Effect of Modification with Methyl Methacrylate on the Mechanical Properties of *Plectocomia kerrana* Rattan

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This study aims to evaluate the mechanical properties of rattan/polymer composites prepared by polymerization with methyl methacrylate (MMA). The *P. kerrana* rattan samples were impregnated in a vacuum system and polymerized in an oven at 60 °C for 8 h, using 0.5 wt.% of azobisisobutyronitrile as a catalyst. The macro-mechanical properties of the treated and untreated samples were analyzed. The bending modulus and strength of the treated rattan increased by 206% and 215%, respectively. Additionally, the compressive modulus and strength increased by 109% and 107%, compared to untreated rattan. Scanning electron microscopy (SEM) images showed that MMA penetrated the cell lumen. Furthermore, Fourier transform infrared spectroscopy (FTIR) analysis revealed that MMA diffused into the parenchyma and vessels, but it was not found in the fiber wall. Thus, it can be inferred that the improvement in the mechanical properties of treated rattan was mainly caused by the strengthened parenchyma and vessels. Modification with MMA was shown to be an effective way to enhance the macro-mechanical properties of *P. kerrana*.

Keywords: Plectocomia kerrana; Methyl methacrylate; Mechanical properties; Modification

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INTRODUCTION

Rattan is a major non-timber forest product that comes from tropical forests. Rattan canes are an important natural biomass used for the production of furniture and other products, such as traditional items like crossbow strings, handbags, fish traps, and sports equipment (Olorunnisola and Adefisan 2002). There are about 600 species of rattan, most of which are native to South and Southeast Asia, and 40 of those species are found in China. However, less than 10 species are utilized and marketed. Most identified rattan species lack commercial value due to relatively high variability in the structural composition of the stem and poor mechanical properties (Liese 2002; Sunderland and Dransfield 2002). Efforts have been made to modify these less-valuable rattan species and to enhance their mechanical properties. Many methods of modification have been investigated, for instance, heat, chemical modification, impregnate modification, and densification (Militz and Zbik 2009). Among these technologies, impregnation has been commercially applied, for example by heat treatment, acetylation, furfural, resin impregnation, and thermo-compression (Evans *et al.* 2000; Wan and Kim 2006; Gunduz *et al.* 2009; Wei *et al.* 2013).

Resin impregnation modification involves impregnation of monomeric or pre-polymeric resins of low molecular weights into porous structures such as vessels and tracheid. Such treatment has been reported to improve dimensional stability and

mechanical strength. This method involves either the bulking of polyethylene glycols or phenol/formaldehyde resins, or the filling of wood lumen by impregnation and polymerization of vinyl monomers. In order to develop this process, studies were performed in several countries such as Canada (Ding *et al.* 2013), Brazil (Magalhães and Silva 2004), India (Devi and Maji 2013), China (Li *et al.* 2012), Malaysia (Islam *et al.* 2014), and others. These studies used low quality wood from fast-growing plantations such as pine (Wei *et al.* 2015a), polar, beech, and rubber. The impregnation monomers included methyl methacrylate (Enamul Hoque *et al.* 2014), glycidyl methacrylate, styrene, acrylonitrile, furfuryl alcohol, and phenolic resin.

The MMA monomers are inexpensive and accessible, mainly due to the large production in Asian industries (in China, Singapore, and South Korea). Moreover, they are stable, translucent, low-viscosity, and can be easily polymerized and impregnated into other materials. Also, the inherent properties of its polymer, polymerized methyl methacrylate have good impact and weather resistance, and the final product is durable and does not contain toxic chemicals.

In contrast with pinewood (Mattos *et al.* 2014), *Fagus sylvatica* (Souloungangа *et al.* 2004), meranti wood (Enamul Hoque *et al.* 2014), Chinese fir, and poplar, modification with MMA of rattan has not been extensively investigated. The MMA wood had been tested worldwide for its physical, mechanical, and biodegradable properties. An increase in mechanical properties of the composites has been reported due to reinforcement of the lignocellulosic cell wall by means of cross-linked/polymerized MMA resin matrix (Enamul Hoque *et al.* 2014).

