Flame retardant and Moisture Adsorption Behaviour of Kenaf/Flax/Coconut- Shell Biochar Based Hybrid Composites

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Abstract. This study examined the effects of biochar produced from coconut shells on the moisture absorption and flammability of flax and kenaf bio-composite epoxy hybrid. Three types of composites were developed using hand lay-up and compression molding for composites A, B, and C (2, 4, and 6 wt% biochar, respectively). Moisture absorption test, limiting oxygen index (LOI) test, vertical burning (UL-94) test, and cone burn test were performed on the composites. It was found that with each addition of biochar, composite C (6 wt% biochar) exhibited the greatest improvement in flame-retardant capability, with an LOI of 27.5%, a char yield rated as UL-94 V-0, and better performance in other comparative tests, including lower peak heat release and longer time to ignition. The C-type biochar reduced moisture absorption to the greatest extent due to the combined effect of complex diffusion pathways and improved matrix adhesion. The addition of 6 wt% biochar from coconut shells to sustainable multifunctional natural fiber composites increased fire resistance and moisture absorption durability.

1 Introduction

Natural fiber-reinforced composites are inexpensive and lightweight alternatives to synthetics. They are also widely available, easy to process, renewable, and environmentally friendly (biodegradable).

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Some studies have reported promising stiff and strong specific properties of flax and kenaf fibers (i.e., specific strength and specific modulus) [1]. Additionally, flax and kenaf fibers can potentially replace glass fibers in construction panels, packaging, and automotive interiors. Natural fiber composites are attracting more attention in many industries due to the increasing demand for eco-friendly alternatives. There is also the growing interest in the use of bio-based reinforcement to composites due to its close alignment with the areas of focal concern of most industries [2]. Nonetheless, these fibers have some serious disadvantages as well because of their nature: they are highly inflammable and sensitive to moisture. These considerations limit their application in the engineering projects involving natural fibers with grave consequences because the massive hydrophilic polymers absorb a considerable amount of water and become susceptible to fire [3]. In lieu of cross-linked structures which tend to burn, composites reinforced with natural polymers and polyamides are cross-linked and reinforced to hold hydrogel at low temperatures. The restriction is specially prominent in the cases of construction of buildings and transport. These are problems that should be eliminated to enhance the stability and uniformity of the natural fiber composites over the long term [4]. One of the new measures that are being used to overcome these limitations is the practical application of biochar. Some of the agro-wastes that can be pyrolyzed to produce biochar include coconut shells. Biochar is resistant to high temperature and contains high content of carbon and is porous [5]. Polymer composites that are filled with biochar are capable of delaying the burning process by promoting the formation of char and provide a barrier to slow the transfer of heat. It can also reduce the uptake of moisture by untreated natural fibers by hydrophobically sealing the pores and avoiding water uptake. Thus, incorporating bio-based composites into kenaf and flax fibers and coconut shell biochar is one of the possible methods of increasing fire and moisture resistance [6]. In addition, some studies report that the addition of fire-active, carbon-rich fillers (i.e., biocarbon and biochar) noticeably influences the moisture and fire properties of natural fiber composites. For example, Dahal et al. [7] studied the water absorption and diffusivity of bi-epoxy composites with biocarbon fillers (hemp and switchgrass). The composites showed a water absorptivity reduction of 44.17% and a water diffusivity drop of 42.02%, which depended on the particle size, filler loading, and pyrolysis temperature. Kandola et al. [8] studied bio-based flax/furan composites compared with the conventional carbon/glass fiber systems and, on the fire-retardant side, found that flame-retardant composites—fire-retardant agents added to the resin—reduced fire hazard without substantially impairing mechanical strength. Direct application of fire retardants to the fibers, however, reduced both water tolerance and interfacial adhesion. Also, Campana et al. [9] showed that flax fibers, upon immersion in water, absorb approximately 62.5% moisture (or ~12% in humid atmospheres), and that hygrothermal aging can, through fiber swelling and fiber–matrix debonding, lower the dynamic elastic modulus (by 20%) and increase the damping ratio (by 50%), indicating debonding and inter-fiber matrix slipping.

