WPF was fixed
238 on the automatic video micro contact angle measuring instrument (DCAT 21, DataPhysics,
239 Germany). 3 μL distilled water was deposited on the fiber surface. The clear images can be
240 obtained by the optical imaging system. The contact angle was calculated by SAC20 software.
241 Each sample measured 10 points, and the average was the contact angle of the WPF.

242

243 2.10 Mechanical properties and digital image correlation

244 The mechanical properties of WPF reinforced plywood were tested by electronic universal
245 strength testing machine (CMT 4304, Shenzhen, China). The modulus of elasticity (MOE) of
246 WPF reinforced plywood was measured according to EN-310-1993 [33]. The sample size was
247 300 mm \times 50 mm (length \times width). The bonding strength (BS) was tested according to
248 EN-314-1-2004. The determination of dipping peeling performance was analyzed according
249 to the China National Standard (GB / T 19536-2015 [34]) for class I plywood. The sample
250 size was 100 mm \times 25 mm (length \times width), and the loading speed of universal strength
251 testing machine was 1 mm / min. Each condition was repeated 10 times. Digital image
252 correlation was used to record the strain of the sample during the bonding strength test. The
253 image correlation was performed using the commercial software package Correlated
254 Solutions (www.correlatedsolutions.com). After the calibration of the reference ruler, two
255 CCD cameras were used to record the experimental process according to a photo every 0.1
256 second, and the resolution was 30 μm .

257

258 **3. Results and Discussion**

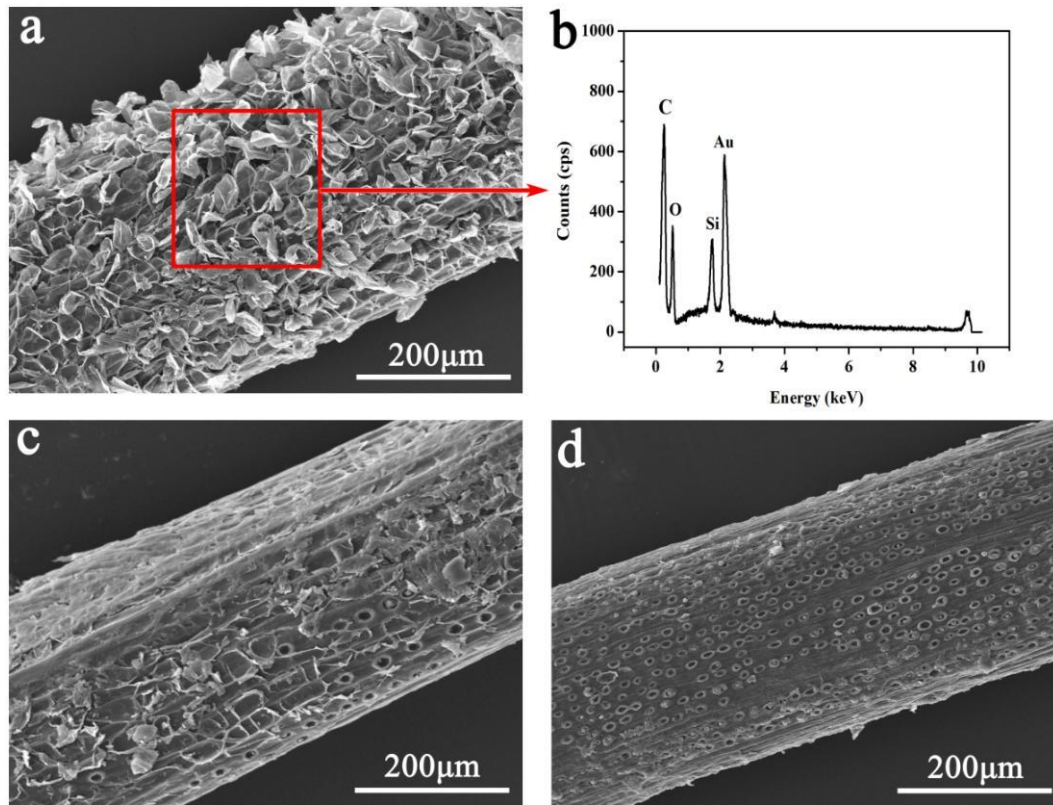
259 *3.1 Characterization of acid / alkali pre-treated windmill palm fiber*

260 *3.1.1 Morphology of pre-treated windmill palm fiber*

261 The surface of WPF (**Fig. 2a**) is unsmooth and has a fish scale morphology. Previous
262 studies have shown that these "fish scale" substances were mainly composed of silica [19]
263 which was supported by the results of microanalysis (EDS analysis) shown in **Fig. 2b**. After
264 acid treatment, the silica component of the ACWPF surface (**Fig. 2c**) was partially removed,
265 and the longitudinal gullies were exposed on part of the fiber. The silica fell off from the holes,
266 and most of it was also scattered on the surface of the palm fiber. As shown in **Fig. 2d**, the
267 longitudinal gullies of palm fibers were deepened after alkali treatment, which suggested that
268 palm fibers were bundles, composed of several single fibers. Most of silica fell with the lignin,
269 pectin and other substances, and a small amount of residual silica was dispersed on the fiber
270 surface, which caused the fibers to be exposed, thus promoting the surface performance of
271 palm fiber, exposing the active hydroxyl groups, and improving the wetting performance of
272 the fiber [18]. The surface of the treated fibers revealed micro holes, as shown in **Fig. 2c** and
273 **2d**.

274 **Figure 2** Morphology of different windmill palm fiber. (a) RWPF. (b) microanalysis spectra
275 (EDS analysis) of RWPF. (c) ACWPF. (d) ALWPF.

276 **Figure 2** Morphology of different windmill palm fiber. (a) RWPF. (b) microanalysis spectra
277 (EDS analysis) of RWPF. (c) ACWPF. (d) ALWPF.



278

279 **Figure 2** Morphology of different windmill palm fiber. (a) RWPF. (b) microanalysis spectra

280

(EDS analysis) of RWPF. (c) ACWPF. (d) ALWPF.

