Review of Fire-Retardant Materials in Bamboo and Other Lignocellulosic Substrates: Mechanisms and Efficacy

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Abstract

The combustion behaviour of wood and bamboo presents a significant challenge for their use as construction materials due to their lignocellulosic nature. Historically, various chemical treatments have been employed to enhance the fire resistance of lignocellulosic materials. Early methods utilized boron-based and phosphorus-based compounds. Recently, advancements have introduced silicon-based and nanomaterial-based fire retardants (FRs). Additionally, carbon-based materials, such as carbon nanotubes and graphene, have garnered considerable interest from researchers. This paper examines the range of fire-retardant materials applied to lignocellulosic substrates.

Key Words: Fire retardant, Bamboo, lignocellulosic, Charring, Mechanism

Abbreviations: FRs – Fire retardants

Introduction

The behaviour of materials in a fire depends on several factors such as ease of ignition, flame spread, fire endurance, rate of heat release, ease of extinction, smoke evolution, and toxic gas evolution. The reduction of any of the above parameters than base material is defined as fire retardant material [1]. Natural fibers are Flammable, i.e. 'fuel' evolved from the decomposing polymer react with oxygen from the surrounding air. When the lower flammability limit is reached the mixture will either 'flash' ignite due to the presence of an external flame or spark, or auto ignite if the temperature is above the auto-ignition temperature [2]. The effect of heat on fibers will be either physical or chemical or both. Glass Transition Temperature (T_g), Melting Temperature (T_M) are associated with Physical changes. Pyrolysis Temperature (T_P), Combustion temperature (T_c) are associated with Chemical changes with heat. At T_p volatiles and gases produced and they are the fuel for the combustion process. When combustion starts heat released with process will be transferred again to the adjacent fibers and this cyclic temperature transfer process will continue until the enough fuel present [3]. in cellulose pyrolysis occurs via two different competing reaction. In one reaction, cellulose undergoes direct dehydration in which processes includes removal of water content , cross linking of cellulose fibers with evolution of water and decomposition of this dehydrocellulose in to char and volatiles. And in other, an intermediate product called Levoglucosan is produced. It is an unstable product and reduced into various flammable gases and also char. Combustibility of the material can be measured using the Limiting oxygen index (LOI), wich represents the volume percentage of oxygen present in the air required for a sustained burning to occur eg; LOI of cotton fiber is 19. Normal air contains 21% by volume oxygen. So LOI with less than 21 will burn readily in air [3][4].

Different components in wood, ie cellulose hemicellulose and lignin decomposes under heat in different range of temperature. Decomposition of cellulose takes place between 260^0C and 350^0C and produce Flammable volatiles and gases, non-combustible gases (eg; CO_2), tars and char[4]. Hemicellulose decomposition takes place in between 200^0C and 260^0C and forms more noncombustible gases and less tar than cellulose. Lignin has larger temperature for decombosition due to the presence of aromatic rings. Lignin decombosition starts at about 160^0C and continues upto 400^0C. higher temperature required to break aromatic rings. Lignin contributes to more char formation as compared to cellulose and hemicellulose [5]. And it is proposed that higher cellulose content contribute to the higher flammability and higher lignin content helps for lower flammability.

As natural fibers are prone to ignition, and bamboo is a construction material since centuries, it become necessarily need to protect it from fire and heat. Other polymers which are using for various products are also subjected for fire retardant application in it. As natural fiber is a renewable resource and its usage also promote carbon locking which helps against global warming and climate change activities, safer and durable natural fiber for construction and other usages should be promoted [6]. For making safer usage along with strength properties of natural fiber fire safety is also important.

The history of fire retardants dates back to ancient civilizations. In Egypt and China, substances like vinegar and alum were used to reduce flames. In China, a mixture of vinegar and alum was applied to wood and covered with clay, helping to slow the spread of fire. Around 3,000 years ago in Egypt, reed and grass were used for roofing after being soaked in seawater. Evidence also shows that alum was used as a fire retardant in ancient Rome. The first scientific approach to fire retardancy came in 1821, when Gay-Lussac developed fire retardants for lignocellulosic materials using ammonium phosphate and borax. In recent centuries, most advancements in fire retardants for cellulosic materials have focused on textiles[7].

Fire retardancy of the natural fibers can be achieved by several ways. Physical modification of Natural Fibers through thermal treatment charring, Chemical modification by additives incorporation into fibers or by applying some external material which can act as barrier between heat source and natural fiber.

