# Potential Use of Waste Cotton in Production of Biomass Composites

Junlin Qu, Zhongwei Wang, Changchun Hu, Qian Yin, and Yan Pang \*

Mechanical and waterproof properties were evaluated for hot-pressed cotton boards produced from different layers (3, 4, and 5) of cotton veneers under the same weight of cotton fibers and melamine-urea-formaldehyde adhesive. The mechanical and waterproof properties of cotton boards exceeded the specifications for particleboard and medium-density fiberboard of the China national standard requirements, and the four-layer cotton boards performed better. Scanning electron microscopy images showed that fibers were intertwined to form a dense network structure after hot-pressing with water, and thicker veneers were not conducive to the penetration of adhesives after cold-pressing. Fourier transform infrared spectra indicated that hydrogen bonding, physical, and mechanical bonding took place in the cotton veneers, and stronger absorption peaks were shown for the chemical functional groups of the five-layer and four-layer cotton boards. X-ray diffraction spectra revealed that the cellulose crystallinity of the cotton boards (3, 4, and 5 layers) increased to 74.5%, 74.4%, and 73.2%, respectively. Thermal gravity/ differential thermal gravity curves showed that the thinner cotton veneers of the cotton boards showed better thermal stability. These results showed promise for the revaluation of this textile waste to produce biomass composites and for its potential use as a raw material in the preparation of biomass composites.

Keywords: Textile waste; Cotton board; Mechanical properties; Microscopic appearance; Chemical functional group; Cellulose crystallinity; Thermal stability

Contact information: School of Logistics and Transportation, Central South University of Forestry and Technology, Changsha 410004, China; \*Corresponding author: 3441296495@qq.com

## INTRODUCTION

China is a large agricultural country with abundant agricultural and forestry resources, and it is both a major textile producer and a major textile consumer. The application of textiles covers almost all areas of the production and lifestyle of an individual. As living and consumption levels increase and the textile life cycle shortens, a large number of discarded textile materials are produced (Lin et al. 2016; Ma et al. 2018). Statistics show that annual global textile waste is more than 30 million tons, which mainly includes scraps generated by chemical action and mechanical action in the textile process: waste varn, varn return, and scrap produced during the cutting process in textile production, as well as discarded textile fibers and their products that are produced in the lives and activities of residents (Mishra et al. 2014). Most textile waste materials are directly incinerated or buried, and only a minimal portion is recycled into waste resources (Li et al. 2017). Consumers discard approximately 5.8 million tonnes of textiles every year in the European Union. Only 25% of these textiles are recycled by charities and industrial enterprises, while the remaining textiles are distributed to landfills or municipal waste incinerators, which causes serious environmental pollution (Barbero-Barrera et al. 2016). However, landfill treatment requires a large amount of space, and textile waste contains a large amount of chemical fibers that cannot be naturally degraded. In particular, incineration produces a large amount of smoke and toxic gases that cause serious environmental pollution (Reis 2009). Therefore, to save resources and protect the environment, the recycling of textile waste has gradually become a research hotspot in the field of textile materials.

At present, the recycling process of textile waste materials mainly includes recycling the product to its original state, regenerating the product into new products with lower physical, mechanical, and chemical properties, converting waste into basic chemical materials and fuels, and utilizing thermal energy from the combustion of fiber solid waste (Wang 2006). However, research on converting recycled and reused waste textile materials (biomass composites, pallets, logistics packaging boards, etc.) into new products, such as fiber-reinforced concrete as building materials and textile waste as insulation material, is still ongoing (Algin and Turgin 2008; Binici et al. 2009; Binici et al. 2010; Akadiri and Olomolaiye 2012; Briga-Sá et al. 2013). Furthermore, Bajwa et al. (2011) used waste materials, such as cotton stalks and cotton linters from ginners, as reinforcements to reinforce thermoplastic composites. Tasdemir et al. (2007, 2008, 2010) added different lengths of discarded silk fibers and waste cotton fibers to high-density polyethylene and polypropylene polymers in a certain proportion and prepared composite materials using a twin-screw extruder, which improved the mechanical properties and inherent chemical bonds of the composite materials. Some researchers have also used carpet waste to prepare carpet-polymer resin composite sheets through surface treatment methods or other chemical means to enhance the mechanical properties of the composite (Bateman and Wu 2001; Lin et al. 2007; Jain et al. 2010). In addition, other researchers have examined the preparation of textile waste in unsaturated polyester resin (Jurumenha and Dos Reis 2010; Rimdusit et al. 2011).

