

UTILIZATION OF BAGASSE FIBER FOR PREPARATION OF BIODEGRADABLE FLAME RETARDING COMPOSITES (BFRCs)

Wenjia Han,^a Kefu Chen,^a Ren-Dang Yang,^{a,*} Fei Yang,^a Chuanshan Zhao,^b and Wenhua Gao^b

Bagasse is a renewable resource characterized by its low cost and environmental friendliness. In this work a novel technological process was proposed to make flame retarding composites (BFRCs) by using bagasse fiber. The bagasse was disintegrated by twisting it up and applying high consistency refining, and then it was used to prepare BFRCs via hot pressure. Chemical groups and thermal properties of bagasse fiber were studied through the use of FTIR spectroscopy, a universal mechanical testing machine, and TGA, while properties of BFRCs were also analyzed by SEM, and the surface water resistance and burning characteristics were measured. Results showed the pyrolysis temperature of bagasse fibers to be about 273°C. Chemical groups were not changed, while the content of groups was reduced a little during the manufacturing process. The BFRCs showed good performance for water resistance, and the optimum value was 1.7g. They also had good flame retardant performance. The index of flame spread was 13.6 and the smoke index was 108, which reaches Class A by the ASTM E84-08 Standard.

Keywords: Bagasse; Fiber; Biodegradable flame retarding composites (BFRCs); Thermoforming; Properties

Contact information: a: State Key Lab of Pulp and Paper Engineering, South China University of Technology, Guangdong Public Laboratory of Paper Technology and Equipment, Guangzhou, China; b: Key Lab of Paper Science and Technology of Ministry of Education, Shandong Institute of Light Industry, Ji'nan, China; *Corresponding author: rdyang@scut.edu.cn

INTRODUCTION

Sugarcane bagasse, one of the largest cellulose agro-industrial by-products of the sugar and alcohol industry, is a widely available renewable resource (Satyanarayana et al. 2007). About 54 million tons of dry bagasse is produced annually throughout the world (Mulinari et al. 2009). It is usually used as a fuel for the sugar factory, but the heating value is low. It has also been used as material for papermaking, as a biomass source, and as a sorbent for wastewater treatment, but only a small portion of the material has been treated so that it can be used for a second time (Huang et al. 2008; Khristova et al. 2006; Hernandez-Salas et al. 2009; Rao et al. 2002). The use of bagasse in the preparation of composite material has recently become a hot research topic around the world (Bhattacharya et al. 2009; Liu et al. 2006; Sun et al. 2004). As a natural polymer, the use of natural fiber offers environmental advantages such as reduced dependence on non-renewable sources (Joshi et al. 1999), degradability, and suitability for combustion as part

of a carbon-neutral cycle. Such superior environmental performance is an important driver for the increased future use of natural fiber composites.

Composite materials are commonly selected based on functional and financial requirements, which currently mostly favor production of composites from non-renewable resources (Vanden et al. 2006). However, recently more attention is being paid to biodegradable composites and environmentally friendly characteristics during the complete life-cycle of production and usage (Worapon et al. 2009; Princi et al. 2006). The properties of resource impact, energy efficiency, and water efficiency, along with the impacts on human health, are all considered. Biodegradable composites have some disadvantage relative to retarding of inflammation, and this drawback may restrict their further applications in some fields. People have become increasingly interested in fire-retardant natural plant fiber composites (Liu et al. 2008; Marney et al. 2004; Marney 2008). BFRCs are one kind of biodegradable flame retarding composites.

In this study a variety of BFRCs was developed with bagasse, waterproofing agent, retention aid, and reinforcing agent. Factors affecting mechanical, waterproof, and flame retardant properties were tested. In addition, to better understand the mechanisms, the effects of chemical changes were examined by thermal analysis and FT-IR, and the structure of BFRCs was analyzed by SEM.

EXPERIMENTAL

Materials and Chemicals

The bagasse used in the laboratory trials was kindly supplied by Sichuan Ya'an Pulp Co. Ltd. (Sichuan, China). The initial moisture content was about 10%. The flame retardant chemical (FR-5) was purchased from JinGe Fire & Emergency Manufacturers Co., Ltd. (Guangdong, China). The waterproofing agent (AKD-5), retention aid (CPAM-II), and reinforcing agent (Cationic starch, SM633) were all purchased from Bai Si Te Fine Chemical Co. Ltd (Guangdong, China).

