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Review Paper

A Review on the Flammability and Flame Retardant Properties of Natural Fibers and Polymer Matrix Based Composites

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ABSTRACT: Natural fibers reinforced polymer composites are being used in several low strength applications. More research is going on to improve their mechanical and interface properties for structural applications. However, these composites have serious issues regarding flammability, which are not being focused broadly. A limited amount of literature has been published on the flame retardant techniques and flammability factor of natural fibers based polymer matrix composites. Therefore, it is needed to address the flammability properties of natural fibers based polymer composites to expand their application area. This paper summarizes some of the recent literature published on the subject of flammability and flame retardant methods applied to natural fibers reinforced polymer matrix composites. Different factors affecting the flammability, flame retardant solutions, mechanisms and characterization techniques have been discussed in detail.

Key Words: Flammability, Natural fibers, Polymer composites, Flame retardants, Nano-fillers

1. INTRODUCTION

Light weight, low cost and ecofriendly characteristics of natural fibers based polymer matrix composites have become a need of the hour due to the rising prices of petroleum products and their environmental risks [1]. Due to the uncertainty of aluminum supply during the Second World War, Mr. Gordon (an undergraduate) at Trinity College presented an idea to utilize flax fiber. The Aero Research Limited Company adopted this idea and the first natural fiber reinforced composite (flax/phenol formaldehyde) was developed and used in the spar of a bomber aircraft [2]. Natural fibers reinforced composites (NFRC) are also being used extensively in the interior parts of automobiles and construction industry [3,4]. Aside numerous potentials, The NFRC has inferior flame resistant characteristic [5-7]. Several in-flight flame incidents have been recorded in the transport airplanes history [8]. According to a report of society of automotive engineers, in USA, in-flight smoke incidents happen frequently [9]. This smoke may sometime erupt flame, which causes major accidents. Flames in automobiles can be extinguished if detected in the beginning. According to Boeing Company, most of the flame incidents are caused by the fume or smoke created through electrical sources [10].

Due to their undesirable flammability characteristics, more research is needed on the flame retardant approach and mechanism of NFRC [11-13]. Researchers are trying to invent new mechanisms which can make NFRC able to resist to the flame propagation [14]. Still not much literature is available on the flammability and flame retardancy of NFRC. Fig. 1 shows an estimated comparison of the number of articles published in the last five years in Elsevier publishing journals on the topics of natural fiber composites and flame retardant natural fiber composites. The comparison clearly shows how little research is being carried out on the flammability of natural fiber composites. However the encouraging factor is that the trend is going on increasing with time. It is expected to increase more in future.

The different phenomenon has been introduced to achieve flame retardancy. One way is to modify the chemistry of the polymer matrices. Sauca *et al.* obtained phosphorous-containing polymers by chemically modifying poly(vinyl alcohol) [15]. Flame retardant coatings of polymers and composites are also a well-known method against flammability. Zeytuncu *et al.* deposited UV-curable boron containing hybrid coatings on

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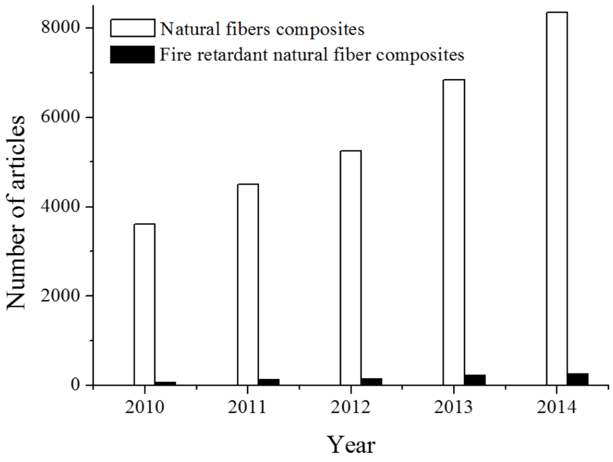