Broadly chemical Fire retardants applying for wood/Lignocellulosic materials can be classified in to [8];

1, Intumescent

2, Non-Intumescent

Intumescent types are applied only on the surface of the material. Non intumescent types are allowed to impregnate in to wood material.

There is different mechanism for fire retardancy. Each type of FRs follow one or more type of methods based on the chemical constituents present in it. There are different mechanisms for fire retardancy [9]

1. Physical Dilution
2. Chemical Interaction
3. Inert Gas
4. Thermal Quenching
5. Char forming

In physical dilution flame is suppressed by cooling, barrier formation, changing heat capacity. chemical mechanisms are always accompanied by one several physical mechanisms such as endothermic dissociation dilution of fuel. Nano clays, Glass fiber compounds function in this way. Chemical interaction is a radical mechanism, in which FR additives or their by-products interferes and catch the free radicals which are produced by decomposing polymers through combustion. The free radicals are seized so that the system cools down and resist/reduce the flammability. Halogenated (brominated) FR additives usually act by this mechanism. The free radicals i.e., OH* and H* formed from polymers react with halogen free radicals in gases phase so that the flame inhibition occurs. FR additives in inert gas mechanism, vaporize and evolve large quantities of non-flammable gases/inert gases such as nitrogen, ammonia, carbon di-oxide) into the gas phase or by condensation reaction. These inert gases contaminate the oxygen which is nearer to the combustion source, so that it dilutes the flammable fuel concentration. Metal hydroxides, metal carbonates and some nitrogen producing compounds function in this way. FR additives in thermal quenching mechanism release crystal water molecules on combustion (high temperatures) through endothermic chemical reaction. It thermally quenches (sudden cooling) the materials (polymer matrix) and dilute the combustion process. Halogen free FR additives, metal hydroxides and carbonates usually act by this mechanism. Fire retardants in Char-forming mechanism form carbonaceous char layer on polymer matrix, which can inhibit the oxidation of carbon atoms in the polymer, so that it is protected from the further action of flame. Carbonaceous char layer also reduced mass volatiles, increase thermal insulation, obstructs the flammable gases and increases the thermal capacity. Halogen free FR additives such as phosphorus, nitrogen etc., usually act by this mechanism [9]. Or this mechanism can be categorised in to two basic mechanisms called, Gas phase mechanism and condenced phase mechanism.

In gas face mechanism FRs removes active radicals which present in combustion area. These radicals, H,O,OH react with combustible gases and release heat which in turn use for further propagation of flame. By removing these radicals from flame area, the combustion reaction will slow down and there by the flame also. Amount of combustibles released will be same as that of without adding FRs, but as the amount of flame retarding agent increases the heat released will be less. Halogen containing FRs are example for thise type of mechanism [2].

Condensed face mechanism is work either by production of char layer eg; Phosphorous based FRs or Production of water molecules which inhibits flame propagation. Metal hydroxides $Mg(OH)_2$ is example for water producing FRs. Char layer formed will protect inner material from combustion. FRs acting via this mechanism alters the pyrolytic path of the material and reduces the amount of combustible gases, and favours the production of char and water. [2]

Fire retardants Ligno - cellulosic materials

Several chemicals are used for protecting natural fibers from heat and flame. Based on the elements present in the fire retardant material, we can broadly classify them. Mineral flame retardants, halogen containing flame retardants, Phosphourous based flame retardants, Nitrogen based flame retardants, Silicon containing flame retardants, Nano metric particle etc are different categories of flame retardant classification based on above mentioned criteria [10].

Halogen Based flame retardants

Free radicals released on polymer decomposition H. and OH. Which helps to continue the combustion by a cascade chain mechanism. These free radicals are highly reactive and react with combustible gases and release heat. A part of this heat transferred to polymer and thus burning process continues [2]. Halogenated flame retardants works in mechanism such that they help to remove these free radicals, stopping the chai decomposition and therefor the combustion of the polymer:

$RX \rightarrow R. + X.$ X= Br or Cl

$X. + R'H \rightarrow R'. + HX$

$HX + H. \rightarrow H2 + X.$

$HX + OH. \rightarrow X.$ [10]

These FRs are specially used for making consumer articles. These are efficient for reduce or prevent the propogation of fire. Aromatic halogen FR additives are less effective than aliphatic and alicyclic due the bond energy. Flourine and iodine in halogen group cannot be use as FRs because of high electronegativity and large molecule size respectively [11].