The objective of this study was to investigate the improved mechanical properties of *P. kerrana* based on the use of MMA. The MMA uptake and distribution in specimens were evaluated using scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR).

EXPERIMENTAL

Materials

The rattan species *P. kerrana* was acquired from native stands in Yingjiang country, Yunnan province, China. Seven canes of wild rattan with an average length of 19.6 m and average diameter of 31.51 mm were collected. Each cane was cut about 30 cm from the ground, and then labeled at every node. The average node length was 26.80 mm, and it was sampled at every three nodes. An image of the end's surface was taken with a high-resolution camera and is shown in Fig. 1A.

Rattan canes were cut from internodes into bending and compressive specimens. The specimen diameter for the compression test was 30.25 $\pm$ 2.84 mm and the height-to-diameter ratio was kept at 2 for effective experimentation. The two ends' surfaces were parallel and vertical to the centerline of the specimen. The bending specimens had a diameter of 29.71 $\pm$ 2.42 mm.

The MMA resin was purchased from Fuchen Chemical Corporation, Tianjin, China. Azobisisobutyronitrile (AIBN) was obtained from Fuchen Chemical Corporation and was used as the heat catalyst.

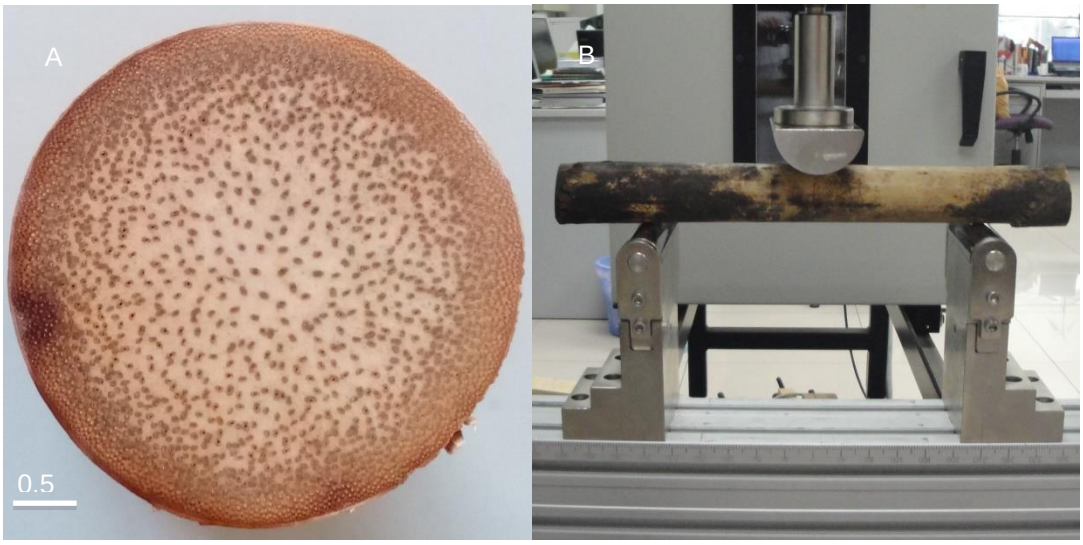

Fig. 1. Sampling and testing of rattan: cross-section showing a distinct variation in density between outer and inner portion of *P. kerrana* (A), image of bending test (B)

Methods

Preparation of treated rattan

The weights of all oven-dried specimens were measured before and after MMA resin treatment using a balance with accuracy to ±0.001 g. The rattan specimens were placed in a vacuum stainless cylinder, and a relative vacuum of 0.1 MPa was applied for 1 h to remove air from the rattan voids.

A monomeric solution consisting of 0.5% AIBN radical was added into 100% MMA by weight. The rattan specimens were immersed into the monomer solution at atmospheric for 4 h for impregnation.

After impregnation, the excess resin was wiped off the surface with paper towel. Then, the specimens were wrapped in aluminium foil and heated for 8 h at 60 °C to start the thermally-induced polymerization process.