281

At the same time, the hydrophilicity of the fiber was improved by the removal of hydrophobic substances such as silica [19]. The change of surface properties also meant that the surface properties of the fiber were improved when it was used in composites. The silica removal was confirmed by Elemental Analysis (**Table 1**). The Si% decreased from 3.86% for RWPF to 0.97% and 0.07% for ACPF and ALPW respectively.

286

Table 1 Elemental analysis of windmill palm fiber

Elemental	C (%)	O (%)	Si (%)
RWPF	46.45	49.68	3.86
ACWPF	50.69	48.34	0.97

ALWPF	55.74	44.19	0.07
-------	-------	-------	------

287

288 *3.1.2 Chemical composition and structure of windmill palm fiber*

289 It can be seen from **Table 2** that the WPF chemical composition was strongly changed after
 290 acid / alkali treatment. After alkali treatment, the content of cellulose increased from 28.16%
 291 to 51.80%, lignin and hemicellulose decreased from 44.07% to 35.46% and from 20.60% to
 292 9.90% respectively. The content of cellulose also increased after acid treatment, and lignin
 293 decreased significantly from 44.07% to 35.46%. This indicates that lignin and hemicellulose
 294 have chemical changes by acid / alkali treatment, and the original polymer macromolecules
 295 were hydrolyzed.

296 **Table 2** Major chemical compositions of the windmill palm fiber

Composition	Cellulose	Lignin	Hemicellulose
RWPF	28.16	44.07	20.60
ACWPF	42.30	36.98	14.35
ALWPF	51.80	35.46	9.90

297

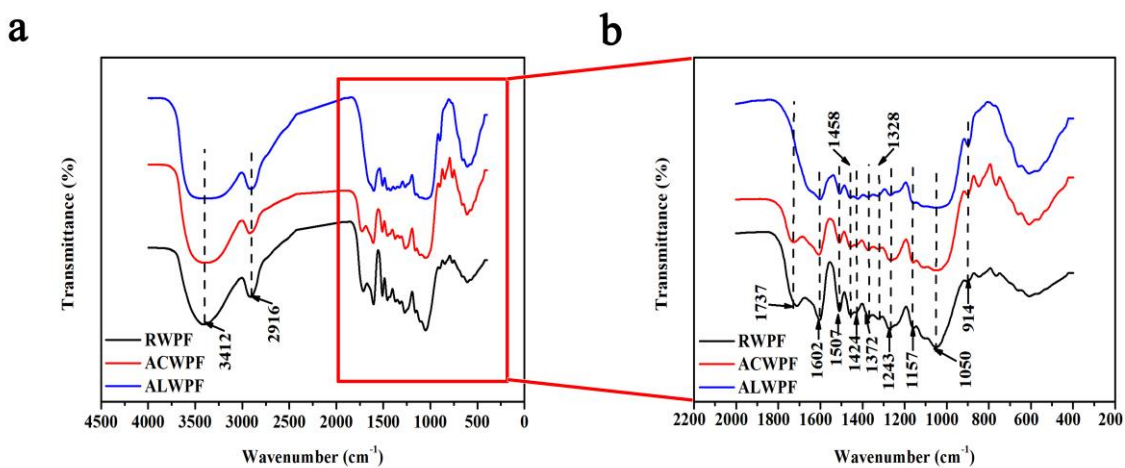
298 In order to further analyze the effect of acid / alkali treatment on the chemical composition
 299 of palm fiber, FTIR analysis was carried out. **Fig. 3** showed the FTIR spectra of WPF after
 300 different treatment. According to **Table S1**, the main functional groups on the material surface
 301 before and after pretreatment can be compared [19]. 3412 cm⁻¹ is the O-H stretching vibration
 302 peak. The peak at 1737cm⁻¹ is the characteristic absorption peak of carboxyl group in ferulic
 303 acid and p-Coumaric acid, which is the characteristic peak of hemicellulose. The absorption

304 peaks at 1602 cm^{-1} and 1507 cm^{-1} are the skeleton vibration peaks of aromatic ring, which are
305 the characteristic peaks of lignin.

306 - After acid / alkali treatment, the peak at 2916 cm^{-1} which represents the symmetric
307 stretching vibration of $-\text{CH}_2$ was obviously slowed down. They are the characteristic
308 absorption peak of cellulose, and also the characteristic absorption peak of hydrophobic fatty
309 acids and cerolipoid. The flat absorption peak indicated that hydrophobic substances were
310 removed [35].

311 - The characteristic peaks of ACWPF at 1737 cm^{-1} , 1602 cm^{-1} and 1507 cm^{-1} became
312 slower, indicating that hemicellulose and lignin were removed partly;

313 - There was no characteristic absorption peak in 1737 cm^{-1} of ALWPF, which meant that
314 alkali treatment could remove hemicellulose in WPF, and it was an effective method to
315 remove hemicellulose. The absorption peak at 1602 cm^{-1} became flat, indicating that lignin
316 was removed slightly [17].



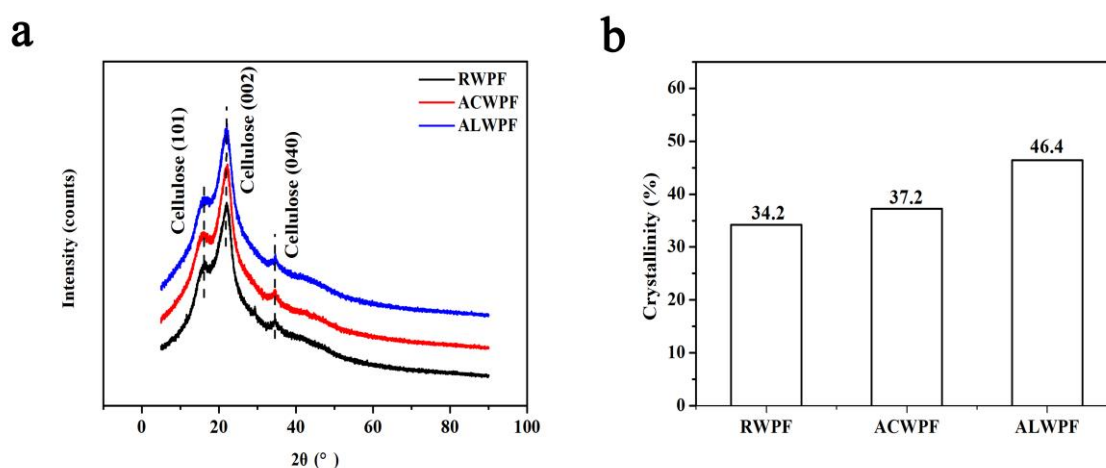
317
318 **Figure 3** FTIR spectra of windmill palm fiber with different treatments for the wave numbers
319 of (a) 4000 to 400 cm^{-1} , and (b) 2000 to 400 cm^{-1} .