Fig. 1. Number of articles published on NFRC and flame retardant and flammability phenomenon of NFRC in Elsevier journals. Source: www.sciencedirect.com (19 Nov 2014). Keywords: natural fiber composites, flame retardant natural fiber composites, flammability of natural fiber composites

polycarbonate substrates to obtain flame retardancy [16]. Tsafack *et al.*, Caiwong *et al.*, and Paosawatyanyong *et al.* used plasma coating technology to achieve cotton fibers based flame retardant composites [17-19]. The most famous way to induce flame retardancy in polymeric composites is incorporating micro/nano flame retardants (FRs) in the materials. It has been broadly reviewed by different authors in literatures [20-23]. Umemura *et al.* used ammonium polyphosphate (APP), melamine polyphosphate (MPP) and aluminum hydroxide to modify flammability of wood-plastic composites [24]. Shah *et al.* used oyster shell powder as a calcium carbonate resource to achieve flame retardancy in polypropylene [25]. Sabbagh *et al.* used two different additives of non-organic mineral and organic phosphate FRs in flax fiber reinforced engineering plastic composites [26].

Although NFRC are cheaper and can replace glass fiber in some of the applications if the flammability problem is addressed, but this may cause an increase in the total cost of the composite. The increase in flame retardant and thermal properties may also affect some of the other properties. Most of the FR fillers decrease the mechanical strength of composites and polymers [27-29]. However, this effect can be controlled (or avoided) with different physical and chemical techniques, which include proper distribution of the FR filler, their surface treatment and the use of compatibilizer [30,31]. This will be one of the future research focus in the field of flame retardant natural fiber composites.

2. NATURAL FIBERS

To understand the properties of the natural fibers, it is important to investigate their structure and composition. It depends mainly upon the source from where the fibers are obtained. These sources include plants, animals and minerals. Animal fibers are obtained from hair, wool or silk and they are mostly composed of protein [32,33]. Plant fibers mainly consist of lignin matrix, hemicellulose and cellulosic micro-fibrils as shown in the structure in Fig. 2. Secondary wall S2 is normally responsible for the mechanical properties of the fiber. Natural fiber itself is a composite structure where the microfibrils act as fibers while the lignin and hemicellulose act as a matrix [33]. Therefore the cellulosic micro-fibrils are the load bearing components of natural fibers. Beside this, the hemicellulose is responsible for thermal and biological degradation properties while lignin and hemicellulose together are responsible for flame degradation properties [34-36]. However the final properties of NFRC do not only depend upon the fibers, but also depend greatly upon matrix, compatibilizer and pretreatment of the fibers. The flame properties of natural fibers have been described by different researchers in the literature [37-40].

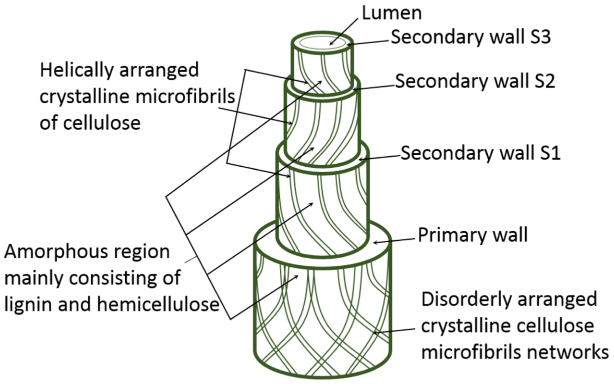


Fig. 2. Structure of natural fiber, source [33]

3. POLYMER MATRICES

Polymeric matrices are generally divided into thermoplastic and thermoset polymers. Besides the two major types, biopolymers are known to be a third type of polymers which are more common in research now-a-days. The properties of composites greatly depend upon the polymeric matrices and the fabrication processes. Generally matrix plays a role to support the reinforcements against compression and shear. But polymeric matrices are weak against flame propagation and thermal loads [41]. Several articles have been published on the flammability of polymers or polymer composites [42-45]. Flammability of polymers is explained on the basis of some parameters, e.g. limiting oxygen indices (LOI) and the heat release rates (HRR). Flammability properties of some of the commonly used polymers are listed in Table 1. A variety of fibers or additives are used in combination with polymer matrices to develop composites. The fibers or additives may be of different kinds, including natural, synthetic, organic or inor-

Table 1. Flammability properties of some polymers [46-48]

Polymer	HRR ($W \cdot cm^{-2}$)	LOI (%)
Polypropylene	150.9	18
Polystyrene	110.1	18
Polycarbonate	42.9	27
Poly(vinyl chloride)	17.5	42
Polyethylene	140.8	18
Polylactic acid	27.2	21

ganic. Different methods to fabricate polymers based composites include compression molding, extrusion, injection molding, infusion molding, resin transfer molding etc.