The macro-mechanical properties test

The treated and untreated rattan specimens were used to test the macro-mechanical properties. Specimens for three-point flexural tests were $8D \times D$, where D was the diameter of cane, and those for compressive strength parallel to grain were $2D \times D$. Due to the absence of rattan mechanical property testing standards, the methods used for the bending tests were taken from Lv *et al.* (2012), and the compressive test was carried out according to GB 1935-2009 (2009). The mechanical properties of the specimens were determined using an electronic universal material testing machine (Instron 5582, Instron Co., USA), as shown in Fig. 1B. All testing was carried out in an environment of 20 °C at 55 to 60% RH. Eight replicates were used for each formulation.

Scanning electron microscopy

Approximately 5 × 5 × 5 mm cubes ($R \times T \times L$) from treated and untreated specimens were sawn as SEM specimens. The surfaces of the specimens were finished with a sliding microtome (SM2000R, Leica, Germany). Specimens were then observed under a scanning electron microscope (ESEM-XL30, FEI, USA).

Fourier transform infrared spectroscopy

The infrared spectra used for evaluating the chemical structure of untreated and treated rattan specimens were measured with a Thermo Scientific Nicolet iN10 FT-IR microscope (Thermo Nicolet Co., Madison, WI, USA), in direct transmittance at a resolution of 8 cm^{-1} for 16 scans in the range of 720 to 4000 cm^{-1}. The alignment of the light equipment and the background spectra were ensured before all tests.

RESULTS AND DISCUSSION

Macro-Mechanical Properties

The static bending properties and compressive properties of the treated and untreated rattan were determined. Typical load-displacement curves of the treated and untreated specimens in bending and compression are presented in Fig. 2. As shown in Fig. 2A, the treated rattan composite exhibited a higher ultimate bending load than that of the untreated rattan, which indicated the effectiveness of the mechanical enhancement provided by the rattan composite of MMA. However, the load-displacement curve of treated rattan exhibited more brittleness than that of untreated rattan because of the nature of the resin itself. The compressive load-displacement curves of treated and untreated specimens were similar, which is displayed in Fig. 2B. There were two distinct stages, identified as the rapidly increasing linear stage and slowly increasing or nearly stable yield stage. Liu *et al.* (2014) observed the same compression curve in *P. assamica* Griff. However, it exhibited a prolonged plastic deformation and higher loading capacity after treatment. Previous research about two-phase composite materials showed that the long stable yield stage after yielding point was due to the parenchyma, which could absorb large compressive deformation owing to its highly compressible foam-like structure. In addition, the foam-like parenchyma under longitudinal compression might be able to prevent the fibers from large-scale buckling (Obataya *et al.* 2007).

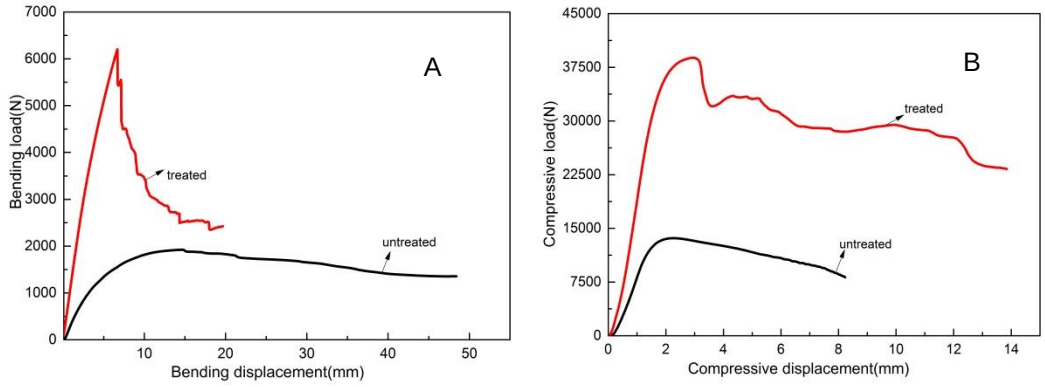

Fig. 2. The mechanical load-displacement of untreated and treated rattan: (A) bending load-displacement, (B) compression load-displacement