Despite the positive attributes of biochar as a filler in natural fiber composites, much of the existing work focuses on single-fiber systems or biochar originating from non-lignocellulosic residues. There is a paucity of information on hybrid natural fiber composites reinforced with flax and kenaf, alongside coconut shell biochar. Coconut shells, rich in lignin and fixed carbon, are a readily available agricultural waste and, thus, an economical and excellent precursor for biochar with superior thermal stability and hydrophobicity [10]. The absorption and moisture characteristics of kenaf and biochar with flax fibers are still unknown and unexplained. This research application could provide opportunities to easily manufacture cost-effective, durable composites with increased resistance to fire hazards, saving lives and reducing damage to the interiors of vehicles, scaffolding, and green materials used in sustainable packaging [11].

This research focuses mostly on applying biochar obtained from coconut shells to kenaf and flax fiber polymer composites and studying the polymer composites crafted from biochar, designed specifically for their fire-retention properties. The incorporation of biochar in the composite materials helps to improve the thermal-based permeability barriers, capture active fillers, and enhance the composite fire-retardant properties while reducing the moisture uptake. The innovative value of the current research being tested is the combination of two types of plant materials like flax/kenaf fiber with different weight proportions of coconut shell biochar, and provides a sustainable alternative to traditional fire-retardants and moisture absorption additives. Through the resolution of two major issues with natural fiber composites, this work aims to contribute to the development of durable, sustainable materials for structural and semi-structural applications: resistance to fire and sensitivity to dampness.

2 Experimental Works

2.1 Material

The fabric had an approximate weight of 300 grams per square meter (300 gsm) and a density of 1.29 g/cm³. We purchased kenaf and flax from Deekshi Natural Fiber Industry, located in Salem, Tamil Nadu, India. The smooth sepals of these fibers make them excellent candidates for backing plies and top surface layers in composites. Biochar from coconut shells, with an average particle size of 50 µm, was sourced from Green Solution in Madurai, Tamil Nadu, India. For this work, Max Clear grade epoxy resin was utilized as the polymer matrix. To improve the adhesion of the flax and kenaf fibers to the resin during the fabrication of the composites, an alkali treatment was performed on the fibers for four hours using a NaOH solution at room temperature. This treatment removed some components of the fibers, such as hemicellulose, lignin, some waxes, and surface contaminants, which in turn made the fibers more cellulose rich. The fibers became more exposed and formed a stronger chemical bond with the polymer matrix. The NaOH treatment also improved the mechanical bond of the chemical bond, as it increased the fiber crystallinity. This increased the fiber mechanical strength and reduced the hydrophilicity of the fibers.

2.2 Composite Fabrication

After the initial hand lay-up step, the composite plates were made using a compression technique. Every laminate produced was of the same size, as reflected in the 300 × 300 × 3 mm³ mold that was used during molding. Two layers of flax fiber were alternated with two layers of kenaf fiber to construct a dual-balanced reinforcement structure. To investigate the influence of biochar addition, the composite plates—Plate A, Plate B, and Plate C—were prepared with 2, 4, and 6 wt.% biochar, respectively. All epoxy–biochar mixtures were first mixed by adding biochar to the epoxy, and the fabric layers were then impregnated with the epoxy. In the containers, mats of fibers and other plates were arranged in the order: pour and then distribute. Each layer was compressed to create a molding with zero air voids. Thermally capable systems were used to control temperature during the initial cure and the 24-hour post-cure, which helped to improve fiber cross-linking [12]. Trimming of the plates to the correct size was done to create specimens that met the standard requirements stated by ASTM. Figure 1 shows the photographic image of the fabricated composite materials.