320 In addition, 1157 cm^{-1} and 1050 cm^{-1} are the stretching vibration of ether bond (C-O) in the

321 glucose of cellulose, and the peak at 914 cm^{-1} is weak, which is attributed to the β - Glycosidic
322 bond of cellulose monosaccharides. These characteristic peaks belong to cellulose absorption
323 peaks. The shape and strength of palm fiber were enhanced after acid / alkali treatment, which
324 proved that the content of cellulose in palm fiber increased [35].

325 3.1.3 Aggregated structure of windmill palm fiber

326 The X-ray diffraction pattern of WPF, ALWPF and ACWPF are given in **Fig. 4a** The main
327 diffraction peaks of palm fiber were 16.2° (101), 22.1° (002) and 34.5° (040), corresponding
328 to cellulose I. Segal et al. [30] proposed that crystallization index can be used to characterize
329 the crystallinity of natural cellulose. The higher the crystallization degree, the higher the
330 crystallinity of natural cellulose. **Fig. 4b** was the crystallization index of raw and pretreated
331 palm fiber. The crystallinity index was relatively low for the raw fibers (RWPF 34.2%) but
332 increased after acid or basic treatment to reach 37.3% and 46.4% respectively due to
333 hemicellulose, lignin and cerolipoid removal.



334
335 **Figure 4** (a) X-ray diffraction patterns of different treatments; (b) Fiber crystallinity of
336 different treatments

337

338 *3.1.4 Mechanical characterization of windmill palm fiber*

339 Fiber fineness was usually measured by linear density and geometric size. The larger the
 340 linear density, the thicker the fiber, the larger the diameter. The diameter of fiber had an effect
 341 on the tensile properties, the larger the fiber diameter, the lower the tensile strength. The linear
 342 density of RWPF was 39.74 tex, which was in accordance with a previous study dealing with
 343 palm fibers [36] and larger than other natural fibers (such as jute, sisal, flax, etc.) [16]. The
 344 results in **Table 3** show that the linear density of treated fibers decreased, probably because of
 345 the removal of components on the fiber surface, as shown in **Fig. 2c** and **2d**. The cross section
 346 of WPF was composed of vessel, sieve tube, sclerenchyma and phloem tissue, so there is a
 347 certain cavity in the palm fiber. The previous study showed that the cell wall was swelled to a
 348 certain extent in the alkali treatment, which made the cell wall thicker, the cavity smaller and
 349 the hollowness decreased [37]. Therefore, when the fiber diameter is reduced, the cavity size
 350 is lower, and the cell wall becomes thicker, the material can bear more tensile force and show
 351 better mechanical properties at the same conditions.

352 **Table 3** Fineness and mechanical properties of windmill palm fiber by different pretreatments

	RWPF	ACWPF	ALWPF
Linear density (tex)	39.74 ± 4.67	32.55 ± 2.01	24.37 ± 1.82
Modulus (Gpa)	2.46 ± 0.85	3.45 ± 0.34	6.57 ± 0.46
Breaking strength (Mpa)	309.94 ± 12.94	324.96 ± 7.29	392.13 ± 4.52
Elongation at break (%)	26.58 ± 4.32	24.15 ± 3.13	23.60 ± 3.41

353

354

355 The mechanical properties of treated palm fiber were shown in **Table 3**. It can be seen that

356 the tensile strength of treated palm fibers increased slightly. These results agree with previous
357 works done on jute and palm fibers [38,39]. When hemicellulose was removed, the fibers may
358 be became not compact and the rigidity became smaller, so that the fibers can rearrange
359 themselves along the tensile deformation direction. The softening of the fibers affects the
360 stress transfer between the raw fibers, thus affecting the increase of fiber stress under tensile
361 deformation. With the removal of lignin, the micropores of the fibers are gradually eliminated,
362 resulting in the middle sheet connecting the cells was more plasticity and uniformity.
363 Hemicellulose is a kind of disordered and irregular nonlinear material, so its mechanical
364 properties are poor. The removal of partial hemicellulose is beneficial to increase the
365 mechanical properties of the fiber. The significant increase in modulus and tensile strength
366 observed for alkali-treated fibers (6.57 GPa and 392.13 MPa, respectively), can be explained
367 by the greater removal of non-cellulosic polymers associated to the increase of crystallinity
368 previously described (**Fig. 4b**). It has been also described that alkali treatment of natural
369 fibers can reduce the spiral angle of cellulose microfibrils. However, Beckermann et al. [40]
370 reported that alkali treatment reduced the strength of untreated fibers, which may be due to
371 the degradation of cellulose chain after alkali treatment. From **Fig. 2c**, some materials (lignin,
372 wax and oil) were removed from the fiber surface after acid treatment, which may be the
373 reasons for the increase of tensile strength and Young's modulus of ACWPF. Different from
374 alkali treatment, the modulus and breaking strength of ACWPF were slightly higher than that
375 of RWPF. It showed that acid treatment method has little effect on the mechanical properties
376 of the composites.

377 After hemicellulose and lignin were removed, the elongation at break decreased, and the

378 values were shown in **Table 3**. The elongation at break of RWPF was 26.58% when the
379 stretch length was 3 cm. After acid treatment and alkali treatment, the elongation at breaks of
380 ACWPF and ALWPF had almost results with 24.15% and 23.60%, respectively. The higher
381 elongation at break makes the palm fiber used as an elastic reinforcing material for the
382 preparation of composite materials [38].

