



Burning Properties of Lignocellulosic Fiber (Natural Fiber) and Polymer Composites Reinforced by Flame Retardant

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Abstract Natural fiber reinforced polymer composites are fast replacing the synthetic ones in some areas of the composite world. Light weights, non-susceptibility to corrosion, and ecofriendly benefits of natural fiber composites are some of its attractions. Natural fibers consist of three constituents namely; cellulose, lignin and hemicelluloses with the cellulose in major and are extremely responsive to flammability of natural fibers. For this reason, their applications have been limited to regions where fire is not of great threat. The incorporation of flame retardants (FR) during the fabrication of natural fiber composites can reduce their flammability potentials and enhance thermal stability. In this study, an overview of the types of flame retardants, standard fire performance tests, cone calorimeter flammability parameters and flammability of selected natural fibers was carefully done. The global trend in halogen free flame retardant selection was emphasized and it is clearly noted that fiber surface treatment and synergetic effect of metal hydroxides flame retardant, and/or phosphorus and nitrogen based flame retardant with Biobased flame retardant may be the most effective.

Keywords Lignocellulosic fiber, Polymer composites, Fire retardant, Burning properties, Fire tests

Introduction

Natural fibers being environmentally friendly and renewable in nature interests a lot of scientists, researchers and engineers for their use in polymer composite reinforcements. The type of fiber, fiber orientation, interfacial bonding and fiber aspect ratio are some common factors affecting the properties of natural fiber reinforced polymer composites (NFRPCs) which results in poor flame resistance, high water absorption and lower mechanical properties than synthetic fibers and thereby restricts their applications in various fields. The applications of NFRPCs are very numerous such as in sports, construction, automobile, and textile materials [1]. Surface treatment of natural fibers helps to improve the adhesion between the fiber surface and the polymer matrix which eventually results in improved physical and mechanical properties of the NFRPCs [2, 3]. It is being believed that high cellulose in natural fibers provides chances of higher flammability while higher lignin content provides a greater chance of char formation during the burning of composites which creates a barrier for the mass transfer of heat [4]. The very purposes of flame retardants addition includes (1) improve their flammability resistance (2) help delay and obstruct fire propagation (3) inhibit flaming processes. A good number of flame retardant additives have been studied to improve flammability (fire reaction properties) of materials and there are many compounds from which flame retardant additives could be formulated [5]. This study aims at providing a brief overview of flammability and flame retardancy of natural fiber composites in a fashion that clearly defines the trend. It clearly highlighted on the types



of flame retardants, standard fire performance tests, cone calorimeter flammability parameters and flammability of selected natural fibers.

Natural Fiber

Table 1: Physicochemical Properties of selected Natural Fibers, Sources

Type of fiber	Origin	Density (g/cm ³)	Cellulose (%)	Lignin (%)	Hemicellulose (%)	Pectin (%)
Oil palm	Fruit	1.3	65	29	22.1	-
Jute	Stem	1.3	58 - 63	12 - 14	21 - 24	-
Hemp	stem	1.48	57 - 77	3.7 - 13	14 - 22.4	0.9
Wood	stem	0.65	40 - 45	20 - 30	22	2 - 4
Flax	stem	1.5	64.1	2.0	16.7	-
Bagasse	stem	1.25	81	25.3	9.45	-
Sisal	leaf	1.37	65	9.9	12	10
Coir	fruit	1.2	32 - 34	40 - 45	0.15 - 0.25	-
kenaf	stem	1.5	31 - 57	15 - 17	21.5 - 23	-
Ramie	bast	1.51	68.6 - 91	0.6 - 0.7	5 - 16.7	1.9
Banana	stem	1.35	50 - 56	21 - 31	25 - 30	-
Henquen	leaf	1.3	77.6	131	3 - 8	-

The word natural implies a particular substance which exists naturally and not manmade. The word fiber is defined as a hair-like or thread like structure which has high aspect ratio (length to diameter ratio). These natural fibers are hair like materials that are continuous filaments which are similar to pieces of thread. They can be spun into filaments, thread or rope and can be used as a reinforcement component of polymer composites materials. It can also be pulped to make products such as paper or felt, [6]. Dunne et al. (2016) [7] reported that there are two major classifications of plant producing natural fibers: primary and secondary plants. The primary plants are those grown basically for their fiber content while the secondary plants are those where the fiber come as a by-product from some other primary uses. Sisal, cotton, jute, hemp, kenaf are examples of primary plants while banana, pineapple, oil palm, cereal stalks and coir are examples of secondary plants.