In conventional processes, environmentally friendly biomass composites are made from lignocellulosic fibers combined with synthetic adhesives, such as phenol formaldehyde (PF), and then bonded together under heat and pressure (Hüttermann *et al.* 2001). Melamine-urea-formaldehyde (MUF) resin has some advantages compared to PF resin plywood, such as a lower price, higher bonding strength, and shorter processing time (Kim and Kim 2005; Aydin *et al.* 2006; Oh and Kim 2011). Some studies have reported that MUF resin could potentially be used to enhance water resistance and reduce formaldehyde emissions because of the material's high functionality and the stability of its molecular structure (No and Kim 2004, 2007; Murata *et al.* 2013; Zhang *et al.* 2013). However, research on the preparation of artificial boards and other related products from textile waste has not been observed in recent years, except for Pinto *et al.* (2013), whose research centered on the use of textile wastes as fiber reinforcement as an alternative to polypropylene fibers (Chan and Bindiganavile 2010; Izaguirre *et al.* 2011; Islam *et al.* 2012) or other types of fibers, such as glass, basalt (Iucolano *et al.* 2013), or hemp (Le Troëdec *et al.* 2011).

Hence, this paper summarizes the results of the research conducted to evaluate the potential use of textile fiber wastes in composite boards. Discarded cotton was prepared into veneers of different thicknesses, and these veneers were bound with MUF adhesive in the case of the same total mass of cotton of boards. The main objective of this study was to investigate the mechanical properties, chemical structure, intrinsic microscopic appearance changes, and thermal properties of cotton boards. In addition, these results were compared with the physical properties of particleboard (PB) according to China national standard GB/T 4897 (1992) and medium-density fiberboard (MDF) according to China

national standard GB/T 11718 (2009) with the same proportion of adhesive to obtain the best processing technology of cotton boards.

## EXPERIMENTAL

#### Materials

Cotton fibers were collected from the Guangzhou Xintang Jeans Factory, Guangzhou, China by ripping discarded cotton jeans, and the cotton fiber was floc-shaped of 10 to 20 mm in length. The MUF adhesives were purchased from Xiamen Taier Chemical Co., Ltd., Xiamen, China. The solids content and pH value of the MUF adhesive were 65.0% and 7.77, respectively, at room temperature. The foil paper was purchased from the supermarket and was mainly used as release paper.

#### Preparation of cotton boards

Every cotton board (thickness 5 mm) had the same weight of cotton fibers and MUF adhesive but had a different number of layers, and the cotton veneers were brushed with MUF adhesive. Different weights of cotton fibers were sprayed with 40 wt% water and then hot-pressed (Dongguan Huajia Hydraulic Machinery Manufacturing Co., Ltd., Guangzhou, China) into veneers under a pressure of 6 MPa at 140 °C. To exclude the internal moisture of the veneers during the hot-pressing, the pressure was lowered to 0.5 MPa for 2 min after 5 min of hot-pressing, and then the veneers were pressurized for 10 min to obtain the cotton veneers. Before producing the cotton boards, the veneers for every cotton board were stacked together and cold-pressed for 2 h so that the adhesive could fully penetrate the veneers. Then, the cotton boards were hot-pressed under the same process conditions used to prepare the cotton veneers. The experimental design is presented in Table 1.

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Samples	Weight of Each Veneer (g)	Number of Layers	MUF (g)
CB1	48	5	100
CB2	60	4	100
CB3	80	3	100

Table 1. E	Experimental	Design
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## Methods

#### Physical properties analysis

The static bending strength, internal bond strength, and thickness expansion degree of cotton boards after water absorption were tested in accordance with the Chinese national standard GB/T 17657 (2013).

#### Scanning electron microscopy (SEM)

A scanning electron microscope (model Philips XL 30; Japan Hitachi Company, Tokyo, Japan) was used to evaluate the sectional morphology of the different layers of the cotton boards and the surface morphology of cotton fibers and veneers. The samples were placed on the stub and incubated in an oven at 60 °C. The test specimens were then gold coated using a vacuum sputter coater (model SC 500; Japan Hitachi Company, Tokyo, Japan) before analysis. The thickness of the gold layer was ca. 0.01  $\mu$ m to 0.1  $\mu$ m. The accelerating voltage was fixed at 15 kV.