Preparation of the BFRCs

Mechanical pulping

The bagasse was treated by alkali at the optimum condition of 25% consistency, 2% of alkali, and 80°C for 30 min. Then the pretreated bagasse was twisted up by an extruder and defibered with a high consistency refiner at the concentration of 25%, with two passes. The equipment was provided by the pulp molding laboratory in South China University.

Thermoforming

The experimental device for preparation of the BFRCs used in this study was a fully automatic pulp molding machine (provided by State Key Lab of Pulping and Papermaking of SCUT). The machine consists of a cool mold as shown in Fig. 1, two hot molds as shown in Fig. 2, and other auxiliary equipment such as an air compressor, vacuum system, and so on. Figure 3 shows a detailed structural schematic for the mold.

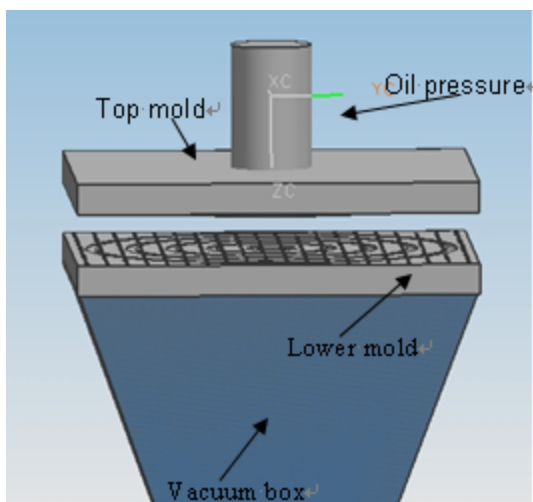


Fig. 1. One type of cool mold

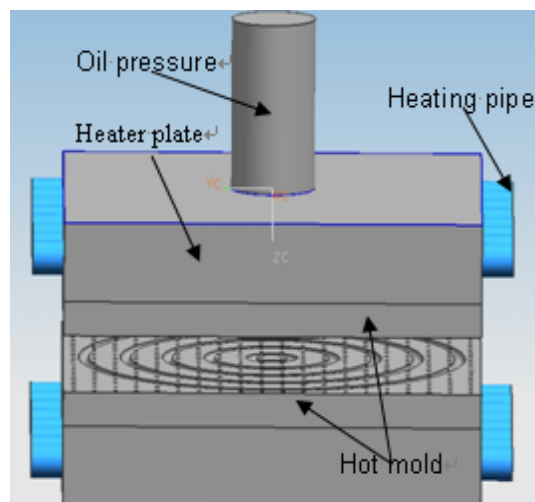


Fig. 2. One type of hot mold

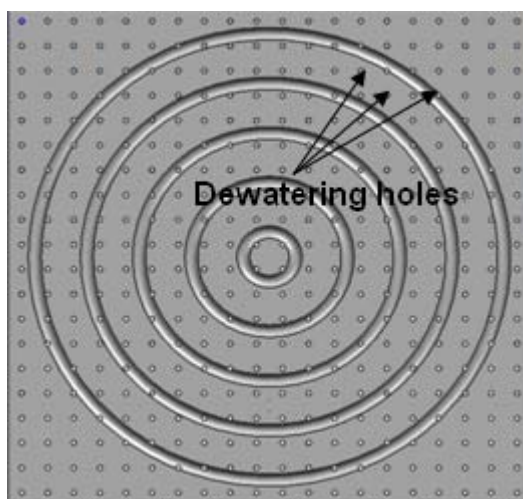


Fig. 3. Detailed structure of mold

A large number of 2 mm diameter spray-holes were drilled in the cool mold to allow water to escape, and in the hot mold to permit steam flow. The distances between two adjacent holes depended on the mold shape. During the experiment with a cool mold a maximum pressure of 40 kN and a vacuum degree of 0.8 bar were generated. The maximum pressure of the hot mold was 60 kN.

The pre-model was formed by dewatering in the forming zone under a specific vacuum degree. A wet-model with 60% to 70% moisture content took shape after the pressing by the cool mold. Then the wet-model was transferred from the cool mold to the hot mold to be simultaneously to be dried and pressed for about 2 min to 6 min at 120°C to 200°C and a pressure of 1 kN to 60 kN.