Flame Retardant Compounds

Natural fibers and polymers are naturally considered organics and have a high susceptibility of flaming if exposed to sources of heat. As such, flame retardants are incorporated during the manufacturing stages in order to lower their flammability properties and thus extend their applications within the industry and their safety while in use. Systems for flame-retardant can either be reactive or active. The active flame retardants are not chemically bound to the chemical materials or polymers utilized in the product, but are intermixed with other product materials utilized during the processing of the product. These flame retardants retain their chemical properties and are spread evenly throughout the product. These flame retardants are also able to exit their matrix via release to the air and pool into dust [8]. Conversely, the reactive flame retardants are chemically fixed towards the polymers throughout polymerization, grafting or coupling stages, and become an important aspect of the product's structure. The whole purpose of integrating flame retardants is to inhibit or delay combustion [9].

There are health and safety restrictions and regulations on the usage of FRs that are based on halogen. Mineral fillers including aluminum hydroxide (ATH), magnesium carbonate, magnesium hydroxide or mixed calcium/magnesium carbonates and hydroxides, and naturally existing mixtures of hydro-magnetite and huntite are greatly demanded as sustainable and environmentally friendly fire retardants, as reported by Hull et al. (2011)[10]. Depending on their natural properties, the flame retardants are able to perform in the gas or condensed phase via chemical or physical process as reported by Zhang et al 2014. Flame retardants (FR) can be further grouped based on their mechanism of action: (I) Halogen based FR (II) Halogen free FR (III) Intumescent FR (IV) Biobased FR.



1. Halogen Based Flame Retardants (HBFRs)

Theoretically, there are four groups of chemical compounds that may be utilized as HBFRs; chemical compounds that possess iodine, chlorine, fluorine or bromine. The effectiveness of these halogens in an increasing order is as such: Fluorine < Chlorine < Bromine < Iodine. Nonetheless, bromine and chlorine based flame retardants are more frequently utilized. The naturally low thermal stability and difficulty in processing iodine and fluorine compounds with commercial polymers make chlorinated and brominated halogen flame retardants a much suitable option and most diversified group of retardants for inhibition of flame [11]. Chlorine and bromine integrated into a polymer chain via copolymerization resulted in a reactive flame retardant that is able to regulate the temperature of fire's flame [12]. It was further reported that the release of halogen in the form of halogen or radical halide at the identical range of temperature or below the polymer's decomposition temperature affects their effectiveness. The main issue with compounds that are based on halogen is the emission of smoke, possession of acidic, toxic and corrosive gases that are detrimental to health and the environment.

2. Halogen Free Flame Retardants (HFFRs)

It is widely known that halogen free flame retardants are environmentally friendly and are closely integrated into the polymer during the processing stage, despite the fact that it does chemically react with the polymer. Blending is carried out by using a high mechanical stirring tool including a bartender mixer. In order to raise efficiency, the substances are used at levels that are lower than 20% of the polymer resin. HFFRs are important materials, usually being preferred in the condense stage of the burning mechanism [13]. The mechanism of action of HFFRs that are incorporated in composites is that they serve as a heat sink by emitting water vapour. They go through an endothermal decomposition and release non-combustible volatiles that give out a blanketing role in the flame. The blanket coating that is resulted removes the formed heat and essentially reduces the temperature of the substrate to a level that is lower than that necessary to maintain combustion. In addition, water comes into contact with the flame, where it reduces burning via dilution of the flammable gases emitted from the polymer matrix and hindering the oxygen from reaching the gases on the surface of the composite. The relatively simple method of handling, excellent anti-corrosion characteristics, non-toxicity and inexpensive cost of HFFRs make them a suitable choice instead of the HBFRs class of flame retardants. In addition, their disadvantages such as low efficiency for loading up to 50% from the material's mass, a reduced strength in sustaining use and poor thermal stability has drawn concern for their use [14]. Nonetheless, it was shown by Hirschler that HFFRs deliver formulations that are able to meet standard test for various applications.

3. Intumescent Flame Retardants (IFRs)

According to Wikipedia, the word intumescent is defined as a substance that swells following exposure to heat, thus experiencing a reduction in density and rise in volume. Intumescent flame retardants act by forming voluminous, insulating protective layer via carbonization and subsequent foaming [15]. They have great potential in applications to shield combustible materials such as wood or plastic, as well as steel, which loses their strength following exposure to high temperatures, against the effects of fire and heat. The intumescent flame retardants are made up of (i) spumific compounds, which are known as gas evolving compounds or blowing agents, (ii) acid donors (e.g. ammonium polyphosphate), and (iii) carbon donor (e.g. polyalcohol such as starch, and pentaerythritol). Enormous amount of gas (e.g. urea, melamine) are produced by them (Girods et al. 2008). The combination of pentaerythritol (PER)/ ammonium polyphosphate (APP)/ without or with melamine produces a synergistic effect that functions as the source of acid and blowing agent for combustion. Nonetheless, the IFRs systems with polymers do possess several weaknesses such as thermal stability, resistance to moisture, unfavorable compatibility and reduced efficiency of flame retardant at low IFR concentrations, which then leads to significant reduction in mechanical characteristics. There is an urgent need to assess these weaknesses and their actual toxicity level.