#### Fourier transform infrared (FTIR) spectroscopy analysis

The powder of the cotton fibers, veneers, and boards was used for FTIR analysis. The FTIR spectra of the samples were obtained on an FTIR spectrophotometer (IR100; Thermo Fisher Scientific, Massachusetts, America) using KBr discs containing 99% KBr and 1% sample powder.

#### X-ray diffraction (XRD) analysis

After preparation, the samples were examined using an XD-2 diffractometer (Beijing General Instrument Co., Ltd., Beijing, China) with Cu radiation ( $\lambda = 1.5406$  nm), 36 kV voltage, and 20 mA current. The  $2\theta$  value was scanned continuously with a linkage scanning system (rotary half-cone  $2\theta$ ) from 5° to 45°, at a scanning velocity of 4°/min and a scan step of 0.01°. A graphite crystal monochromator was used, with slit device widths of divergence slit = 1°, anti-scattering slit = 1°, and receiving slit = 0.3 mm.

#### Thermogravimetric/ Differential thermal gravity (TG/DTG) analysis

Less than 10 mg of the powder of cotton fibers, veneers, and boards were crushed to 100- to 200-mesh and air-dried prior to use. The TG spectra were measured from room temperature to 680 °C on a TG20 thermal gravimetric analyzer (209-F1 TG; Netzsch, Selb, Germany) using a carrier gas (N<sub>2</sub>) velocity of 40 mL/min and a heating rate of 20 °C/min.

# **RESULTS AND DISCUSSION**

#### **Mechanical Properties of Cotton Boards**

The mechanical properties of the cotton boards are presented in Figs. 1 and 2, and the values shown for PB and MDF in Fig. 1 and 2 are the minimum values for those standard products. The static bending strength and internal bond strength of the cotton boards produced with the four-layer cotton veneers were better than those of the cotton boards produced with the five- and three-layer cotton veneers. For example, at the same cotton fiber content, the static bending strength of the four-layer cotton boards was 59.3 MPa, while these properties were 48.4 and 41.0 MPa for the five-layer and three-layer cotton boards, respectively. Similar results were obtained from the internal bond strength tests.

Compared to PB and MDF, all cotton boards that were produced with different cotton veneer layers showed obvious increases in static bending strength and internal bond strength properties, and all exceeded the minimum standards for PB and MDF. There were no obvious differences in the internal bond strength of the four-layer and five-layer cotton boards, but this property was noticeably better than that of the three-layer cotton boards. Compared to the three-layer cotton boards, the four-layer cotton boards showed increases in the static bending strength and internal bond strength of 18.31 and 0.17 MPa, respectively, which was mainly because the thicker veneers were not conducive to the penetration of adhesives during cold-pressing, such that they could not be completely combined when the cotton veneer was hot-pressed. Notably, the four-layer and five-layer cotton boards had similar internal bond strengths because the adhesive fully penetrated the cotton veneers of the four-layer and five-layer cotton boards.



**Fig. 1.** The static bending strength of cotton boards; PB: particleboard, MDF: medium-density fiberboard, CB1: five-layer cotton boards, CB2: four-layer cotton boards, CB3: three-layer cotton boards; the physical properties of PB and MDF were referred to in the China national standards GB/T 4897 (1992) and GB/T 11718 (2009)



**Fig. 2.** The internal bond strength of cotton boards; PB: particleboard, MDF: medium-density fiberboard, CB1: five-layer cotton boards, CB2: four-layer cotton boards, CB3: three-layer cotton boards; the physical properties of PB and MDF were referred to in the China national standards GB/T 4897 (1992) and GB/T 11718 (2009)

#### Waterproof Performance of Cotton Boards

The waterproof performance of the cotton boards is presented in Fig. 3. The thickness expansion percentages of water absorption for the five-layer and four-layer cotton boards were 6.38% and 5.64%, respectively, which were obvious lower than that for the three-layer cotton board (10.9%). Compared with PB and MDF, the five-layer and four-layer cotton boards showed excellent waterproof performance; notably, the thickness expansion rate of water absorption for the five-layer and four-layer cotton boards decreased 28.62% and 29.36%, respectively. The waterproof performance of the four-layer cotton

board was slightly better than that of the five-layer cotton board, mainly because the fourlayer cotton board had a better internal bond strength and a better combination of adhesive and fibers. However, the thickness expansion rate of water absorption for the three-layer cotton board was worse than that for PB, mainly because thicker veneers are not conducive to the penetration of adhesives during cold-pressing and cannot be completely combined when the cotton veneer is hot-pressed, such that more moisture was absorbed into the crosssection of the three-layer cotton boards.