4. FACTORS EFFECTING FLAMMABILITY OF NATURAL FIBER REINFORCED POLYMER COMPOSITES

According to the famous flame triangle, fuel, heat and oxygen are the three important elements in a combustion process. Once the flame is ignited then stopping any of the above three elements may stop (or reduce) the propagation of flame. Natural fibers rapidly decompose under heat sources while polymer matrices melt. The hemicellulose decomposition starts first in the beginning of flame and with the increase in temperature lignin also gets decomposed. Mass is lost continuously during the decomposition process because of the evaporating gases.

Different techniques are used to study the flammability and thermal behavior of natural fiber reinforced polymer composites (NFRP). These methods include flame tests (horizontal & vertical), limiting oxygen indices (LOI) test, cone calorimetry, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA) [49,50].

The amount of fiber content, matrix type, surface treatment and filler concentration are the factors which affect the final properties of the NFRP. The flammability and thermal behavior of the composites are also affected by these factors. Different authors have studied the effects these factors on the flammability of composites.

4.1 Effect of fiber reinforcements

Helwig and Paukszta evaluated flammability of flax fibers reinforced polypropylene composites [51]. They found that flax fiber concentration of 30% and above resulted in a decrease in HRR and mass loss rate (MLR) of flax/PP composites. However, flax reinforcement reduced the burning time of the composites.

Chai *et al.* studied the effects of natural fiber reinforcement on the flammability of composites and compared its performance with glass fiber composites [52]. They observed that flax fibers reinforced epoxy composites have higher HRR and more burning rates as compared to the glass/epoxy composites.

Borysiak *et al.* presented flammability properties of wood-polypropylene composites [53]. They concluded that the wood-PP composite specimens have shorter time to ignite than the pure PP specimens. However HRR, MLR and toxic gas emissions are reduced by wood reinforcements in PP.

4.2 Effect of matrices

Thermal degradation of polymers is fast, therefore polymeric matrices have poor flammability behavior. Shah *et al.* studied the properties of polypropylene against flame and heat [25]. Polypropylene is weak against fire, how they can be improved after particulate reinforcement? Being thermally weak, a polymer matrix itself has no prominent role in improving the flame resistance of the composites; it depends upon the reinforcements and fillers. Natural fiber reinforcements generally reduce the thermal stability of the polymer matrices and increase their flammability. Russo *et al.* studied the effect of kenaf fiber on the flammability of different polymers [54]. They found that the degradation temperature of virgin polymers was higher than the kenaf fiber reinforced polymers. However, some nano particle fillers may increase the thermal stability and flame resistance of polymers through different mechanisms. Flammability of different polymeric matrices can be compared to each other on the basis of their LOI and HRR values as shown in Table 1.

4.3 Effect of treatments

Different types of chemical or physical treatments modify the interfacial chemistry which leads to the improvement of different properties of composites. Carosio *et al.* investigated the influence of plasma surface treatment on PET fabric nanoparticle composites [55]. They concluded that plasma treatment has a positive effect on thermal stability. The results also showed that plasma treatment affected the combustion behavior and improved the flame resistance. Lee and Wang reported the effect of lysine-based diisocyanate (LDI) as a coupling agent on the properties of bamboo fiber based biocomposites [56]. The thermal stability of the composites was improved by increasing the LDI content, but still lower than the virgin polymers. Lee *et al.* studied the effect of silane coupling agent on the properties of the kenaf/polylactic acid (PLA) composite [57]. They observed that silane treatment improved the storage modulus of the composite. Sudhakara *et al.* characterized borassus fruit fiber/polypropylene (BFF/PP) composite after NaOH surface treatment and used MAPP as a compatibilizer [58]. They found that the alkali surface treated composites performed better under thermal loads. MAPP also played a role in the thermal stability of the BFF/PP composites.

4.4 Effect of flame retardant fillers

4.4.1 Types of flame retardants based on mechanism

4.4.1.1 Gas phase flame retardants

Gas phase flame retardants minimize the heat release in the gas phase from combustion by releasing the reactive free radicals that react, inhibit the chain reactions whereby the decomposition products of combustible materials propagate combustion [59,60]. Examples of gas phase flame retardants are halogen (F, Cl, Br, I), phosphorus (P-O or P) etc.