The bagasse fiber was dispersed in a large amount of water at a concentration of 3 percent. A certain content of flame retardant (0-50% relative to oven dry bagasse fiber), waterproofing agent (3% relative to oven dry bagasse fiber) and reinforcing agent (2% relative to oven dry bagasse fiber) were sequentially added into the pulp. Its components were distributed during formation by mixing.

During formation, a specified amount of pulp was injected into the grooved assembly, and it was agitated in air. Then the pulp was dehydrated through the small holes in the cold mold under negative pressure. The wet-formed product was pressed into the cool mold. At this step, we obtained a wet-formed body with water content of about 30%.

During drying, the product was transferred from the cool mold to the hot mold and hot pressed immediately. The drying process was completed under the conditions of 160°C and 30 MPa for 4 min. After a specific holding time, the BFRCs was released from the hot mold. The resulting sample was a square plate with all sides 450 cm in length. The thickness was 2 mm. It was cooled in air for 24 h. One kind of hot mold is shown in Fig. 2.

TGA Analysis

Thermo-analytical studies, using a NETZSCH TG209 Jupiter Thermal Analysis System that combined TGA measurements, were conducted. Thermo-gravimetric analysis (TGA) was used to study the thermal stability. The weight loss was recorded in the range from room temperature to 700°C with a heating rate of 10 °C min⁻¹. The carrying gas was N₂, and the flow rate was 10 mL min⁻¹.

Fourier Transform Infra-red (FTIR) Spectroscopy

IR spectroscopy was carried out in transmission mode using macro techniques (13mmΦ pellet; ca.1.5mg sample with 350mg KBr). The spectra were recorded with a Nexus Vector spectrometer made by Thermo Nicolet under the following specifications: apodization: triangular; detector: DTGS/KBr; regulation: 4 cm⁻¹; number of scans: 32 (Faix 1971; Michel 1988).

Surface Water Resistance

A working temperature of 21±2°C and a humidity of 50±5% were achieved for evaluation of the test samples. The specimens were cut into the following size: 127 mm×127 mm. The test method was according to ASTM C473-03 Standard Test Methods for Physical Testing of gypsum Panel Products.

The conditioned test specimens were placed in the Cobb Tester with the side to be exposed to water face up. The Cobb ring was filled with tap water. After 2 min had elapsed, the water was poured out of the Cobb ring and the specimen was removed from the Cobb tester. Immediately the excess water was removed from the specimen by blotting, and the specimen was weighed to the nearest 0.05 g. The average mass difference between dry and wet weights of the test specimens was determined to the nearest 0.1 g, and this was reported as the surface water absorption of the panel product.

Mechanical Testing

Static compression tests were carried out at room temperature of 20 °C and a relative humidity of 60%, using a computerized Universal Mechanical Testing Machine operated at a speed of 10 mm·min⁻¹. The tests included both compressive stress-strain and elastic modulus under static compression. The length and width of a sample were 100 mm and 20 mm.

Burning Characteristics

Test samples were conditioned before testing, until a constant mass was achieved at the condition of the temperature of $23\pm 2.8^{\circ}\text{C}$, the humidity $50\pm 5\%$ for 18 days. The test method was according to ASTM E84-08 standard for surface burning characteristics of a sample.

SEM Analysis

The morphologies of the fracture surfaces were examined with a scanning electron microscope (SEM, Philips XL-30 ESEM). The samples of cellulose fibers and the BFRCs were coated with gold, using a vacuum sputter-coater, before observation.

RESULTS AND DISCUSSION

Functional Group Analysis of Bagasse Fiber

The nature of the fiber that had been treated at the different temperatures of 120°C , 160°C , and 200°C was investigated by FTIR spectroscopy. We obtained similar infrared spectra, one of which is shown in Fig. 4 as an example.