4. Biobased Flame Retardants (BFRs)

In general, biobased compounds are compounds that may be collected from biological matter on earth. Some of the commonly found elements include Ti, Mn, Na, P, Cl, S, Fe, Mg, K, Si, Ca, H, N, and C. These aforementioned elements possess flame retardant properties [16]. A significant point when using biomass for the generation of flame retardants is chemical composition. The compounds can be segregated into four major families and their derivatives



can be differentiated for this property: phenolic, lipid, protein and carbohydrate compounds. The compounds that are biobased may be utilized directly or further developed to form flame retardant abilities. Lipids are composed of mostly tannins and lignin, while carbohydrates are composed of chitosan, cellulose and starch. Charring effect brings about the enhancement of the biobased flame retardant's fire behaviour. This effect is developed throughout combustion on the surface of the sample. Two positive outcomes are formed from the char: (i) on one side in which polymer structure that is made up of carbon atoms is integrated inside the residue, thus reducing the quantity of volatile fuels and heat emitted via combustion reaction; (ii) on the other side, it serves as a protective shield that dampens the transfer of heat to the underlying polymer and makes modification to the fuel diffusion's kinetics to the flame. The flame retardant systems that are biobased may be utilized in one of the following modes (i) in combination with conventional nitrogen or phosphorus flame retardant, (ii) as intrinsic flame retardant system, (iii) following chemical modification and grafting into the chain of polymer. Intrinsically used biobased compounds include tannin, chitosan, lignin, starch, DNA and proteins [17]. There has also been studies on the combination of compounds that are bio-based with phosphorus or/and nitrogen. A novel flame retardant system composed of stable complex of a phosphorus compound and cyclodextrin nano sponges (NS) was reported by Alongi et al. (2010)[18]. B-cyclodextrin, which is a derivative of starch, serves a role in the synthesis of the cyclodextrin nano sponges (NS). An intumescent layer was generated during the combustion and allowed a reduction in the total heat released (THR) and peak heat release rate (pHRRR), as assessed by cone calorimeter. An intumescent composition with 10wt% - 30wt% starch was utilized by Réti et al. in combination with ammonium polyphosphate (APP) in a polylactic acid (PLA) biopolymer. A 40% limiting oxygen index (LOI) and V0 rating at the UL94 test was obtained. Lignin was integrated with mono-ammonium phosphate and APP, melamine phosphate and aluminum hydroxide in a polypropylene (PP) matrix by De Chicro et al. (2003) [19]. The synergistic effects brought about a rise in temperature of thermal degradation and char yield. It also caused a reduction in the rate of weight loss during combustion and rate of release of heat.

Fire Performance Tests and Flammability Parameters

Standard Fire Performance Tests

Various laboratory test approaches are available for the characterization of the composite materials' flammability properties and are established within these references.

1. Laboratory standard UL-94 test

The most commonly utilized small flame burner test is the under laboratory (UL) 94 test, which allows a study of flammability for various plastic materials that are to be utilized in distinct applications. Several testing methodologies exist within the UL 94 standard; the most popular methodology utilized is the 20 mm vertical burning tests (V2, V1 or V0). There are two distinct versions of the test depending on the holding position of the sample: vertical (UL94 V) and (UL94 HB) tests. Each test is carried out according to a particular standard for procedural tests and set up. A common test method case is the vertical burn approach. In this approach, a test specimen bar of 125 mm by 13 mm with distinct thickness is burned while lying suspended 10 mm above a calibrated methane Bunsen burner. The ignited flame is carried out on a total of five investigation specimens twice for duration of ten seconds. Following each application of flame for each test bar, the burn time is recorded [19].

Table 2: Standard ratings for UL94 vertical burning test

Rating	Description
UL-94 V-0	Flame must be out in 10 seconds or less. No glow beyond 30 seconds and no burning material can fall.
UL-94 V-1	Flame must be out in 30 seconds or less. No glow beyond 60 seconds and no burning material can fall.
UL-94 V-2	Flame must be out in 30 seconds or less, no glow beyond 60 seconds and burning material can fall.