4.4.1.2 Endothermic flame retardants

Endothermic flame retardants work in both gas and condensed phase through endothermic decomposition of releasing non-flammable gases like H_2O, CO_2, which dilute the fuel and cool the polymer. The lower substrate temperature slows the pyrolysis rate. These materials also leave behind a ceramic-like residue, which protects the underlying polymer [61]. Examples are magnesium hydroxide, aluminium hydroxide, mixture of huntite and hydomagnesite.

4.4.1.3 Char forming flame retardants

Char forming flame retardants works in the condensed phase by preventing fuel release through binding up fuel as non-pyrolyzable carbon (char) and providing thermal insulation for underlying polymer through the formation of char protection layers [62]. Charing mechanism in polymers has shown in Fig. 3. Examples are organophosphorus, ammonium polyphosphate, etc. For example the organic groups on POSS cages undergo homolytic Si-C bond cleavage at 300-350°C in air. This process is immediately followed by fusion of POSS cages to form a thermally insulating and oxidatively stable silicon oxycarbide “black glass” surface char (Si-O-C ceramified char.

4.4.2 Types of flame retardants based on mode of action

4.4.2.1 Reactive flame retardants

Reactive flame retardants producing a special kind of components by assembling with polymers. This resists polymer composites from the propagation of flame. In addition, they have no plasticising effect and do not affect the thermal stability of the polymer [63]. Examples are aluminium hydroxide, organochlorines, etc.

4.4.2.2 Additive flame retardants

Additive flame retardants are not chemically bond with base polymer, but they act as plasticiser, otherwise they are considered as fillers. These fillers grafts the chemical groups onto these materials to enable them to become integrated without losing their retardant efficiency [64].

4.4.2.3 Combinations reactive and additive flame retardants

The combinations of reactive and additive flame retardants are able to create a modified additive, synergistic or antagonistic effect. The synergistic effect occurs when they are used together with specific flame retardants. The flame retardants/synergist systems have achieved great importance in practical use because they are usually less expensive than flame retardants used alone.

4.4.3 Types Flame retardants based on their functional element

4.4.3.1 Mineral flame retardants

Metal hydroxides: Metal hydroxide flame retardants are low-smoke flame retardant compounds. The flame retardant are decomposing endothermically into water and metal oxide upon heating. The released water vapour, in an amount of about a third of total metal hydroxide used, dilutes the combustible ambience while removing the combustion heat. The oxide by-product formed from the hydroxide decomposition chars on the surface of polymeric materials to prevent heat and oxygen from approaching the polymer [65,66]. Examples are aluminium tri-hydroxide ($Al(OH)_3$), Magnesium di-hydroxide ($Mg(OH)_2$), Antimony trioxide (Sb_2O_3), Expandable graphite, etc.

Hydroxycarbonates: Hydroxy carbonate flame retardants are not only alternative to metal hydroxides, but also environmentally compatible and act both condense as well as gas phase through endothermic reaction. They release water to the gas phase, which dilutes the flame. All carbonates release CO_2

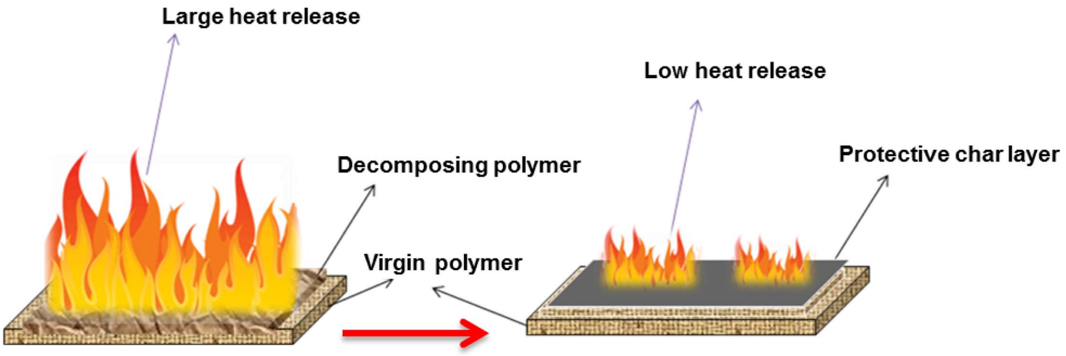


Fig. 3. Charing mechanism in polymers

at high temperatures but only magnesium and calcium carbonates release it below 1000°C, with magnesium carbonate presenting the lowest release temperature (550°C). The oxides produced by the decomposition can contribute to the formation of an insulating charred layer [67].