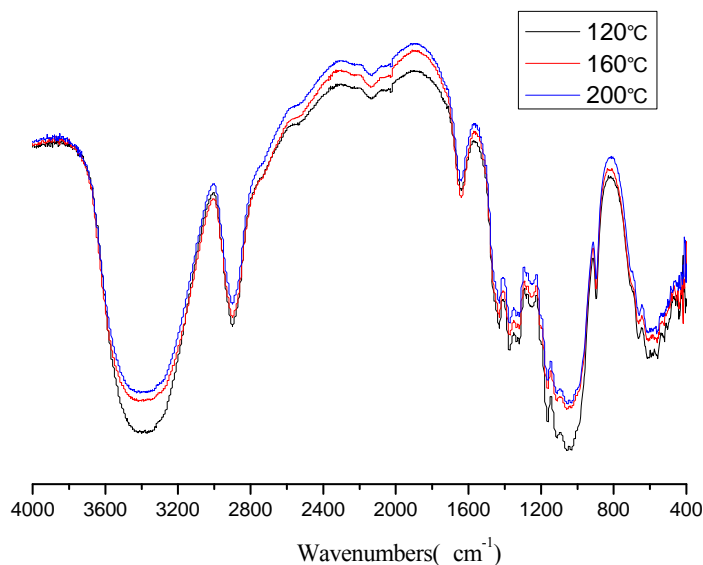


Fig. 4. FTIR spectra of bagasse fiber after being exposed to different temperatures

The FTIR spectra for the samples showed a characteristic absorption around 1163 to 1060 cm^{-1} , stretching bands that are attributable to the structure of aralkyl ether linkages. Every macromolecule of cellulose is composed of glucose molecules linked by glucosidic bonds. The position of 1373 cm^{-1} corresponds to flexural vibrations of C-H. The characteristic absorption around 1634 cm^{-1} is for C-O stretching bands, the characteristic absorption peak of lignin. The characteristic absorption around 1428 cm^{-1} is attributable to a scissoring vibration of CH_2 . The peak at 2900 cm^{-1} indicates the absorption peak of the saturated C-H. The 3400 cm^{-1} peak was attributed to hydroxide

groups (OH) among the fibers (Liu et al. 2008; Horikawa et al. 2008; Meireles et al. 2008). The characteristic peaks of fiber exhibited a small reduction with increasing temperature. The chemical groups of fiber were not changed during drying. Thus, the fiber can maintain its natural performance.

Thermal Degradation of Bagasse Fiber

The thermogravimetric curve and DTG curve are shown in Fig. 5 for the bagasse fibers subjected to the heating rates of $10\text{ }^{\circ}\text{C min}^{-1}$.

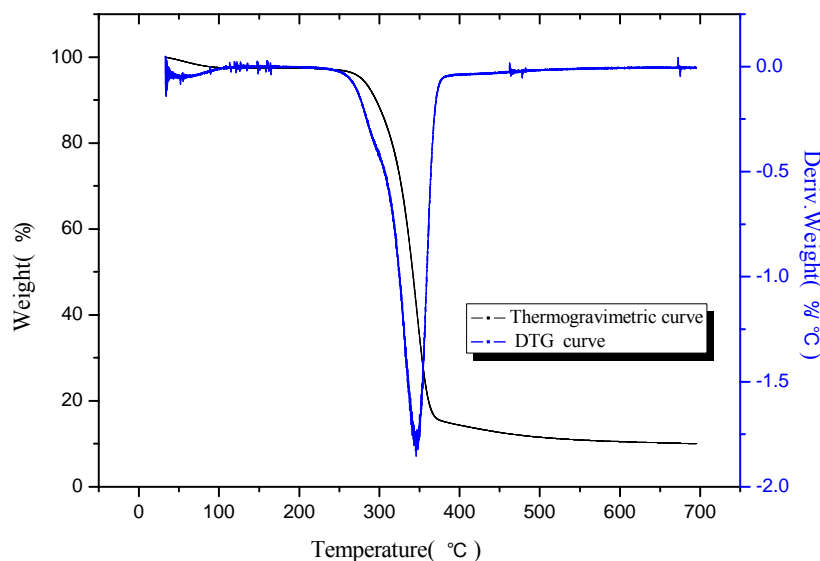


Fig. 5. Thermogravimetric and DTG curves of bagasse under the heating rates of $10\text{ }^{\circ}\text{C min}^{-1}$

As shown in Fig. 5, the weight losses of samples in nitrogen atmosphere that occurred within the range from room temperature (30°C) to 200°C was 2.60%, with heating rates of $10\text{ }^{\circ}\text{C min}^{-1}$. In this stage, the weight loss was due to the elimination of absorbed or combined water (Tomczak et al. 2007). The greatest decomposition of cellulose fibers occurred at a temperature around 273°C . The combined weight loss from 300°C to 400°C was 73.97% for the above heating rate, which resulted from the thermal degradation of cellulose, hemicellulose, and lignin. The results show that the weight loss first increased and then was reduced in a specific temperature range with the increasing temperature.