The mean values of different strength properties for untreated *P. kerrana* rattan and other rattan species found in the literature are presented in Table 1 (Bhat and Thulasidas 1992; Wahab *et al.* 2004; Wang *et al.* 2011). Among the seven species, *C. manna* was the strongest cane, and it displayed the highest mean values of MOR and MOE, as well as relatively high compressive strength. In contrast, *P. kerrana* rattan had

the lowest strength with exceptionally low values of MOR, MOE, and compressive strength. The other species were considered moderately strong canes. Poor mechanical properties are some of the reasons that *P. kerrana* rattan is not used commercially.

Table 1. Mean Value for Mechanical Properties for Seven Raw Rattan Species

Species	Bending modulus MOE (MPa)	Bending strength MOR (MPa)	Compressive modulus (MPa)	Compressive strength (MPa)
Plectocomia kerrana	846.8	31.1	831.6	17.9
Calamus simplicifolius	1375.3	67.9	1571.2	31.6
Daemonorops margaritae	1525.5	57.6	1198.5	23.5
Calamus manan	3450.0	94.0	-	39.1
Calamus nagbettai	4057.0	91.0	-	33.6
Calamus thwaitesii	2156.0	51.3	-	29.2

The bending and compressive properties of untreated and treated rattan are summarized in Table 2. The bending and compressive elastic moduli of untreated rattan were 846.8 and 831.6 MPa, respectively. The elastic moduli of treated rattan were 2593.8 and 1735.4 MPa, respectively, which were considerably higher than that of untreated rattan. These increases, which were 206% and 109%, respectively, were larger than the results of previous research. Ariffin *et al.* (1993) found that the increase in bending modulus for PF-impregnated *C. manna* was 11 to 15%. Xu (2010) indicated that the increases in bending modulus for MMA-impregnated *Calamus simplicifolius* and *Daemonorops margaritae* were 36% and 75%, respectively. The greater increase in *P. kerrana* probably arose from its considerable quantity of voids including parenchyma and lumen. Compared with wood particles impregnation, the PMMA chain molecules intercalated both voids structure and surface in the rattan, which could have been more effective in improving the mechanical properties of the composites.

Table 2. Mechanical Properties of Untreated and Treated *P. kerrana* Rattan

Mechanical properties	Treated rattan	Untreated rattan	Increase (%)
Bending modulus (MPa)	2593.8	846.8	206
Bending strength (MPa)	97.9	31.1	215
Compressive modulus (MPa)	1735.4	831.6	109
Compressive strength (MPa)	37.0	17.9	107

The bending strength and compressive strength of the treated rattan were 97.9 and 37.0 MPa, which were much higher than those of untreated rattan. These results were reasonable because MMA polymerized throughout the rattan with good adhesion and improved the mechanical strength. According to previous reports, after MMA impregnation, the structures inside the vessel were of the category of smectite clay (Morris and Zbik 2009; Zbik and Frost 2010), and these types of particles had the ability to improve the mechanical properties of some materials, particularly wood-related materials (Islam *et al.* 2012; Iman and Maji 2012). The bending and compressive strength of treated rattan were almost equal to that of *C. manan* rattan, the widely used industrial rattan species at present, which has bending and compressive strengths of 94.0 and 39.1 MPa, respectively. This indicated that the *P. kerrana* rattan modified with MMA could be used in place of *C. manan* rattan in furniture products.

Microstructural Analysis

Observation by SEM

SEM images of untreated and treated rattan specimens are presented in Fig. 3. They show that the cross-section surfaces of untreated rattan were covered with voids and uneven layers in the parenchyma, fiber cells, and vessels. The parenchyma cells accounted for the largest proportion. Figure 3B shows that the vessels and parenchyma of the treated rattan were partially filled with MMA, which indicated that MMA penetrated the voids. Similar reports have been reported for wood materials (Enamul Hoque *et al.* 2014). This observation confirmed that the enhancement mechanical properties were attributable to the filling of voids and covering the surface with MMA. It is important to note that MMA does not always completely fill the lumen because of shrinkage during polymerization (Li *et al.* 2010). As shown in Fig. 3B, some voids still existed between the cell lumen and MMA.