Fig.1. Photographic image of fabricated composite materials

2.3 Mechanical characterization

2.3.1 Flammability Characteristics

The fire performance of the fabricated composites was determined using the Limiting Oxygen Index (LOI) test, UL-94 vertical burning test, and cone calorimeter analysis. The LOI test, conducted as per the ASTM D2863 guidelines (specimen size: 150 × 10 × 3 mm³), determined the oxygen concentration necessary to sustain combustion and thus indicates the flame-retardant efficiency. These tests are used to categorize samples based on self-extinguishing and burning characteristics, including after-flame time and active dripping, and UL-94 categorizes the corresponding composites. Also, cone calorimeter analysis according to ISO 5660 (specimen: 100 × 100 × 3 mm³) was employed to determine vital fire traits such as heat release rate (HRR), peak heat release rate (PHR), and time to ignition (TTI). Bringing together all these studies provided a broader understanding of how biochar made of coconut shell improves flame-retardant attributes, increases time to ignition, decreases heat release, and increases char formation. The fracture surfaces of the specimens were analyzed using a scanning electron microscope (SEM).

2.3.2 Moisture Absorption Behavior

Moisture absorption was investigated using a specimen size of 76.2 × 25.4 × 3 mm³, as per ASTM D570. Polymer fibers' water absorption tests showed water closure in the fibers. A total of 180 hours of immersion in water was divided into 12-hour intervals to check the samples. Each sample was weighed and measured to determine the mass loss due to water leaching and the moisture increase resulting from the absorption–drying process. Along with coconut shell biochar, the hydrophilic character and water penetration of flax and kenaf fibers were tested. The biochar was strategically placed to increase the sample's tortuosity, decrease void volume for absorption, and fill the voids in the sample.

3 Results and Discussion

3.1 Flammable Properties

Composites A, B, and C made of flax, kenaf, and biochar were examined using the Limiting Oxygen Index (LOI) and UL-94 vertical burning tests, combined with cone calorimetry, to determine the changes in biochar content and their effects on the fire resistance of the biochar composite. The higher the content of biochar, the greater the resistance observed. Composite A (2 weight percent biochar) had an LOI value of 20.5%, composite B (4 weight percent biochar) had 24.0%, and composite C (6 weight percent biochar) had 27.5%. The increase in biochar content in the composite mixture explains the improvement in flame retardancy due to the formation of a thermally stable char on the surface, which slows heat transfer, flammable volatile release, and oxygen accessibility to the composite. From the UL-94 vertical burning test, composite A was graded V-2 and exhibited a self-extinguishing flame with some drips. Composite B was graded V-1 with no burning drips, and the flame extinguished after 30 seconds. The density of the continuous layer of char can protect the fibers and epoxy matrix directly from flame by increasing the biochar content, as seen in the highest classification of V-0 by composite C, which exhibited the most performance and extinguished flames in 10 seconds without dripping [13]. Figure 2 demonstrates the UL-94 vertical burning of composite specimens.

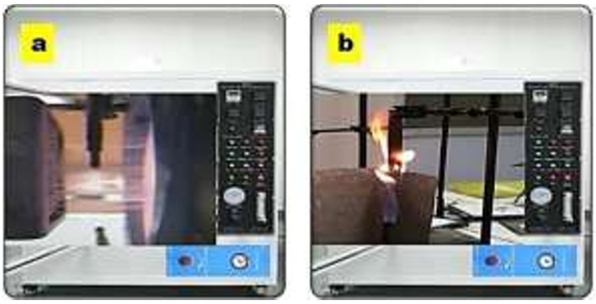

Fig. 2. UL-94 Vertical burning of hybrid composite specimen (a) Before burning; (b) After burning.