The curve of the derivative of weight vs. temperature is also shown in Fig. 5. The curves represent the rate of thermal decomposition. We can see that the rate was nearly zero up to about 240°C . The maximum pyrolysis rate was observed at 347°C . Above all, the results confirm that the bagasse fibers were not decomposed during the experimental drying.

Mechanical Characteristics of BFRCs

The sample was prepared under the optimum conditions of drying at 160°C for 4 min with 30 KN pressure at a 30% content of flame retardant.

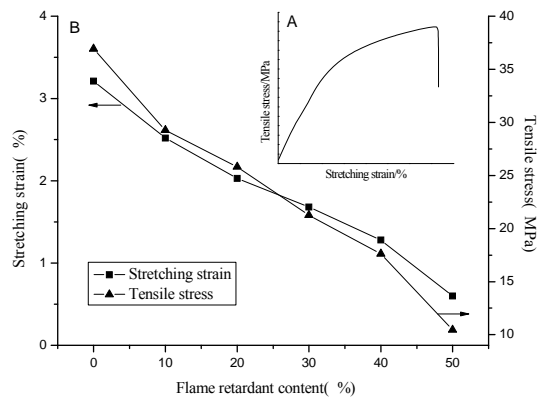


Fig. 6. Influence of flame retardant on mechanical properties

As can be seen from Fig. 6 (A), three compression stages in all etching stress-strain curves were observed. First, stress increased gradually or even linearly with the increase of strain. This is the stage of elastic deformation, which is accordant with Hooke's Law of Elasticity. The slope of the curve stands for elasticity modulus. Subsequently, the curves entered another stage of distortion which included plastic distortion. The slope of the curve decreased with increasing stress. Finally, the inflexion of the curve was the cut-off point for plastic distortion crack initiation.

The corresponding stress-strain curves are shown in Fig. 6 (B). The maximum tensile strength and stretching strain decreased with an increase of the content of flame retardant. This can be explained by the fact that the addition of flame retardant inhibits the formation of hydrogen bonding between the fibers to a certain degree. When the amount of flame retardant reached 30%, fortunately, the values of maximum tensile stress and the stretching strain were still as high as 21.3 MPa and 1.68%, respectively. This is acceptable in real applications. In order to obtain the considerable flame-retardant and mechanical properties of the HFPs in the same time, 30% of flame-retardant was selected for use in the following experiments.

Surface Water-Resisting Property of BFRCs

The influence of flame retardant and drying temperature on surface water-resisting property are shown in Figs. 7 and 8. It is shown in these figures that the water absorption weight increased with increasing levels of flame retardant and drying temperature. The addition of flame retardant increased the interstitial space between fibers, which affects the binding between fibers and the integration between the fibers and the waterproofing material. These influences result in the decreasing of surface water resistance properties. As noted, the higher drying temperature can increase the porosity of the sample, so it leads to an increase of water absorption, as determined by weight.

A dense structure of the sample and well associated property of every part contribute to improvements in surface water resistance. The value of surface water resistance was 1.7g under the condition of the optimum amount of 30% flame retardant and drying at 160°C for 4 min with 30 KN pressure. The contact angle of the sample under this condition is shown in Fig. 9, which shows good hydrophobic interfacial character.