**Fig. 3.** The thickness expansion percentage of water absorbing of cotton boards; PB: particleboard, MDF: medium-density fiberboard, CB1: five-layer cotton boards, CB2: four-layer cotton boards, CB3: three-layer cotton boards; the physical properties of PB and MDF were referred to in the Chinese national standards GB/T 4897 (1992) and GB/T 11718 (2009)

## SEM Analysis of Cotton Fibers, Veneers, and Boards

The visual macroscopic evolution of cotton fibers after each stage of treatment is shown in Fig. 4. The morphology and structure of the external aspect and traversal cross-section of the cotton fibers were characterized using SEM.

The image in Fig. 4a shows that a single cotton fiber was relatively clean and free of impurities. After hot-pressing with water, the fibers were intertwined to form a dense network structure (Fig. 4b), which was the main reason why cotton fiber could be combined without adhesive. Figures 4c, 4d, and 4e show the microscopic appearance of the cotton boards with the same quality but different layers. It was clear that the cotton boards with more layers had a denser structure, and the gap between each layer was also obviously reduced. In addition, there was more adhesive that stuck to the fiber on the five-layer and four-layer cotton board than on the three-layer cotton board. These factors explain why the thicker veneers were not conducive to the penetration of adhesives after cold-pressing and resulted in insufficient bonding strength in the middle of the veneer on the cotton board. Therefore, it is essential to study the thickness of the veneer, which is the key link that determines the strength of bonding in the sheet.

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Fig. 4. Scanning electron micrographs of cotton fibers, veneers, and boards

## FTIR Analysis of Cotton Fibers, Veneers, and Boards

In this experiment, the main component of the cotton fibers was cellulose, which was slightly different from the natural fibers. The chemistry of the cotton fibers, cotton veneer, and cotton boards was identified using FTIR, as shown in Fig. 5. Infrared spectroscopy (FTIR) has proven to be useful for the analysis and identification of functional groups of polysaccharides in plant materials. FTIR spectroscopy also yields information about the chemical composition, molecular conformation, and hydrogen bonding patterns of the cellulose allomorphs (Wang *et al.* 2009).

A wide absorption peak at  $3291 \text{ cm}^{-1}$  resulted from the stretching vibration of the aromatic and aliphatic O–H groups, and the absorption peaks at 2909 cm<sup>-1</sup> belonged to the C–H stretching vibration of CH<sub>3</sub>, –CH<sub>2</sub>–, and –CH– groups. The absorption peaks at 1638 cm<sup>-1</sup> and 1531 cm<sup>-1</sup> are C=C stretching vibrations of alkanes, and the formation of these two new absorption peaks was mainly due to the addition of MUF adhesives. The C–N stretching vibration of amines and amides was found at 1229 cm<sup>-1</sup>. The absorption band at 1016 cm<sup>-1</sup> was the C–O stretching vibration of aldehydes, ketones, carboxylic acids, and esters. In addition, the stretching vibration of C–I was found at 554 cm<sup>-1</sup>, maybe because the materials in the KBr disks were not mixed well, so that some of them acted like they had a higher concentration of cellulose, which led to absorption peaks at 554 cm<sup>-1</sup> representing the C–I stretching vibration (Pednekar and Raman 2013; Tomak *et al.* 2013; Xu *et al.* 2013).