Biochar composites were found to enhance flame resistance, which was further supported by data from the cone calorimeter. The PHRR of composite A was 520 kW/m², but this value was greatly lowered to 360 kW/m² when composites B and C were being considered. Moreover, the ignition time (TTI) of A decreased to 35 and then increased to 45 and 60 seconds in B and C, respectively. The same trend was observed in total heat released (THR), where composites A and C had 85 MJ/m² and 48 MJ/m², respectively. This data suggests that the increased biochar content within the composites extends the time it takes to combust and ignite the composite while simultaneously lowering the total energy released during the combustion process [14]. Thermogravimetric patterns supported these findings, as the onset degradation temperature of composite A at 280°C shifted to 320°C for composite C, and in tandem with the increased biochar content, the char yield also increased from 12% to 25%. The beneficial increase in char formation shields the combustible material, which aids in the reduction of flammability. Figure 3 shows the various flammability characteristics of flax/kenaf/biochar-based hybrid composites. With the increasing content of biochar, the improvement of flammability performance can be explained by the carbon content of the coconut shell biochar itself, the biochar's high ability to support the formation of char, the reduction of combustible volatiles, and the biochar's insulating ability, as well as the promotion of insulation and carbonization of volatiles [15]. Moreover, the fibers with alkali treatment enhanced the epoxy matrix interface to further tighten the pathways for the outward flow of heat and oxygen. For composite C, which had 6 wt.% biochar, it exhibited the best fire-retardant properties by virtue of the highest LOI, the V-0 rating, the lowest PHRR, the longest TTI, and the highest char yield. Thus, it can be used in applications in which fire is a concern. The moderate performance of composites A and B confirms that lower biochar content fails to provide a shield against flame, as it does not form a complete protective char layer.

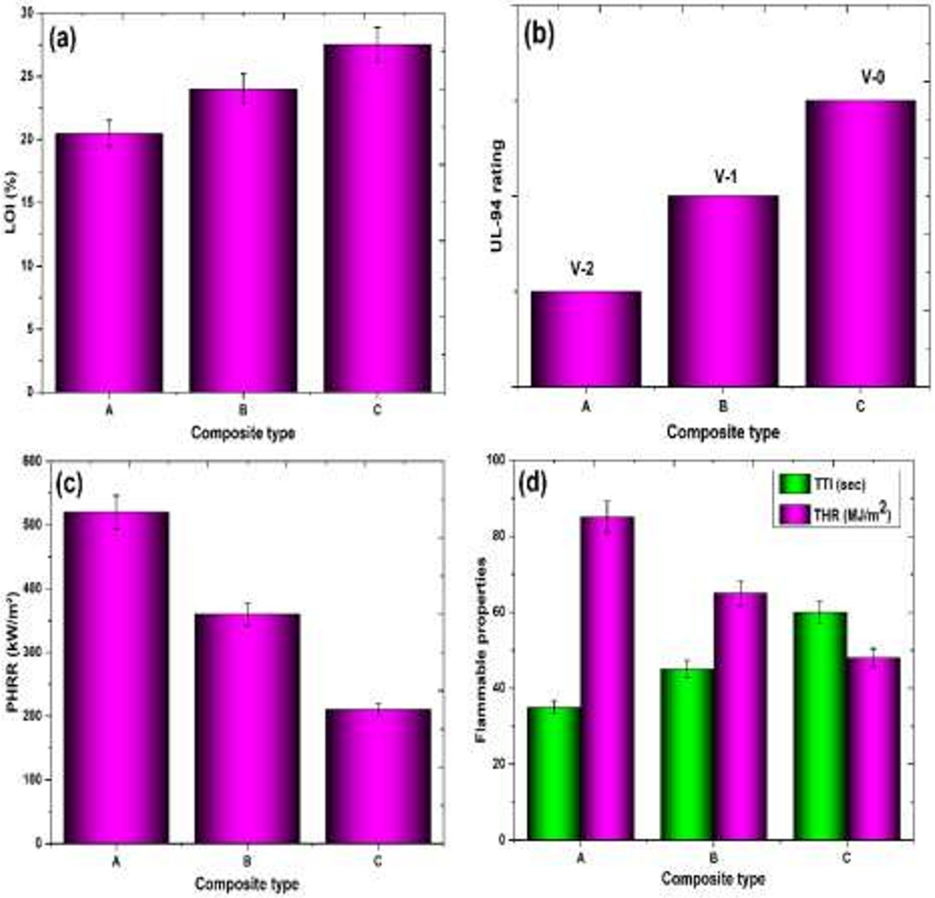

Fig. 3. Flammability characteristics of flax/kenaf/biochar-based hybrid composites