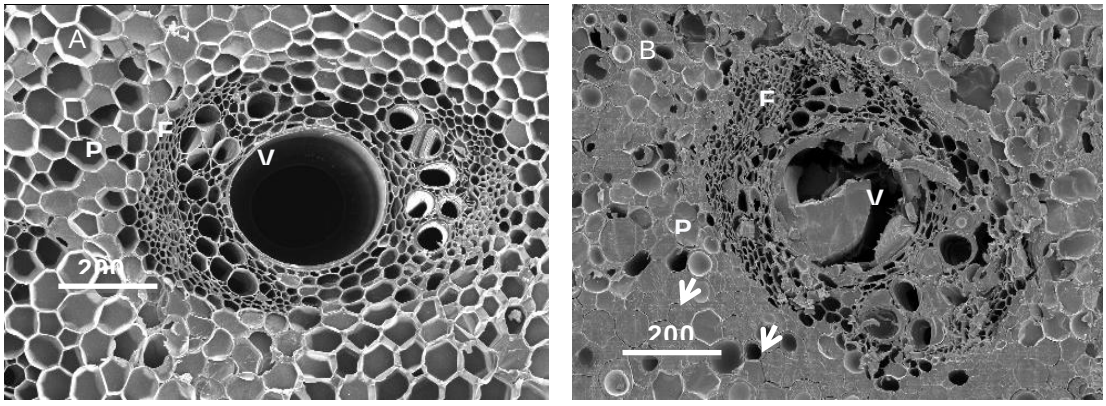

Fig. 3. SEM micrographs of (A) untreated rattan and (B) treated rattan, as well as the components of rattan with fiber cell (F), parenchyma (P), and vessel (V)

Chemical evaluation by FTIR

Figure 4 shows the FTIR spectra of the untreated rattan, pure MMA, and MMA-modified rattan. The rattan had similar chemical constituents to wood.

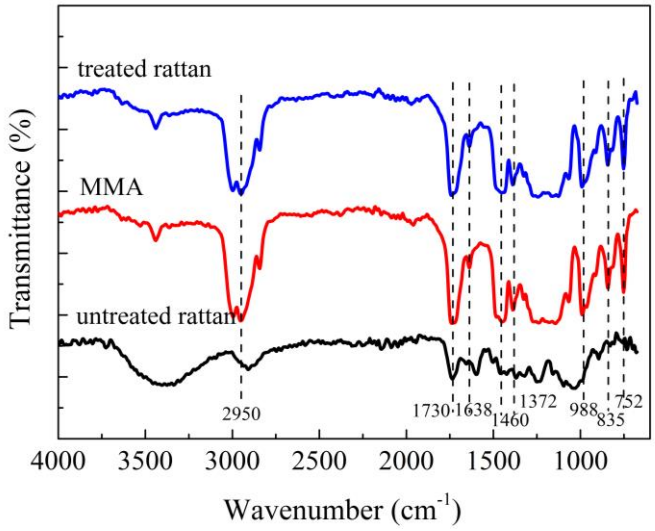

Fig. 4. FTIR spectra of untreated rattan, treated rattan, and pure MMA

Thus, the characterization and assignment of FTIR peaks in rattan was done in reference to wood, which is detailed in Table 3 (Schwanninger *et al.* 2004; Colom and Carrillo 2005; Huang *et al.* 2008; Popescu *et al.* 2010, 2011; Mattos *et al.* 2014; Wei *et al.* 2015b).