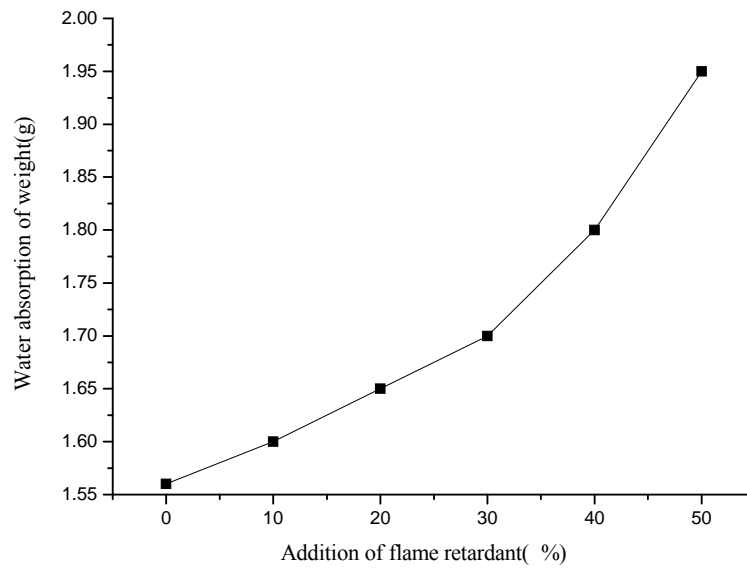


Fig. 7. Influence of flame retardant on water-resisting character

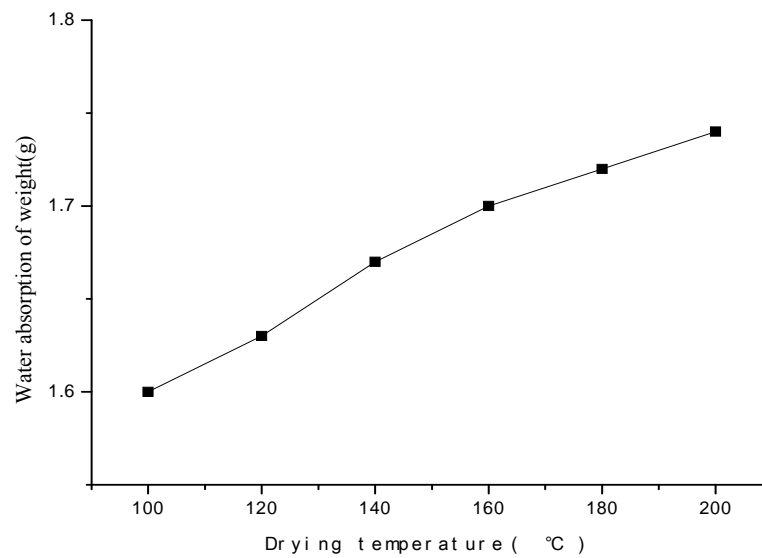


Fig. 8. Influence of drying temperature on water-resisting character

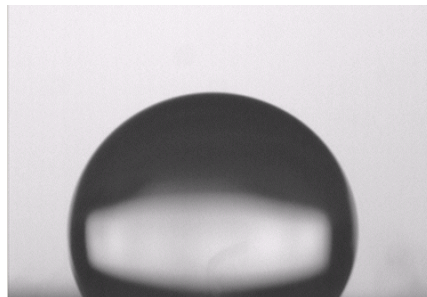


Fig. 9. Contact angle of sample

Burning Characteristics of BFRCs

Samples were prepared under the optimum conditions at 30% flame retardant, drying at 160°C for 4 min with 30 KN pressure. The resulting properties of window number, smoke, and air stream temperature (°C) are shown in Table 1.

Table 1. Data Related to Burning Characteristics Test

Time (min)	Window Number	Smoke Light Absorbed (%)	Air Stream Temp (°C)
0.0	0	0	41.1
0.5	0	8	92.8
1.0	1	9	98.9
1.5	1	11	107.2
2.0	1	14	116.1
2.5	1	20	123.9
3.0	2	32	130.6
3.5	2	28	138.9
4.0	2	27	142.8
4.5	3	25	143.9
5.0	3	23	143.9
5.5	3	21	145.0
6.0	4	17	145.0
6.5	4	14	145.0
7.0	4	10	145.6
7.5	4	7	146.7
8.0	4	8	146.7
8.5	4	9	147.2
9.0	4	7	147.8
9.5	4	8	147.8
10.0	4	7	148.3