3.2 Microstructural analysis

Scanning Electron Microscopy (SEM) was used to analyze the microstructure of composites with interspersed layers of biochar and kenaf fibers and to assess the effect of biochar content on adhesion between the fibers and the matrix and the resulting char. The pre-burn fracture surfaces were analyzed in the SEM images, and differences between the low biochar and high biochar samples were clearly visible. Composite A (Figure 4 (a) & (c)), which contains 2 wt.% biochar, exhibited multiple micro-voids and gaps at the fiber–matrix interface, which indicates that the fibers were not completely wetted by the epoxy matrix and that the interfacial bonding was weak [16]. On the other hand, composite C (Figure 4 (b) & (d)) with 6 wt.% biochar showed a more uniform fiber-matrix interface and better dispersion of biochar particles, indicating that the increased biochar content, along with alkali treatment of the fibers, improved bonding at the interface and lowered void content. Additional post-burn chars were also analyzed to show the contribution of biochar to flame retardancy. The intermittent and broken char coating of Composite A justifies the higher rate of heat release and reduced UL-94 rating because the fractures increase the level of permeability of the composite. Composite C, on the other hand, exhibited adequate layers of continuous char on the fiber surfaces, which are essentially thick enough to block the volatile emission and heat transfer. These SEM studies, which corroborated results of LOI, UL-94, and cone calorimeter, indicated that the structural integrity and flame resistance of flax/kenaf hybrid composites increase with the concentration of biochar [17].

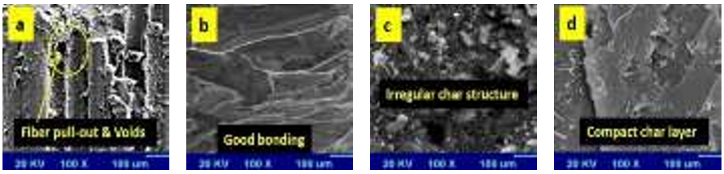

Fig. 4. Microstructural images of (a &c) A-type; (b &d) C-type composites of before and after burning.

3.3 Moisture absorption behavior

The amount of moisture that was taken in by flax/kenaf/biochar composites was found to differ significantly between composites A, B, and C and depended on the quantity of biochar present. Composite A with 2 weight percent biochar and with such kinds of fibers as hydrophilic flax and kenaf has revealed the highest water absorption capacity. The cellulose fibers contain hydroxyl groups, which enable them to bond with water, and this enhances the quantity of water that is taken in. Composite B showed some moisture absorption, although to a lesser extent than Composite A, indicating that an increase in biochar concentration begins to inhibit water absorption. Composite C exhibited the least moisture absorption among the three, which confirms the theory that higher biochar content reduces water uptake [18]. Figure 5 shows the moisture absorption behavior of flax/kenaf/biochar-based hybrid composites. There are multiple reasons that explain the phenomenon of reduced water absorption in composites with more biochar. Biochar particles fill the micro-voids in the composite, eliminating free space and thereby allowing less water to diffuse. Increased biochar also lowers the total free space for water, increases the tortuous path that water must travel, and thereby delays water penetration [19]. Bonding between the biochar and the matrix also reduces moisture uptake and supports the argument that higher biochar content decreases the free volume available for water absorption [20]. These combined effects are shown to improve the dimensional stability of the composites, especially C, which has the highest compactness as well as the lowest water absorption. Absorption of water vapor is also one of the differences observed in hypothetical composites, revealing the effectiveness of biochar in natural fiber composites. Increased biochar content also improves the mechanical, thermal, and moisture-affected durability of the composites by reducing moisture ingress. This makes composite C the most ideal candidate for moisture-laden environments, as it shows the least water-induced transverse swelling, fiber-matrix debonding, and eventual degradation. This also demonstrates the multifunctional role of coconut shell biochar, which improves the structural and moisture stability of hybrid natural fiber composites [21].