Table 3. Assignments of the Peaks Characterized in the FTIR Spectra of the Untreated and Treated Rattan

Peaks	Assignments
3440	O-H stretching in hydroxyl groups
2997 and 2950	C-H and CH_3 stretching in methyl and methylene groups
1730	C=O symmetric stretching of aliphatic groups (xylan), stretching of carbonyl group
1638	C=C stretching in MMA and composite
1589	Benzene ring stretching in lignin
1460	C-H deformation in MMA and composite, CH_2 deformation stretching in lignin and xylan
1372	CH_2 bending in cellulose and hemicellulose, Aliphatic C-H stretching in methyl and phenol OH
1230	C-C and C-O stretching vibrations
1157	C-O-C asymmetric band in cellulose and hemicellulose, C=O stretching in aliphatic groups
1140	C-O ether bond stretching vibrations
1086	C-O stretching
988	C-H out-plane bending vibration
896	Anomere C-groups, C_1-H deformation
835 and 752	C-H out-plane bending vibration

It can be seen that the intensity of the band at 1730 cm^{-1}, which is assigned to the carbon and oxygen (C=O) stretching vibrations typical of non-conjugated ketenes and conjugated carboxyl acids from the lignin and hemicelluloses (Colom and Carrillo 2005; Wei *et al.* 2015c), showed a significant increase because of the C=O groups from the MMA. Meanwhile, a small fluctuation at a lower wave number (1638 cm^{-1}) was observed for the treated rattan specimen, which corresponded to the double bond between carbon and carbon (C=C). The bands at 1460 cm^{-1} and 1372 cm^{-1} were due to C-H deformation and CH_2 bending stretching.

Treated rattan showed higher absorptions at these peaks, which referred to C-H bonding both from resin and chemical bonds between the copolymers and cell wall. An increase of the intensity of the bands at 988 cm^{-1} and 752 cm^{-1}, assigned to C-H out-plane bending vibration, was observed. All of the data provided clear evidence that MMA was attached to the untreated rattan.

A rattan culm is composed of three kinds of cells: matrix tissue cells (parenchyma), vessel, and sclerenchyma cells (fibers). The MMA distribution in treated rattan was evaluated using FTIR microscopy (Fig. 5). In Fig. 5 A and B, typical MMA peaks were observed both in the parenchyma wall and cell corner of the treated rattan. The peaks at 1460, 988, 896, and 752 cm^{-1} corresponding to the stretching or bending vibration of the C-H, appeared or increased in intensity in the spectra of treated rattan parenchyma. This indicated that MMA penetrated the parenchyma cell wall. The same results were obtained for the vessel (Fig. 5E, F).