383

384 *3.2 Effect of HVEF on windmill palm fiber and PF adhesive*

385 *3.2.1 FTIR analysis*

386 The surface functional groups of WPF and PF adhesive after HVEF were first analyzed by
387 FTIR (suppl materials **Fig. S1**). After HVEF treatment, the oxygen-containing functional
388 groups of WPF increased to a certain extent [41]. However, this change was not significant.
389 Due to the limitation of this technology in effective depth penetration, especially in the short
390 wavenumber region, the change of functional groups caused by HVEF cannot be evidenced
391 by FTIR [1]. Therefore, XPS analysis was used to better comprehending the chemical
392 mechanism of WPF treated by HVEF.

393 The characteristic peaks of main chemical groups in PF adhesive were summarized in
394 **Table S2** [41]. It can be seen from **Fig. S1d** that the characteristic peaks at 3412 cm^{-1} (O–H
395 stretching vibration), 2916 cm^{-1} (C–H stretching vibration), 1602 cm^{-1} (C=C stretching
396 vibration), 1450 cm^{-1} (CH_2 shear vibration), 1213 cm^{-1} (C–O stretching vibration, O–H
397 bending vibration) and 1017 cm^{-1} (C–O stretching vibration) increased significantly by HVEF
398 treatment. With the increase of voltage, the intensity of these characteristic peaks increased
399 significantly, and the change rate changed with different voltage. At the condition of 60kV, the

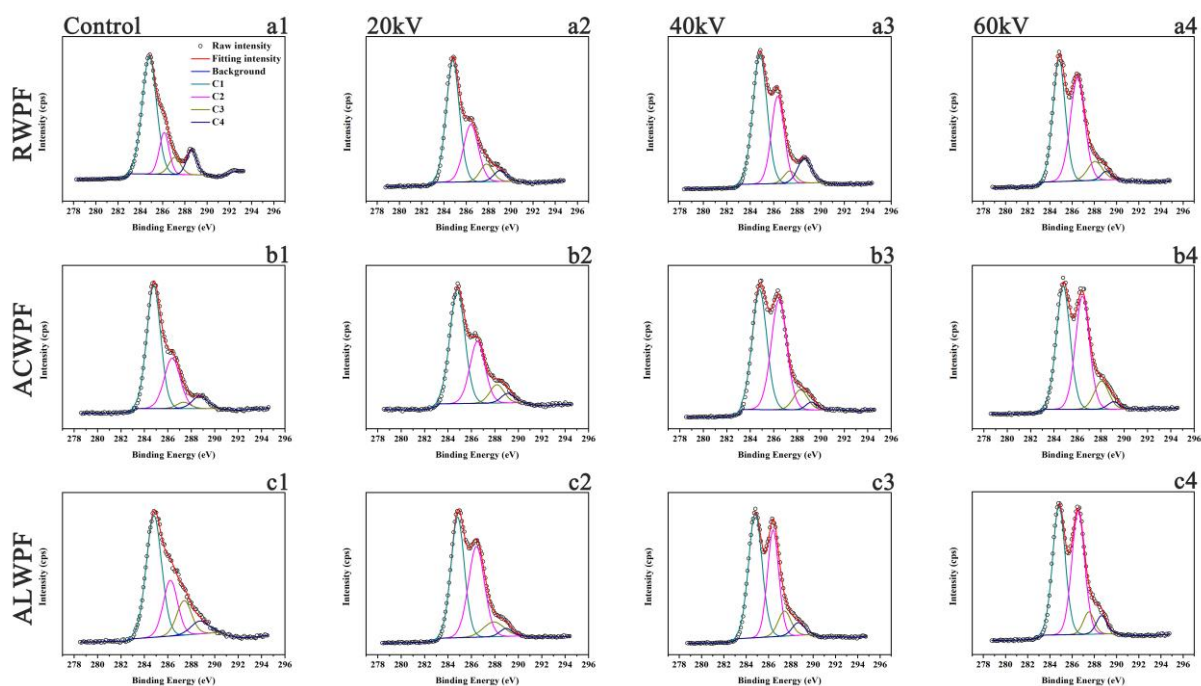
400 intensity of each characteristic peak was the highest compared with that of untreated PF
401 adhesive. These results were mainly attributed to the strong ionization of a large number of
402 water and oxygen molecules in the air under HVEF, and the formation of new electrons,
403 hydrogen ions, oxygen ions, hydroxyl and other chemical groups increased significantly [42].
404 These chemical groups impact the glue, which improved the polarity and charge density of the
405 adhesive. With the increase of voltage, the excited chemical groups increased, which was
406 beneficial to increase the content of hydroxymethyl. At a certain temperature, hydroxymethyl
407 was participated in the polycondensation of the adhesive. The increase of methylene bond in
408 the treated adhesive improved the chemical reactivity of the adhesive. At the same time, a
409 large number of excited electrons and broken chemical bonds (oxygen-containing groups)
410 were collided with the adhesive and reacted with each other, increasing the cross-linking
411 reaction of the resin.

412

413 *3.2.2 XPS analysis*

414 The changes of chemical elements such as oxygen and carbon on the material surface were
415 qualitatively analyzed by X-ray photoelectron spectroscopy. **Table S3** shows the main
416 distribution of C1s in WPF components [43]. **Fig. 5** is the deconvolution of high resolution
417 C1s XPS spectra. **Table S4** summarizes the XPS element analysis of WPF for the different
418 pretreatments (C1s%, O1s%), O / C ratio and deconvolution data of the C1s signals. The
419 carbon content and oxygen content of RWPF surface were 76.29% and 23.71%, respectively.
420 After acid / alkali treatment, the carbon content decreased to a certain extent and the oxygen
421 content increased relatively, which confirmed the partial removal of hemicellulose and lignin

422 after acid / alkali treatment previous observed (section 3.1) [36]. After HVEF treatment (60
 423 kV), the O / C ratio of RWPF, ACWPF and ALWPF increased significantly by 35.48%,
 424 36.11% and 47.06%, respectively. The results showed that the oxygen-containing chemical
 425 groups on the materials surface were increased by HVEF treatment. According to literature
 426 data, this observation is attributed to the fact that HVEFs generate active entities in the air
 427 which can oxidize the surface of the material [44]. The reason for the greater increase for
 428 ALWPF is that the fiber surface is more accessible and exposed after alkaline treatment.