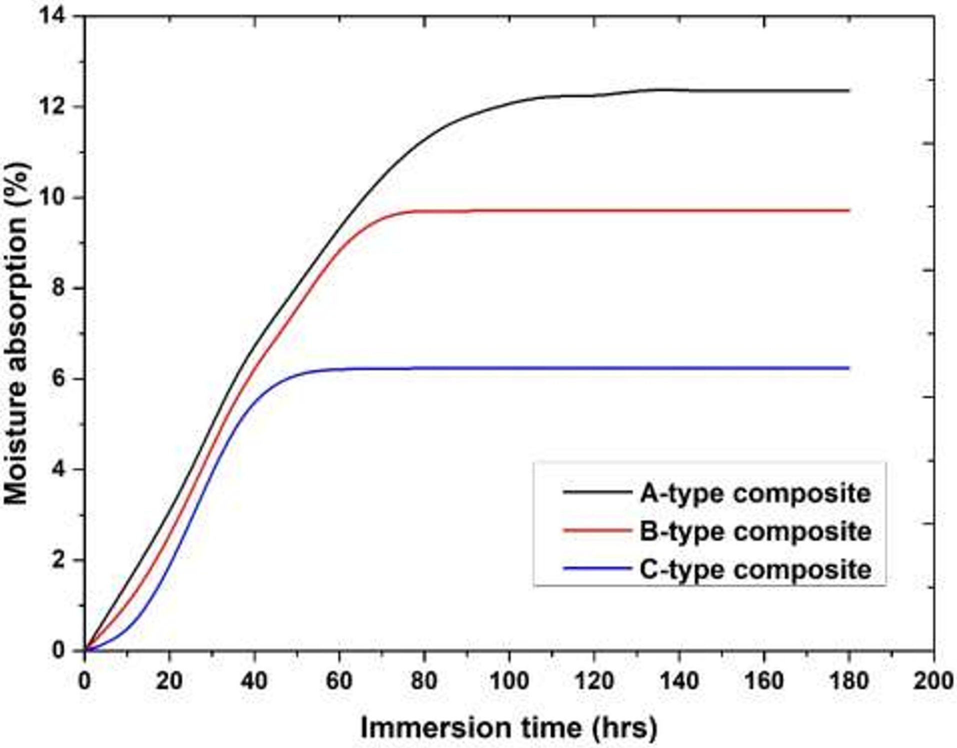

Fig. 5. Moisture absorption behavior of flax/kenaf/biochar-based hybrid composites

4 Conclusion

The investigation revealed that the use of biochars produced from coconut shells improved the flammability and moisture resistance of flax/kenaf hybrid epoxy composites. Increasing biochar content from 2 to 6 wt.% improved fire retardancy, as indicated by corresponding increases in the LOI and UL-94 ratings, decreases in peak heat release rates and ignition times, and an increased char yield. At the same time, moisture absorption was reduced with increased biochar content, with 6 wt.% biochar significantly minimizing water ingress as a result of void filling, tortuous diffusion pathway formation, and enhanced fiber–matrix interfacial bonding. Composite C (6 weight percent biochar) was the only one out of the three composites that showed the optimum ratio of moisture and fire resistance at all times. It shows the versatile usefulness of coconut shell biochar by improving the mechanical, fire, and moisture protection of natural fiber composite and increasing its use in the construction and automotive components subjected to elevated temperatures and humidity.

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