Through comparing all the absorption peaks of the cotton fiber, cotton veneer, and cotton boards, it was found that the absorption peaks of cotton veneer were slightly stronger than those of cotton fiber, especially the absorption peak at  $3291 \text{ cm}^{-1}$ , which indicated that new hydrogen bonds were formed between the cotton fiber after hot-pressing the cotton fibers with water. This result also provided a theoretical basis for fiber binderless bonding. From the FTIR spectra curves, it was clear that the absorption peaks of cotton boards were obvious enhanced, which showed that the MUF adhesive penetrated into the cotton veneer and merged with the cotton fibers to form chemical bonds. The new C–N stretching vibration at 1229 cm<sup>-1</sup> and the new C=C stretching vibrations at 1638 cm<sup>-1</sup> and 1531 cm<sup>-1</sup>

also demonstrated that the glue between cotton fibers was in good condition. In addition, from the viewpoint of the intensity of the absorption peaks, the five-layer and four-layer cotton boards had almost the same bonding effect, which was better than that of the three-layer cotton boards. The differences in the spectra of cotton fibers, cotton veneer, and cotton boards confirmed that there was hydrogen bonding in addition to physical and mechanical bonding in the cotton veneers, and thinner cotton veneers had better bonding effects of the adhesive and the cotton fibers.



Fig. 5. FTIR spectra of cotton fibers, veneers, and boards

## XRD Analysis of Cotton Fibers, Veneers, and Boards

Cellulose has a crystalline structure, contrary to hemicelluloses and lignin, which are amorphous in nature. According to Zhang and Lynd (2004), cellulose has a crystalline structure due to hydrogen bonding interactions and Van der Waals forces between adjacent molecules. The XRD analysis was performed to evaluate the crystallinity of the cotton fibers after different treatment steps (El Oudiani *et al.* 2004; Wan *et al.* 2015). Figure 6 shows the diffraction patterns obtained for cotton fibers, cotton veneers, and cotton boards. All diffractograms exhibited sharp peaks at approximately 14.8° and 22.7°  $2\theta$  angles, which can be attributed to the diffraction planes of (am) and (002), respectively. These features commonly represent typical cellulose I crystal forms (Lu and Hsieh 2010; Chen *et al.* 2011; Bettaieb *et al.* 2015a, 2015b). The Cr was the relative crystallinity and can be determined from Cr (%) = ( $I_{002} - I_{am}$ ) /  $I_{002} \times 100$ .

The peak intensity corresponding to the (002) lattice planes increased and became sharper as a result of the addition of MUF adhesive, which was related to an increase in the crystallinity of the material. As shown in Fig. 6, cotton fibers, veneers, and boards displayed similar diffraction patterns as that of the control. The  $I_{am}$  of the cotton fibers, cotton veneer, and cotton board (5-layer, 4-layer, and 3-layer) samples were obtained as 864 cps, 870 cps, 520 cps, 1112 cps, and 666 cps, respectively. The  $I_{002}$  of the cotton fibers, cotton veneer, cotton board (5-layer), cotton board (4-layer), and cotton board (3-layer) were 2192 cps, 2208 cps, 2039 cps, 4349 cps, and 2481 cps, respectively.

The Cr for the cotton fibers, cotton veneer, and cotton board (5-layer, 4-layer, and 3-layer) samples were 60.6%, 60.6%, 74.5%, 74.4%, and 73.2%, respectively. There were minor changes in the Cr of cotton veneer after hot-pressing the cotton fibers with water. This also provided a theoretical basis for fiber binderless bonding. It was possible that the presence of the dye in cotton fibers had a chemical effect during hot-pressing. The differences in the spectra of the cotton fibers, cotton veneer, and cotton boards confirmed that there was hydrogen bonding in the cotton veneer besides physical and mechanical bonding, and thinner cotton veneers had a better bonding effect of the adhesive and the cotton fibers. After bonding the cotton veneers with MUF adhesive, the Cr of the cotton board (5-layer, 4-layer, and 3-layer) samples obvious increased, and most of the amorphous regions were removed, which led to an obvious increase in crystallinity for the cotton boards. Thus, it would be assumed that the potential mechanical properties and reinforcing capability of the treated fibers should have increased (Rong et al. 2001; Bhatnagar and Sain 2005). This increase in the crystallinity of the cellulose fibers also enhanced their stiffness and rigidity and therefore their strength. As the number of layers increased, the crystallinity of cotton boards also increased, which indicated that a thinner cotton veneer resulted in a better bonding effect of the adhesive and the cotton fibers. However, the five-layer and four-layer cotton boards had almost the same crystallinity.