429
 430 **Figure 5** High resolution of XPS spectra with C1s divided into C1, C2, C3 and C4
 431 components for different treatments

432
 433 The results from **Fig. 5** and **Table S4** showed that the C1, C2, C3 and C4 peaks of RWPF,
 434 ACWPF and ALWPF changed significantly after HVEF treatment. The C2, C3 and C4
 435 components of RWPF-60 increased by 31.92%, 28.68% and 15.15%, the C2, C3 and C4
 436 components of ACWPF-60 increased by 34.86%, 61.61% and 17.32%, the C2, C3 and C4

437 components of ALWPF-60 increased by 39.74%, 71.15% and 15.35%, and C1 component
438 decreased significantly. This may be explained by the fact that a large number of broken
439 oxygen-containing chemical groups by HVEF treatment reacted with the groups (– OH and –
440 CHO) on the material surface, which significantly improved the degree of polarization and
441 oxidation of the material surface [45]. The change of C1s composition indicated that the
442 increase of oxygen-containing functional groups on the WPF enhanced the surface wettability.
443 With the increase of treatment voltage, C2, C3 and C4 components increase significantly,
444 while C1 component decreases. This trend is in accordance with an increase of
445 oxygen-containing groups content and a higher WPF surface reactivity [44, 46]. However,
446 with a further increase of the voltage, the each component change rate decreased, which was
447 mainly due to the existence of limit value in the process of HVEF induced effect [47].

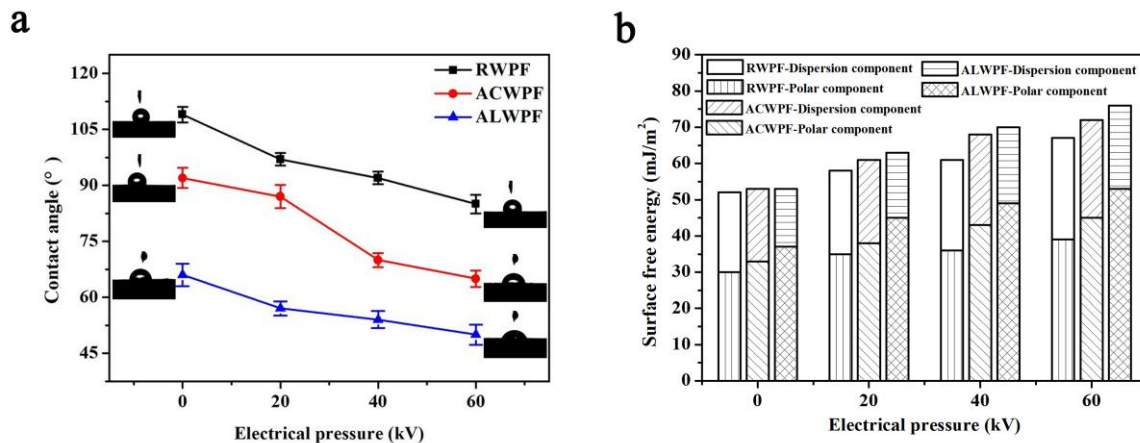
448

449 *3.2.3 Contact angle and surface free energy*

450 **Fig. 6** shows the results of water contact angle and surface free energy on the different
451 WPF treated by HVEF. As shown in **Fig. 6a**, the contact angle (IA) decreased markedly after
452 HVEF treatment and with an increase of treatment intensity. Compared with the untreated
453 different WPF, the IA of RWPF-60, ACWPF-60 and ALWPF-60 decreased from 109.09 ° to
454 85.32 °, from 92.83 ° to 65.41 ° and from 66.96 ° to 50.22 ° respectively.

455 The effect of different HVEF treatment voltage on the surface free energy of different WPF
456 was shown in **Fig. 6b**. The total free energy (T), dispersed component (DC) and polar
457 component (PC) on the surface of WPF effectively increased by HVEF treatment, and the
458 surface free energy of ALWPF changed the most after HVEF treatment. Compared with

459 ALWPF-0, DC of ALWPF-60 increased from 37.2 mJ / m² to 53.4 mJ / m², and PC of
 460 ALWPF-60 increased from 16.7 mJ / m² to 23.6 mJ / m². Due to the high polarity of the
 461 oxygen-containing functional groups, the surface polarity of palm fiber was improved. With
 462 the increase of voltage, the change rate of each component decreased, which was mainly due
 463 to the existence of limit value in the process of HVEF induced effect [47]. Based on the above
 464 analysis, we proved that HVEF treatment was beneficial to the excitation of
 465 oxygen-containing functional groups on the WPF surface in a short time. The results showed
 466 that HVEF treatment enhanced the hydrophilicity of WPF surface.