2. Limiting Oxygen Index (LOI)

The minimum oxygen level that is required in an atmosphere to support the combustion of flame is termed Limiting Oxygen Index (LOI). There is a disparity among polymers that lack flame retardants in their propensity to support combustion under normal atmospheric conditions. In addition, the relative resistance of the aforementioned polymers is influenced by each polymer's chemical composition. An approach for the ranking of polymer's combustibility is the LOI. It is significant for plastics utilized in the aviation industry. Considering the fact that air is made up of 20.95% (roughly 21%) oxygen by volume, any material possessing a limiting oxygen index that is lower than 21% will easily ignite and stay ignited in the air. On the other hand, the burning tendency and behavior in propagating flame for a polymer that has a limiting oxygen index that is more than 21% will be decreased or even zero following elimination of the source of ignition. Should LOI be more than 100, it is not possible for any combustion in any oxygen-nitrogen atmosphere to be self-sustainable. Indeed, such values lack physical meaning [20].

Measurement of LOI is carried out by putting the samples in a flow of nitrogen/oxygen gas and raising the oxygen's concentration until the point when combustion is supported by the sample. The value utilized as the index is the next lowest concentration of oxygen. The greater the value of LOI, the greater the material's non-flammability [20].

In the work entitled "A Dynamical Systems Model of the Limiting Oxygen Index Test", Nelson et al. reported that oxygen index approach describes a material's tendency to support a flame. It has been utilized over years as the foundation for systematic studies into the distinct fire-retardants and fire-retardancy mechanisms' relative effectiveness. Equation 1 describes the limiting oxygen index (LOI), or oxygen index (OI), or critical oxygen index (COI):

$$[\varphi(O_2) + \varphi(N_2)]$$

3. Cone Calorimetric Test

The most advanced method at present for the assessment of materials reaction to fire is the cone calorimeter test (CCT). The investigation allows the evaluation of the combustibility, ignitability, and production of toxic gases and smoke. It provides a quantitative analysis to flammability of materials by studying the parameters such as time to ignition (Tig), heat release rate (HRR), mass loss rate (MLR) and total heat released (THR). Both ASTM standards (ASTM E1354) and ISO standard (ISO DIS 5660) describes the testing steps and measurement observations. Under a forced flaming condition, the test is run by exerting a constant external heat flux to 100 x 100 mm specimens. Should the thickness of the specimen be less than 50 mm, extra insulation layers is usually needed to fill out the depth and to fix the specimen tightly in the holder. This permits the test specimen's full surface to be exposed to a heat irradiance at constant level. Originating from a conical heater, the heat flux's range varies between 0 to 100 kW/m². At the test's early steps, the temperature of the surface rises rapidly and then evens off to a steady state [21].

Fire Sensitivity Parameters

1. Cone Calorimeter Flammability Parameters

The significant flammability aspects gained from fire performance assessments, especially from a cone calorimeter with their standard unit, is summarized in Table 3.

Heat Release Rate (HRR)

An important variable in defining phenomenon such as fire hazard is solely the HRR, which is always presumed to be driving force in any performance of fire. It is defined as the thermal energy that is generated per unit surface when flammable decomposition products are ignited and burned in the material's vicinity in heat flux or fire. The level of HRR denotes the production level of hazardous impact of fire and its products [22]. This implies that the rate of heat release is in line with the production of smoke, toxic gases and other forms of fire hazards.



Table 3: Cone calorimeter flammability property parameters

Parameter	Units
Heat Release Rate (HRR)	KW/m ²
Peak Heat Release Rate (pHRR)	KW/m ²
Average Heat Release Rate (aHRR)	KW/m ²
Total Heat Released (THR)	KW/m ²
MARHE	kW/m ²
Effective Heat of Combustion (EHC)	MJ/Kg
Exhaust Flow Rate	m ² /Kg
Specific Extinction Area (SEA)	m ₂ /Kg
Mass Loss Rate (MLR)	g/s
Final Sample Mass	G
Time to Sustain Ignition	S
CO/CO ₂ Production	g/s
Total oxygen consumed	G
Mass lost	g/m ₂
Carbon monoxide yield	kg/kg
Total smoke release	m ² /m ²
Total smoke production	m ²

Other variables that are associated with HRR include Peak Heat Release Rate (pHRR), which takes place instantly and usually following ignition. It is also an excellent indicator of a material's maximum flammability [23]. Average Heat Release Rate (aHRR) is another significant HRR correlation variable indicator, which is the total heat emitted averaged over the total period. It is also considered a highly reliable measurement of heat of contribution towards sustained fire.