Fig. 6. XRD spectra of cotton fibers, veneers, and boards

#### TG/DTG Analysis of Cotton Fibers, Veneers, and Boards

The thermogravimetric analyzer (TGA) is an essential laboratory tool used for material characterization, and its derivative (DTG) curves present the weight loss rates (Gedemer 1974). The TGA/DTG was used to characterize the materials used in various environments. The weight loss in the cotton fibers, cotton veneer, and cotton boards occurred due to the degradation of cellulose, hemicellulose, lignin constituents, and MUF adhesive during heating. The TGA and DTG curves under a nitrogen atmosphere for cotton fibers, veneer and boards are presented in Figs. 7 and 8, respectively. From the TGA thermograms, the degradation profiles of such natural materials were distinctly characterized by three weight loss steps (Jakab *et al.* 1997).

The initial mass loss below 100 °C was due to the gradual evaporation of absorbed moisture. The initial decomposition temperature for the composite is typical of cotton fibers, in which the pyrolysis of cotton fiber components occurred above 200 °C. After 250 °C, the weight loss rate quickly increased, and two degradation steps were distinguished. The first step corresponded to the decomposition of cotton fiber components, such as cellulose, hemicellulose, lignin, and extractives. The maximum rate of decomposition temperature of the second mass-loss step was at approximately 370 °C. The mass losses of the cotton board (5-layer, 4-layer, and 3-layer) samples were 62.0%, 61.7%, and 72.9% at 360 °C, 380 °C, and 376 °C, respectively. These results indicated that the thermal stability of the 4-layer cotton board was better than that of the 5- and 3-layer cotton boards. The third degradation step starting at a temperature above 380 °C was due to the decomposition of the MUF adhesive. Above 380 °C, lignin degradation can also occur, which is common in the structure of lignocellulose (Mattos *et al.* 2014).



Fig. 7. TG spectra of cotton fibers, veneers, and boards

Although cotton fibers and cotton veneers have obviously higher degradation temperatures than cotton boards, their residue mass were 14.3% and 19.7% at 423 and 449 °C, respectively, which were much lower than those of cotton boards. The higher mass loss (lower residue mass) of cotton fibers than that of cotton veneers might be mainly attributed to the hot-pressing process, since the interactions (especially hydrogen bonds) between the cotton fibers are formed during the hot-pressing. Additionally, cotton boards have higher mass residues than cotton fibers and veneers, higher residual mass can usually be attributed to the formation of char. Often this is due to the presence of compounds (maybe the adhesive) that decomposes at a low enough temperature so that it is able to participate in reactions that form highly aromatic structures that are resistant to high heating (Fig. 7) (Alemdar and Sain 2008). The DTG curves of the cotton fibers, veneers, and boards are displayed in Fig. 8. For the cotton fibers and cotton veneers, the maximum decomposition rates (VMs) were 1.59%/°C and 1.48%/°C, respectively. The VMs of the cotton boards (5layer, 4-layer, and 3-layer) were 0.69%/min, 0.70%/min, and 1.37%/min, respectively, which were obviously lower than those of the cotton fibers and veneers. The lower VMs of the cotton boards than those of the cotton fibers and veneers may be also because of the

higher crystallinities of the cotton boards that endow more stable structure (Alemdar and Sain 2008).



Fig. 8. DTG spectra of cotton fibers, veneers, and boards

# CONCLUSIONS

- 1. The mechanical and waterproof properties of cotton boards with different layers (3, 4, and 5 layers) were better than those of particleboard (PB) and medium density fiberboard (MDF) according to Chinese national standards GB/T 4897 (1992) and GB/T 11718 (2009) for the same proportion of adhesive. Also, the four-layer cotton boards performed better than the five-layer and three-layer cotton boards.
- 2. The SEM analysis showed that the fibers were intertwined to form a dense network structure after hot-pressing with water, and thicker veneers were not conducive to the penetration of adhesives after cold-pressing.
- 3. The FTIR spectra indicated hydrogen bonding in the cotton veneers in addition to physical and mechanical bonding and stronger absorption peaks of the chemical functional groups of the five-layer and four-layer cotton boards.
- 4. The XRD spectra showed that the cellulose crystallinity of the cotton boards (3, 4, and 5 layers) increased to 74.5%, 74.4%, and 73.2%, respectively, but the fibers and veneers only increased to 60.6% and 60.6%, respectively.
- 5. The TG/DTG curves showed that the thinner cotton veneers of cotton boards showed higher levels of char residue.
- 6. This finding indicated that this material exhibits encouraging perspectives for the production of biomass composites.

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