Borates: Borate flame retardants are environmentally friendly, low mammalian toxicity and low volatility fillers. They decomposition leads to the creation of glassy protective layer which acts as barrier for polymer chain oxidation. Their endothermic decomposition (503 KJ/Kg) between 290 and 450°C liberates water, boric acid and boron oxide (B_2O_3) [68]. Examples of most frequently using borates is zing borate ($2ZnO.\ 3B_2O_3.\ 3.5H_2O$).

4.4.3.2 Halogenated flame retardants

Halogenated flame retardants are very effective in capturing the free radicals (Equation 1) so that it resists the capability of the flame propagation. The efficiency of the halogens has decreased with the increasing the size ($F < Cl < Br < I$). Fluorinated compounds are very stable because of its stable bond and decompose at much higher temperatures than most organic matter burns. But in the case of Iodine, it has loose bond so it decompose at slightly elevated temperatures. Consequently, only organochlorine and organobromine compounds are used as flame retardants.

Brominated and chlorinated flame retardants: Brominated flame retardants have higher trapping efficiency and lower decomposing temperature so that it is available at a high concentration in the flame zone. Their flame retardancy mechanism is similar for all compounds and involves decomposition upon heat application, to prevent flammable gas formation [69]. Examples are Polybrominated diphenyl ethers, hexabromocyclododecanes, and tetrabromobisphenol-A.

Chlorinated flame retardants have lower stability than brominated, so higher quantities are needed to accomplish comparable flame retardancy. They have partial plasticising and thermal stability at higher temperatures (222°C). Cycloaliphatic chlorinated compounds have a higher thermostability and used as additive flame retardants in engineering plastics [70]. Examples are Chlorinated paraffins, antimony trichloride, etc.

$$R\text{-}X \longrightarrow R^* + X^*;\ \ HX + H^* \longrightarrow H_2 + X^*;\ \ HX + OH^* \longrightarrow H_2O + X^*\ \ (R\text{-}X = \text{flame retardant};\ X^* = Cl,\ Br) \qquad (1)$$

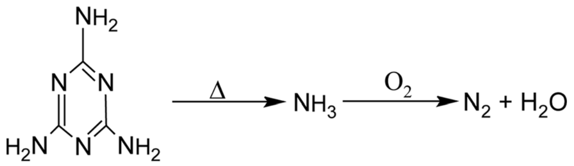
4.4.3.3 Phosphorous

The phosphorus flame retardants act in the condensed phase while metal phosphates may also act in the gas phase. They are very much effective with the materials containing rich oxygen. These flame retardants transformed into phosphoric acid by thermal degradation, and water released. A protective layer consists of interpenetrating networks of carbon and phosphorous oxide is developed by the phosphoric acid (Equation 2). The formation of the radicals P and PO are interrupting the radical chain mechanism of the combustion process [71]. Examples are ammonium polyphosphate, chlorophosphates, bromophosphate, etc.

$$\underset{\text{Ammonium Polyphosphate}}{(NH_4PO_3)} \xrightarrow[-NH_3]{>250^0C} (HPO_3) + Cx\,(H_2O) \longrightarrow (C_x) + (HPO_3).\ H_2O \qquad (2)$$

4.4.3.4 Nitrogen containing flame retardants

Nitrogen containing flame retardants are environmental friendly, low toxicity and low evolution of smoke flame retardants. They may act by the release of inter gases like ammonia, nitrogen into the gas phase or condensation reactions in the solid phase (Equation 3). The reaction with flame of these flame retardants is to make charring because of abundant nitrogen. These flame retardants are suitable for recycling because they have high decomposition temperatures [72]. Examples are Melamine, Melamine cyanurate, Melamine polyphosphate, Ammonium poly phosphate, etc.



Melamine

(3)

4.4.3.5 Silicon-based flame retardants

Many forms of silicon compounds have been explored as potential flame retardants to polymeric materials, such as silicones, silanes, silsequioxane, silica and silicates.