Flammability Studies of Natural Fibre Composites

One of the materials with great potential for different domestic and industrial products is natural fiber reinforced polymer composite. However, its constituents which include polymer and cellulose are highly flammable. At present, this issue is being heavily addressed by researchers all around the world. The coir fiber reinforced polypropylene (PP) composite panel for automotive interior uses is being studied by Ayrimis et al. (2011) [24]. The same study evaluated the composite panels' flammability, mechanical and physical characteristics. Four levels of coir fiber contents (40, 50, 60 and 70 wt%) were mixed with PP powder and coupling agent, 3wt% maleic anhydride grafted PP(MAPP) powder. The rising levels of coir fiber significantly affected the composites' internal bond strength and water resistance. Results show that the most optimal composite panel formulation for automobile interior purposes is a mixture of 3wt% MAPP, 37wt% PP powder and 60wt% coir fiber.

Before incorporation into the resin, the coconut fibers were first treated with NaOH, followed by silage. The untreated fibers composite served as a control. In order to reduce the composite's flammability, the dragon which is a phosphate type flame retardant, was incorporated into the composite. The quantity of Draco ranged between 0 - 10wt% of the whole resin's mass. The addition of Draco raised the limiting oxygen index (LOI) and burning characteristics of the untreated and treated composites.

The impact of flame retardant and coupling agent on the performance of oil palm empty fruit bunch fiber (OPEFBF) reinforced polypropylene (PP) composite was investigated by Beg et al. (2013) [25]. In order to generate composite via melt casting approach, alkali untreated and treated OPEFBF were integrated in PP without and with maleic anhydride grafted PP (MAPP) and magnesium hydroxide as a flame retardant. Characterization of the composites was done via burning and mechanical test along with Fourier transform infrared (FTIR) and scanning electron microscopy (SEM). The significant enhancement in the property of flame retardant was at 60vol% of treated



OPEFBF -PP composite with MAPP. The generation of encapsulated texture discussed the enhanced mechanical characteristic. The flammability of natural woven coconut tree leaf sheath (CLS) reinforced phenol formaldehyde (PF) composites were investigated [26]. At a volume fraction of 40wt% of PF resin and 60wt% sheath, the CLS composites were prepared in untreated and treated forms. 5% NaOH was utilized to chemically treat CLS. A hydraulic hit press was utilized to prepare composite panels at 14000C. The Limiting Oxygen Index (LOI) and Underwriters Laboratory test (UL94) were utilized to assess the composites' flammability. Based on the results of the UL94 investigation, there was a rise in flame resistance and reduction in mass loss and rate of flame propagation for treated fibers. During the LOI investigation, the alkali untreated composites required less oxygen than the treated composites in order to burn. As such, the flammability of composites utilized for decorative and construction will be enhanced by the treatment of CLS fiber.

Table 4: Applications of Selected Flame Retarded Natural Fibre Composites

Fibre	Applications in building, construction and other fields.
husk fibre	Building materials such as building panels, bricks, window frame, panels, decking, railing systems, and fencing.
fibre Rice	Building materials such as windows, door frames, structural insulated panel building systems, siding, fencing, roofing, decking, and other building materials.
fibre Stalk	Building panel, furniture panels, bricks, and constructing drains and pipelines.
Wood fibre	Window frame, panels, door shutters, decking, railing systems, and fencing
Jute fibre	Building panels, roofing sheets, door frames, door shutters, transport, packaging, geo-textiles, and chip boards.
Kenaf fibre	Packing material, mobile cases, bags, insulations, clothing-grade cloth, soilless potting mixes, animal bedding, and material that absorb oil and liquids.
Oil palm fiber	Building materials such as windows, door frames, structural insulated panel building systems, siding, fencing, roofing, decking, and other building materials.
Cotton fibre	Used in products as industrial sewing thread, packing materials, fishing nets, and filter cloths. It is also made into fabrics for household furnishings (upholstery, canvas) and clothing, paper manufacture.

Conclusions

Some natural fiber reinforced polymer composites' flammability characteristics are explored by this paper. The heavy usage of natural fiber reinforced composites in industries is attributed to their intrinsic characteristics such as light weight, eco-friendliness and biodegradability. Despite these favourable characteristics, the natural fibers and polymers' high degree of vulnerability to propagation of flame has narrowed their applications to conditions where there is impending menace of fire. Existing investigations have shown that the integration of flame retardant additives in polymeric materials is able to reduce the flammability aspect and reduce the fire propagation in the composite material.

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