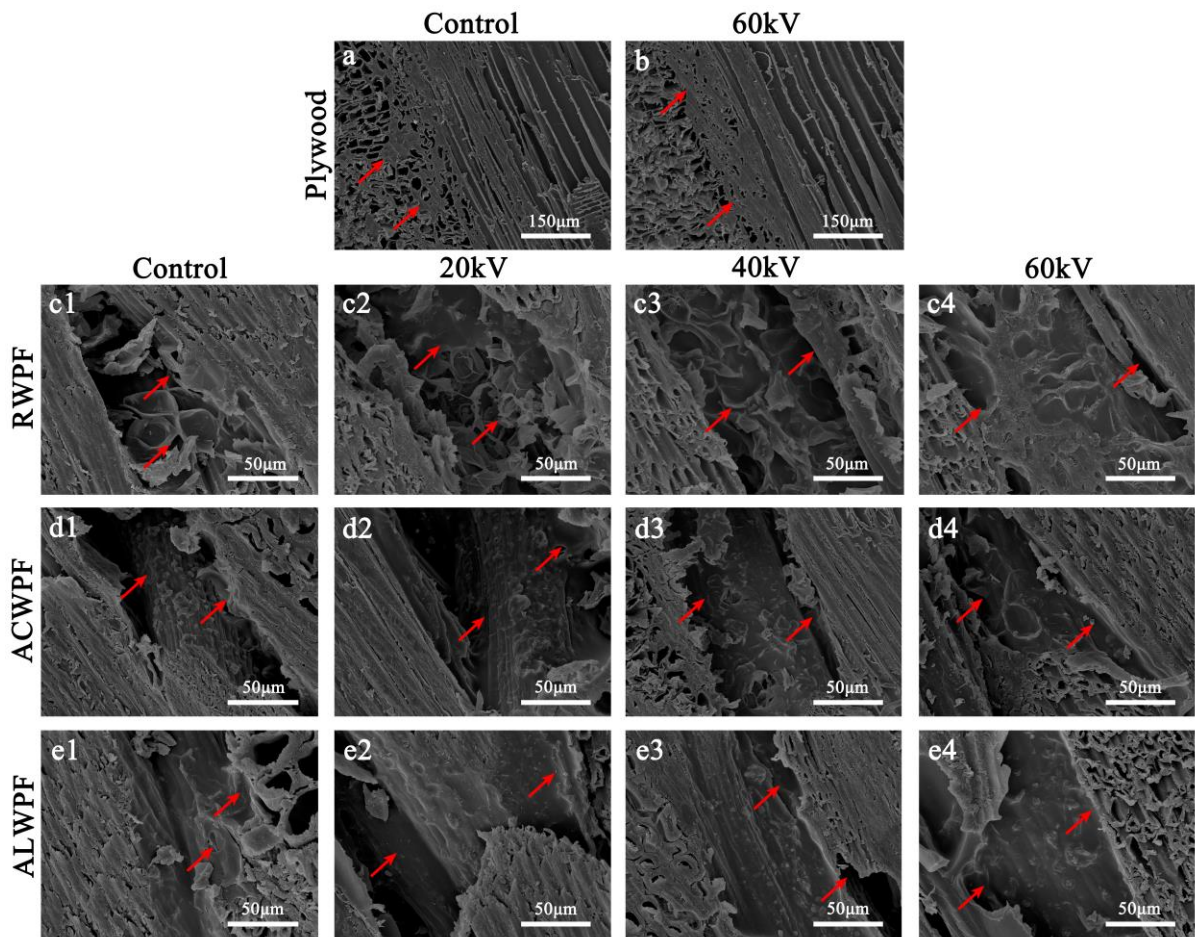
467
 468 **Figure 6** Distilled water contact angle (a) and surface energy (b) of WPF at different electrical
 469 pressures.
 470

471 *3.3 Performance evaluation of windmill palm fiber reinforced wood veneer composites*

472 *3.3.1 Micrographs of the Bonding Interphase*

473 The interface of WPF reinforced plywood with different HVEF treatments was analyzed by
 474 SEM, and the results are shown in **Fig. 7**. From the result of **Fig. 7a**, it can be concluded that
 475 the distribution of adhesive at the untreated bonding interface is dispersed and disordered.
 476 After HVEF treatment (**Fig. 7b**), the penetration depth of adhesive on both sides of the

477 bonding interface decreased significantly, and the tracheid was filled with more PF adhesive
 478 at the interface. It can also be seen that the distribution of PF adhesive at the bonding interface
 479 was more continuous and regular. These results are in accordance with the fact that the
 480 polarization and oxidation degree of the surface of the material are improved by HVEF
 481 treatment [45]. Therefore, HVEF can limit the penetration of glue from tracheid cavity, pit
 482 and gap into the internal structure deeper from the interface, and improve the chemical
 483 cross-linking reaction between adhesive and wood at the interface.