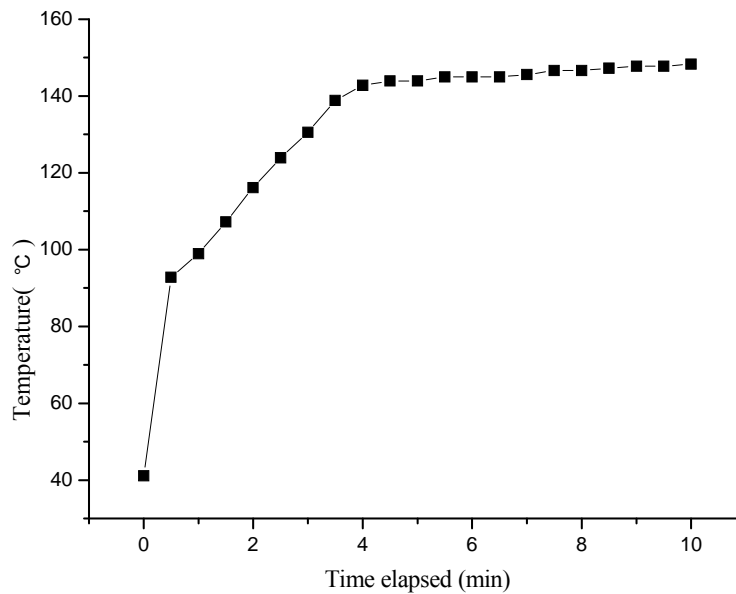


Fig. 10. Changes of air stream temperature with time

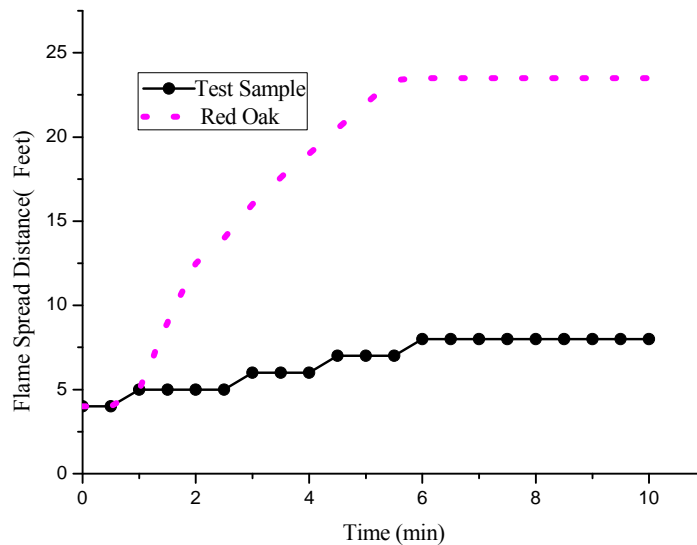


Fig. 11. Flame spread character with time

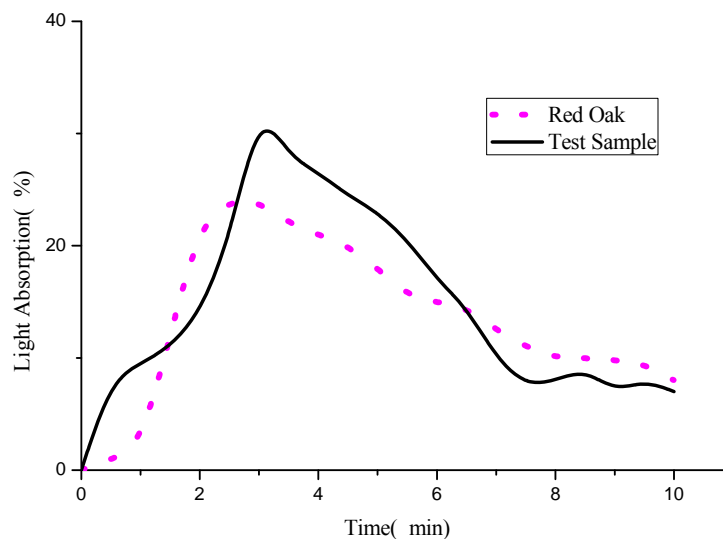


Fig. 12. Light absorption with time

In Fig. 10 one can see the curve of air stream temperature as it increased quickly during the first 30 seconds. Subsequently the air stream temperature reached 92.8°C with a slope of 185.6. In the next moment the temperature increased with a slope of 14.29 until 4 min, and the air stream temperature reached 142.8°C . The temperature changes were very small in the third stage from 4 min to 10 min.