Silicones: Silicone flame retardants exhibit relatively low rates of heat release, and low dependence of the rate of release on external heat flux in the gas phase, because of the lower burning rate is attributed to the accumulation of the silica ash layer at the silicone fuel surface. These are excellent thermal stability and high resistance with very limited release of toxic gases during decomposition [73,74].

Silica: Silicon has been playing a protective role when loaded one of its forms of silica nanoparticles or layered silicates in addition silica affects the thermal stability of thermoplastic polymers. Flame retardant Mechanism, during combustion it forms of an inorganic barrier on the surface that protects the underlying polymer from oxygen and also reduces heat transfer [75].

4.4.3.6 Nano-metric particles

Nano-metric particles when individualized and properly dispersed in polymer matrices are known to contribute to the enhancement of properties such as thermal, mechanical or flame resistance [76,77].

Carbon nanotubes: Carbon nanotube (single, double and multi-walled) flame retardants are more sought after than carbon nano-fibers because of CNT particles not only provides non-flammability in relatively small concentrations compared to the other fillers and also enhancing the material strength. CNTs percolate to form a network at very low loading in the polymer matrix and lead to substantial enhancement of several functional properties such as flame retardant properties [78-81].

Nano clays: Nano-scale particulate flame retardant additives have the ability to incorporate polymers between the layers by intercalation or exfoliation. They do not act as flame retardants on their own, but may together with other flame retardants, enhancing the charring and reduce the heat release of the polymers. Further taking into account, eco-friendliness, mechanical and physical properties are required for end applications but due to processing difficulties better to use nanoscale additives as a flame retardant in polymers, because at low loading with no additional flame retardants in the system, heat release rates and mass loss rates were greatly reduced compared to neat polymers [82-84].

Metallic oxide particles: Metallic oxide flame retardants have proven that, they are efficient additives for improving the thermal stability and morphological structure of char residues and the flame retardant of the polymers because of not only strong interactions between polymer and nano-particle surface and also the degradation temperature of these particles are higher than the polymer processing temperature [85,86]. Reaction mechanism of aluminium trihydraxide on decomposition is shown in Equation (4). Examples are magnesium oxide (MgO), zinc oxide (ZnO), and ferric oxide (Fe_2O_3), Titanium oxide (TiO_2).

$$2\ Al(OH)_3 \longrightarrow Al_2O_3 + 3H_2O$$

$$\text{(endpthermic 1050 kj/kg)} \qquad (4)$$

5. MECHANISM OF FLAME RETARDANCY

Five specific mechanisms by which flame retardancy may occur: physical dilution, chemical interaction, inert gas dilution, thermal quenching and protective coatings [87].

Physical dilution: The flame retardant can act as a thermal sink, increasing the heat capacity of the product or reducing the fuel content to a level below the lower limit of flammability. Inert fillers such as glass fibers and microspheres and minerals such as talc act by this mechanism.

Chemical interaction: The flame retardant dissociates into radical species that compete with chain propagating and branching steps in the combustion process. This is the general flame-retarding mechanism by which brominated flame retardants operate.

Inert gas: Flame-retardant additives produce large volumes of non-combustible gases when the product decomposes during combustion. The gases dilute the oxygen supply to the flame or dilute the fuel concentration below the flammability limit. Metal hydroxides, metal carbonates and some nitrogen producing compounds function in this way when used as flame retardants.

Thermal quenching: Endothermic reaction in the presence of flame to release water molecules that quenching the polymer and dilute the combustion process. Examples are Metal hydroxides and carbonates.

Char-formation: Char-forming flame retardants react to form a carbon layer on the material's surface. This layer insulates the polymer, slowing pyrolysis, and creates a barrier that hinders the release of additional gases to fuel combustion. Phosphorous compounds that decompose to give phosphoric acid and intumescent systems operate by this mechanism.

6. LABORATORY FLAME TESTING

The flammability of the polymer materials can be characterized by the ignitability, flame spread rate and heat release. There are commonly used flammability test for polymers are presented in Table 2; they are radiant panel, limited oxygen indices (LOI), underwriter's laboratories (UL 94), and cone calorimeter.