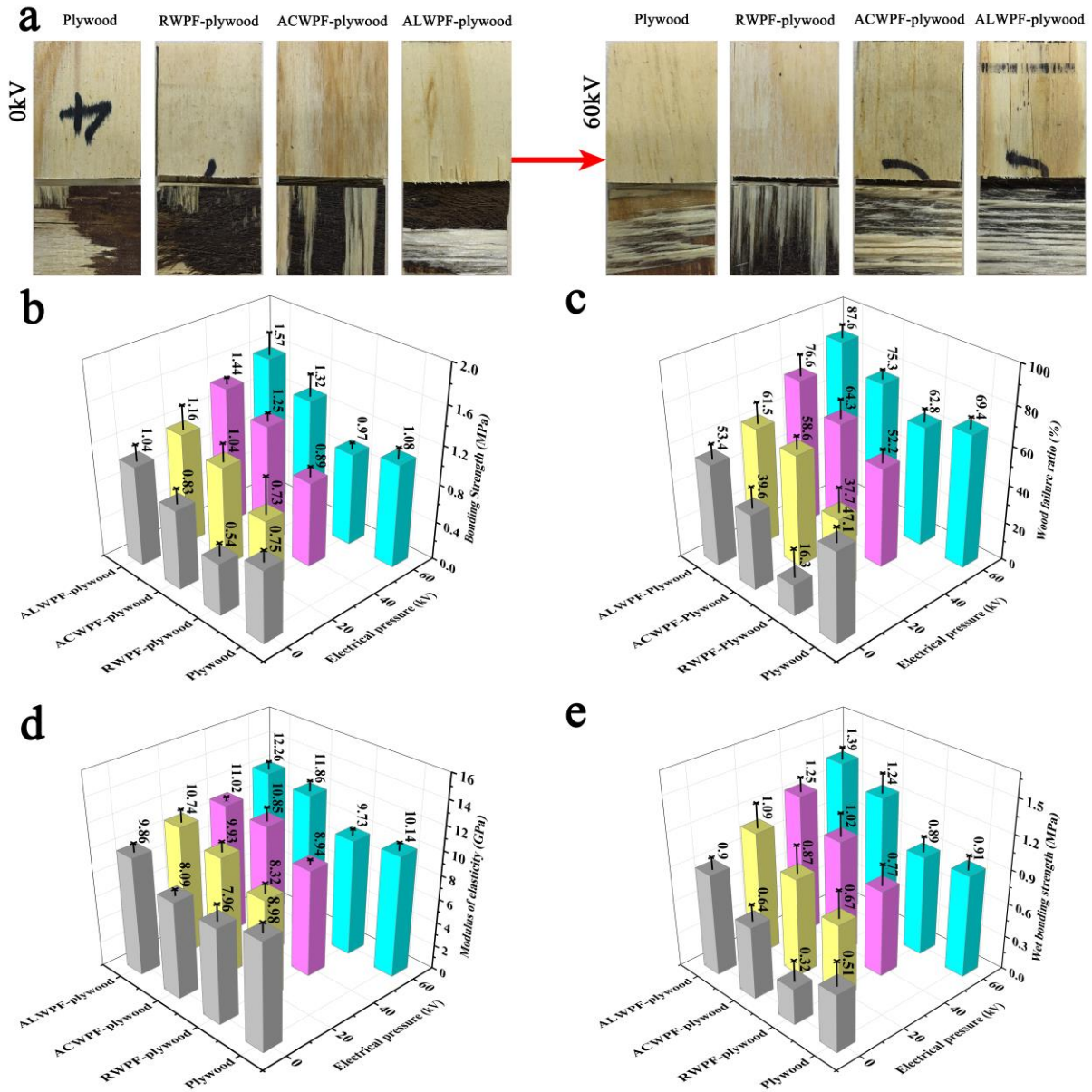


484
 485 **Figure 7** Micrographs obtained from the WPF reinforced wood veneer composites bonding
 486 interphase with different treatment parameters.

487
 488 According to the micrographs of **Fig. 7c1**, **7d1** and **7e1**, it can be observed the surface of

489 untreated fibers (Fig. **7c1**) appeared to be free of adhesive indicating a poor fiber-matrix
490 adhesion. An increase of the interfacial adhesion was observed for ACWPF and ALWPF
491 probably because of the removal of the silica particles and scaly substances on the surface of
492 palm fiber previously described [48]. Compared with the HVEF treated bonding interface, PF
493 treated HVEF can cover the fiber and improve the interface adhesion, so as to enhance the
494 adhesion between fiber and matrix. After 20 kV HVEF treatment, the area of WPF covered by
495 PF increased (as shown in **Fig. 7c2, 7d2 and 7e2**). This can be attributed to the HVEF
496 treatment, which can stimulate the oxygen-containing functional groups on the surface of
497 WPF in a short time, improve the hydrophilicity of WPF surface, and a large number of
498 excited electrons and broken chemical bonds (oxygen-containing groups) to collide with the
499 PF, thus increasing the cross-linking reaction of the PF [45, 49]. When the HVEF treatment
500 voltage increased from 20kV to 60kV, the PF coverage on the surface of WPF further
501 increased (as shown in **Fig. 7c4, 7d4 and 7e4**). Therefore, HVEF can limit the penetration of
502 glue from tracheid cavity, pit and gap into the internal structure deeper from the interface, and
503 improve the chemical cross-linking reaction between adhesive and wood at the interface.

504 A comparison of the with three WPF reinforced Plywood under 60kV (**Fig. 7c4, 7d4 and**
505 **7e4**) showed thatt the bonding interface of ALWPF-reinforced plywood is the best, the PF
506 distribution being continuous and uniform, and the fiber surface being completely covered
507 with PF. Under HVEF treatment, the surface wettability was higher, and more
508 oxygen-containing functional groups (consistent with the results of XPS analysis) were
509 excited to crosslink with the PF, which improved the interfacial properties.



510

511 **Figure 8** Fracture surface analysis (a). The bonding strength (b), wood failure ratio (c), MOE

512 (d), and wet bonding strength (e) of WPF reinforced wood veneer composites with different

513

treatment parameters.

514

515 **3.3.2 Mechanical properties test**

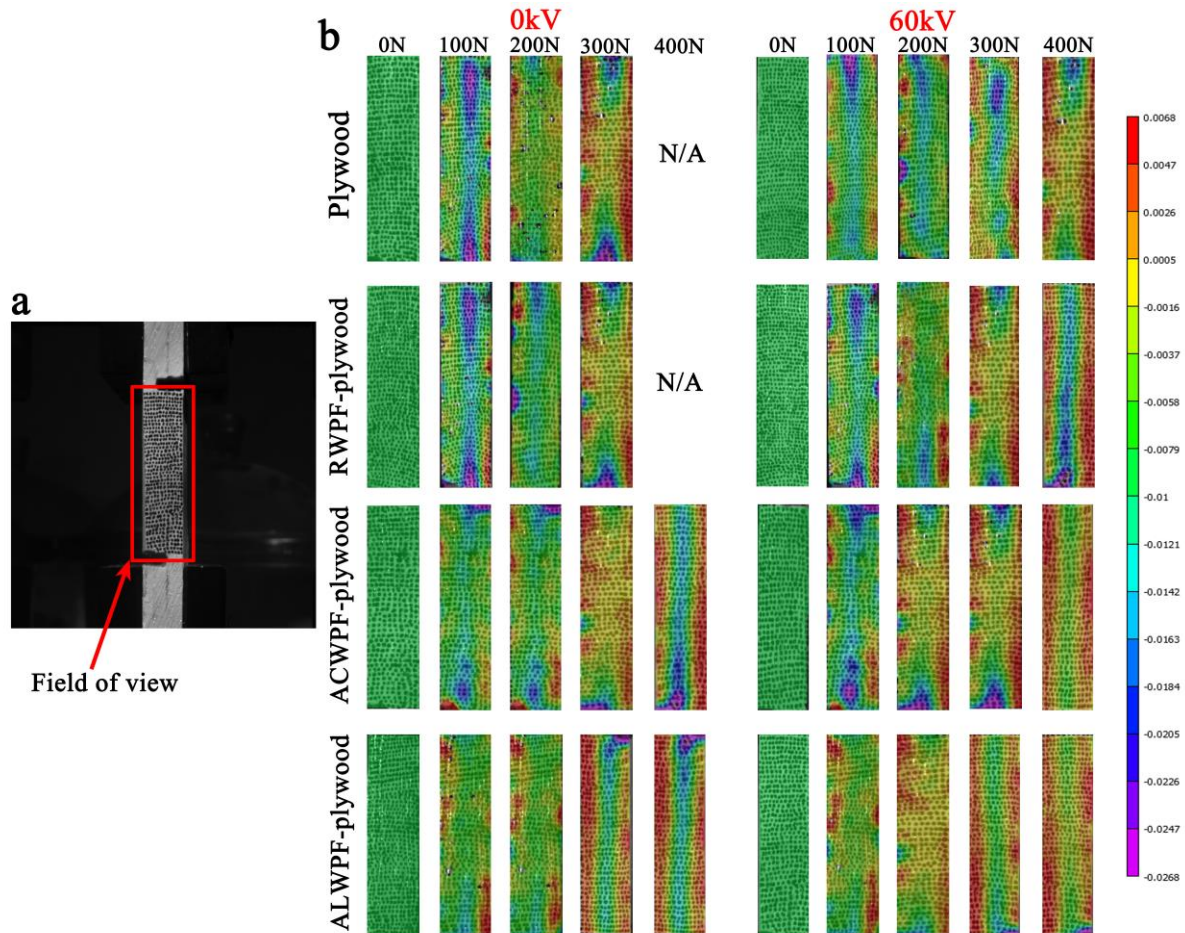
516 The mechanical properties of WPF reinforced plywood were analyzed by bonding strength