The flame spread character is shown in Fig. 11. We know that the flame spread distance was similar between the sample and the reference red oak at less than one minute. After one minute, the FSD of the red oak expanded rapidly, and the entire FSD of the sample showed a slow rise. After 5.5 min, the FSD of the red oak reached a maximum 23.5 feet and it had reached a maximum 8 feet in 6 min for the test sample. Based on these results, we know that the sample exhibited better performance of flame spreading.

The smoke character of the sample was measured by light absorption. The two curves show that the light absorption first increased and then decreased, as seen in Table 1 and Fig. 12. The smoky character of red oak increased slowly during the first 30 seconds as compared to the test sample. After 2.5 min the maximum light absorption was 25%. From this point, the light absorption decreased slowly until a test time of 8 min, and the light absorption was about 10%. At the same time, the test sample curves showed a rapid increase for the first 30 seconds and then entered a slowly increasing stage from 0.5 minute to 1.5 min. Between 1.5 min and 3 min, the curve quickly increased and reached a maximum of 32.0%. After 3 min, the light absorption decreased slowly until a test time of 7.7 min, and the light absorption was about 7%.

We obtained the following experimental results for the test samples: The index of flame spread was 13.6 and the smoke index was 108. They were calculated from the experimental data by ASTM E84-08 Standard Test Methods. The properties of the test samples reached Class A.

The classifications are as follows according to ASTM E84-08 Standards:

- Class A Interior Wall & Ceiling Finish: Flame Spread 0-25, Smoke Developed 0-450.
- Class B Interior Wall & Ceiling Finish: Flame Spread 26-75, Smoke Developed 0-450.
- Class C Interior Wall & Ceiling Finish: Flame Spread 76-200, Smoke Developed 0-450.

SEM Analysis

The SEM images of the surface of BFRCs prepared with different magnification factors are shown in Figs.13 and 14.

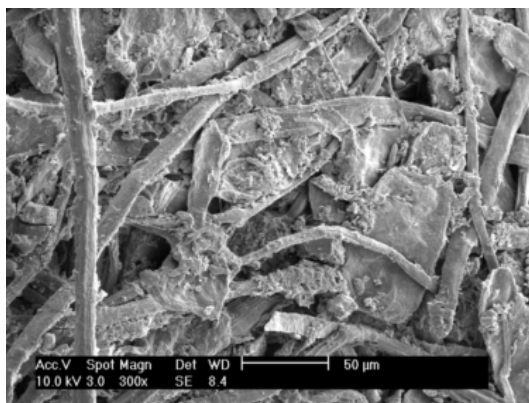


Fig. 13. SEM images of BFRCs 300x

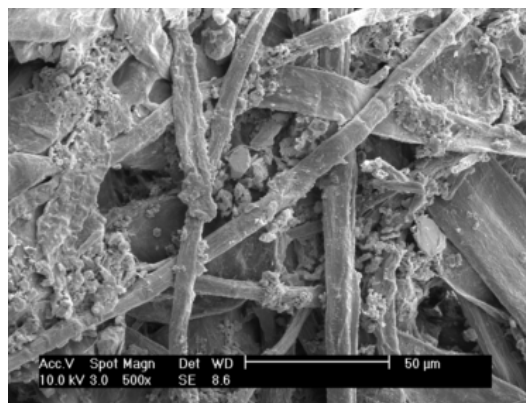


Fig. 14. SEM images of BFRCs 500x

The figures show the structure of BFRCs, revealing a network of fibers that gives structural support. The particles observed in figures are the flame retardant, which adsorbs on fibers or spreads between the fibers, giving a smoother surface morphology. The structure provides good access for the flame retardant during manufacture.

CONCLUSIONS

1. Bagasse can be disintegrated by twisting it up, followed by high consistency refining. When incorporated with correlative additives, the fibers can be used to prepare BFRCs via hot-pressing, with good flame retardancy, waterproofing, and environmentally friendly properties.
2. The bagasse fibers were not decomposed during the experimental drying by the analysis of TGA and FTIR. A great amount decomposition of cellulose fibers occurred at a temperature around 273°C.
3. The BFRCs exhibited suitable mechanical properties and good waterproofing character. The addition of flame retardants and higher drying temperature had adverse effects on waterproofing. The addition of flame retardants also had adverse effects on mechanical properties.
4. The BFRCs showed good performance of retarding flames. The property of flame retardancy reached Class A according to ASTM E84-08 Standards.

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