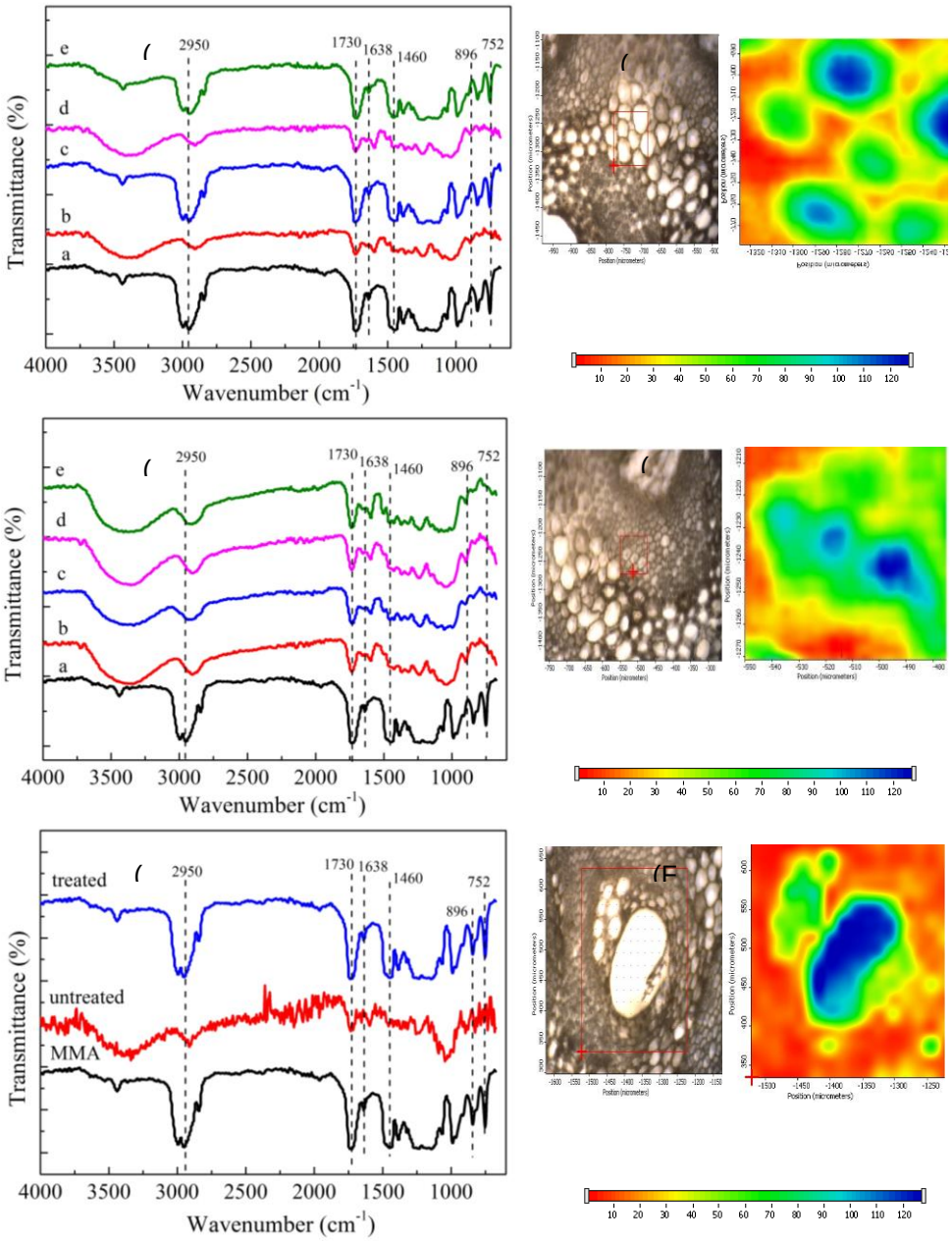

Fig. 5. FTIR spectra (A, C, and E) and image analysis (B, D, and F) of components in rattan: parenchyma (A, B), fiber cell (C, D), and vessel (E, F); a is MMA, b is the fiber wall in untreated rattan, c is the fiber wall in treated rattan, d is the cell corner in untreated rattan, and e is the cell corner in treated rattan.

For the fiber cells, there were no significant changes for the peaks at 1460, 988, 896, and 752 cm^{-1} (Fig. 5C, D). That means the MMA did not successfully enter the fiber cells, consistent with the SEM results, which might be related to the volume of rattan/MMA. Properties such as the greater number of pores in parenchyma and vessel could make them more permeable than fiber cells. If the volume of rattan/MMA reached a certain value, the MMA could penetrate the fiber cells. This will be studied in future experiments. According to the above analysis, it can be inferred that the improvement of

the mechanical properties of rattan composite was mainly caused by the strengthened parenchyma cells and vessels.

SEM and FTIR analyses showed that the voids of rattan were filled with MMA, which enhanced the adhesion between rattan and polymerized MMA matrix. Previous studies had suggested that the enhanced process was established by the filling of the void structure, mainly parenchyma and vessels in the rattan (Koubaa *et al.* 2012). Little interaction was observed between MMA monomer and the functional groups on the wood surface (Zhang *et al.* 2006). The observed higher mechanical value might be due to the restriction in the mobility of the polymer chains inside the voids (Hazarika *et al.* 2012).

CONCLUSIONS

1. Impregnation of *P. kerrana* with MMA can improve its mechanical properties. The MMA penetration has a greater effect on mechanical properties of the treated rattan, which can reach up to approximately 200%.
2. FTIR and SEM analysis showed that the MMA molecules entered into the parenchyma and vessel, but little or no MMA molecules are present in the fiber cells. It can be inferred that the MMA molecules diffused effectively into the pores in the parenchyma and vessel of *P. kerrana*.
3. Therefore, modification with MMA is an effective way to enhance the macro-mechanical properties of *P. kerrana*, and the treated rattan may be used in place of *C. manan* rattan in furniture products for structure.

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