517 (BS), wood failure ratio, modulus of elasticity (MOE) and wet bonding strength (WBS). As

518 shown in **Fig. 8b**, the mechanical properties increased in the order RWPF < ACWPF <
519 ALWPF confirming the impact of the alkaline or acidic pretreatment on the properties of the
520 composite. It can also be seen that HVEF treatment can further significantly improve the
521 properties of the plywood. For ALWPF reinforced plywood, the BS was increased from 1.16
522 MPa to 1.44 MPa then to 1.57 MPa when HVEF voltage increased from 20 kV to 40 kV and
523 to 60 kV. It can be found a synergistic effect between the alkaline or acidic pretreatment and
524 the HVEF treatment, leading to an overall bonding strength of ALWPF and ACWPF
525 reinforced plywood better than that of plywood. From **Fig. 8a** and **8c**, it can be observed that
526 HVEF treatment can significantly improve the wood failure ratio.

527 Digital image correlation (DIC) technology, a non-contact high-precision displacement,
528 deformation and motion measurement method, has the advantages of simple light path, little
529 external influence and low requirements for vibration isolation conditions [50]. In this
530 experiment, DIC technology was used to analyze the shear strain distribution of WPF
531 reinforced plywood before and after HVEF (60kV) treatment (**Fig. 9b**). **Fig. 9a** shows the
532 tensile shear strain measurement process. Under the action of tensile stress, the internal
533 structure of plywood deformed to a certain extent. When the load reached the elastic limit
534 value, subtle cracks appeared in the weak area of the sample and they gradually expanded,
535 leading to tensile failure of the plywood.

536



537

538 **Figure 9** Tensile shear strain measurement (a), the tensile shear strain distribution (b) of WPF

539 reinforced wood veneer composites with different treatment parameters

540

541 Deformation after HVEF treatment (60kV) occurs at a higher stress than that of untreated
 542 plywood. The reason is that the PF of untreated plywood penetrates into the veneer along the
 543 crack of the veneer, and it is easier to disperse the stress from the main glue layer to the wood
 544 matrix, resulting in the absence of PF in the main glue layer and the deterioration of
 545 mechanical properties. As a result, after HVEF treatment the uniform and continuous glue
 546 layer previously observed at the bonding interface significantly improved the mechanical
 547 properties. For the shear strain of WPF reinforced plywood, it can be seen that the strain
 548 distribution of the tensile interface on both sides of the plywood is relatively uniform under

549 the load of 100 N. For 200N, deformation is observed at both ends under the action of tensile
550 stress with an increase in the value of the deformation. When the load reached 300 N, the
551 strain concentration of RWPF and ACWPF reinforced plywood appears to be more important.
552 In contrast, the ALWPF and HVEF samples show a clear and regular deformation field, the
553 deformation zone gradually extending in the length direction and the deformation zone being
554 relatively concentrated. These results indicate that acid/alkali treated WPF can reduce the
555 tensile stress concentration in plywood. The HVEF treatment has a significant effect on the
556 strain distribution in the shear section, leading to an improvement in both the strain transfer in
557 the shear section and the mechanical properties of WPF-reinforced plywood.

558 The bending properties of WPF reinforced plywood were analyzed by three-point bending
559 test (**Fig. 8d**). It was found that the bending properties of WPF reinforced plywood were
560 positively affected by HVEF treatment of 60kV, and the MOE of ALWPF (60kV) reinforced
561 plywood was increased by 36.5%. **Fig. 8e** shows the BS of plywood after cooking in boiling
562 water for 4h and drying in oven-dried at 63 °C for 20h according to Chinese National
563 Standard (GB/T 9846-2015) [51]. A strong decrease in BS for untreated WBS and RWPF
564 plywoods of 32% and 41% respectively was observed while the decrease in BS was more
565 limited for the acid / alkali reinforced plywood and HVEF treated samples (25% and 15%
566 respectively). Therefore, HVEF can effectively inhibit the decrease of BS after cooking.

567

568 **Conclusion**

569 Alkali pretreatments of wind palm fibers have significantly improved their surface
570 properties by removing silica particles and scales. An improvement in their mechanical

571 properties was also observed. Modulus and tensile strength were increased by 167.07% and
572 26.52% respectively. HVEF can improve the polarization and oxidation degree of the WPF
573 surface, and increase the reactive groups contents on the fiber surface (– OH and – CHO).
574 Due to the synergistic effect of acid / alkali pretreatment and 60 kV HVEF treatment, the
575 interfacial properties of reinforced plywood were effectively improved and the strain
576 distribution of the composite was more uniform. Compared with unreinforced plywood, the
577 BS and MOE were increased by 109.33% and 36.53% respectively. We hope that this process
578 will allow a better use of wind palm fibers for the design of innovative wood composite
579 materials.

580

581 **Declaration of Competing Interest**

582 The authors declare that they have no known competing financial interests or personal
583 relationships that could have appeared to influence the work reported in this paper.

584

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594

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