Radiant panel test (RPT) can be used to evaluate flame-retardant coatings for wood-base materials and what extent the thickness of the surface finish material and the substrate to which it is applied govern the flame spread behaviour of composite assemblies. RPT measurements can be used to classify flooring materials. Samples are exposed to an external flux and ignited by an additional burner on one side. Measurement results are the distance of flame propagation. Furthermore, the temperature inside the RPT is measured with a thermocouple [88].

Limiting oxygen indices (LOI) is the minimum concentration of oxygen in a mixture of oxygen and nitrogen that is needed to support the flaming combustion of a material. It is expressed in volume percent (Vol%). It is used to indicate the relative flammability of materials [89].

$$LOI = 100 \times [O_2] \ / \ ([O_2] + [N_2])$$

UL94 testing procedures and rating system for thermoplas-

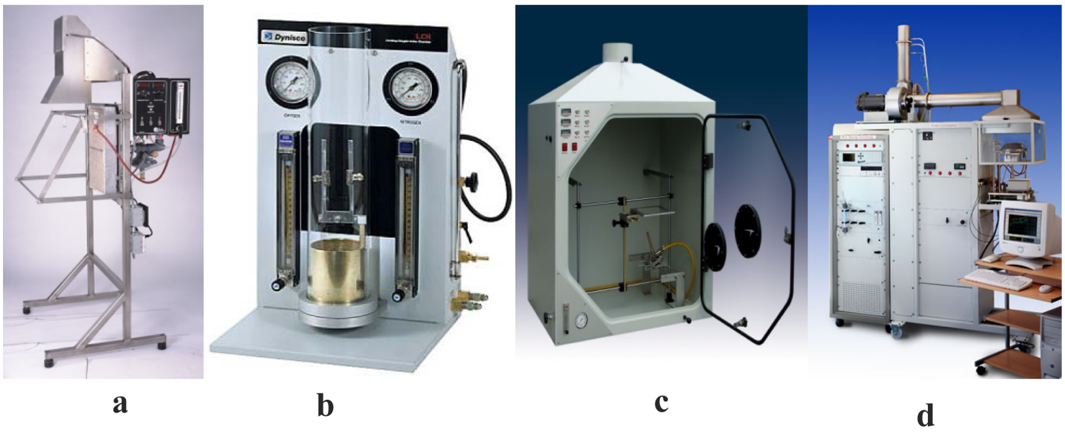


Fig. 4. Flame testing apparatus: (a) Radiant panel; (b) LOI; (c) UL 94; (d) cone calorimeter

Table 2. Flammability tests for polymers [92]

ASTM standards	Description	Characteristic measure
E162-87	Radiant panel (Fig. 4a)	Flame spread
D2863-87	Limited Oxygen indices (Fig. 4b)	Ease of ignition
UL94	Vertical burn (Fig. 4c)	Ignition resistance
E1354-90	Cone calorimeter (Fig. 4d)	Heat release and smoke

tics flammability are the generally accepted standard throughout most of the world. It measures ignitability and flame spread of vertical bulk materials exposed to small flame[90].

Cone calorimetry is a technique used to quantify the flammability of materials by determining various flammability parameters Cone calorimetry is one of the most effective medium sized flame behaviour tests used to study the rate of heat released by materials exposed to radiant heat flux. Its principle is based on the measurement of decreasing oxygen concentration in the combustion gases of the sample that is subjected to a given heat flux (10–100 kWm^{-2}) [91].

7. SUMMARY

In this review, we have presented a broad range of flammability of natural fiber reinforced polymer composites and flame retardant additive systems. Flammability of natural fiber reinforced polymer composites remains a very complex scientific problem for which no single solution can be found, especially with regard to the extensive diversification of polymer matrices available. The selection of suitable flame retardants supports to create flame resistant composites, and prolonging the variety of their uses. The use of relatively low amounts of flame retardants with polymers shows very promising results. Among non-halogenated flame retardant additives, phosphorus and nitrogen-based compounds have proved to be very powerful solutions, especially in matrices containing oxygen or nitrogen atoms in their backbone, while silicon-based additives also appear to provide efficient solutions. However, the improvement of flame retardance in natural flame reinforced composite materials is being a great task, but the effective selection and adding of most appropriate flame retardant additives will hopefully contribute to producing flame resistant natural fiber reinforced polymer composites.

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