Due to low cost, procesability, low reduction on the physical/mechanical properties halogenated FRs were used since 1930s. however Incresed smoke release, release of corrosive gases, (eg; HBr) on burning leads to critical observarion on this type of FRs. These types FRs are needed in higher percentage otherwise, once the halogen consumed by the fire remaining polymer will burn if exposed in air. And this higher percentage of halogen will lead to higher production of smoke and corrosive gases [12]. The FRs must have sufficiently lower decombosition temperature than of material wich is to be protected for efficient result. Most common halogen containing FR compounds are Tetrabromobisphenol A(TBBPA), Polybromodiphenul ether (PBDE), Hexabromocyclododecane (HBCD) and Tetrabromophthalic anhydride (TBPA).

Akhil et.al [13] studied fire retardancy effect of brominated coir fiber. Coir fiber brominated with saturated bromine water and then treated with stannous chlorine solution. After drying and grinding into nano dimension, it used for composite preparation by mixing with epoxy resin. Limiting oxygen index values of coir fiber – epoxy composite showed 26% increase as compared to pure epoxy.

Phosphorous Based FRs

Due to the ecological concerns, phosphorous FR additives are used as halogen free FR additives which have similar flame-retardant mechanism as that of halogenated FR additives. They act in the condensed phase (char former and char enhancers) while metal phosphates may also act in the gas phase. The importance of these FR additives is high effective mode of action on fire with low concentration amounts. They completely rest on the matrix structure and very active with the polymer which contains rich oxygen in their structure.

Mechanism: Phosphorous FR additives are works efficiently in the solid phase of polymeric materials. First these phosphoric FR additives are converted into their relative acids and then to polyacids by the process of thermal decomposition. Acids esterify and dehydrate the pyrolysing polymer to form un-sutured compounds with char. Poly phosphoric acids inhibit further pyrolysis process to from carbonaceous layer, this will protect the polymer materials from oxygen and radiant heat in high temperatures. General reactive flame-retardant mechanism of phosphorus additives is as shown [9].

$[PO]^{-} + [H] = [PO]\ [H]$

$[H]\ [PO] + [H] = H2 + [PO]^{-}$

$[H]\ [PO] + [-OH] = H2O + [PO]^{-}$

Dorez et.al [14] studied fire reaction on different natural fibers in which Ammonium polyphosphate is used as fire retarding agent. Polybutylene succinate (PBS) is used as matrix. Flax used as natural fiber for reinforcement. addition of APP leads to early degradation of PBS matrix. APP enables the matrix charring. Strong charred layer helps decrease pHRR. Decrease in TTI is also found at cone calorimeter. They also found through cone calorimeter test that, thermal degradation of APP leads to the formation of phosphoric acid, which combines with the hydroxyl function of flax forming a phosphorus ester. This ester catalyzes the dehydration of the fibers and leads to the formation of a carbonaceous structure.

Suardana et al. [15] studied the flammability of bio-composites in PLA and PP matrices, using Diammonium Phosphate (DAP) as a fire retardant. The fibers, treated with DAP, were tested for flammability through horizontal burning tests and thermogravimetric analysis. The composites, made from PLA, PP, and natural fibers (coconut and jute), were hot-pressed. DAP-treated composites showed lower decomposition rates compared to untreated ones, with higher DAP concentrations further reducing decomposition. Burning rate and weight loss also decreased with increased DAP. For composites treated with 5 wt.% DAP, burning rates improved by 49.15% (coconut-PLA), 35.4% (coconut-PP), and 23.8% (jute-PP) compared to untreated composites. The char formed was more intact and firmer for DAP-treated samples, while untreated composites produced thin, brittle, and disintegrating char.

Chen et al. [16] investigated the fire-retardant effect of Diammonium Phosphate (DAP) on pine wood. One set of wood samples was treated with glucose-DAP mixtures at concentrations ranging from 24.1% to 61.2%, and heated at 160°C for 2, 4, or 6 hours before drying. Another set was treated by impregnating DAP at concentrations of 10.6%, 14.5%, and 19.4%. Fire tests were performed according to ASTM E69-99, using specimens with a 9.5 × 19 mm cross-section and 1020 mm length. Two sticks (510 mm each) with similar chemical retention were connected to form the specimen. The specimen was positioned 25 mm above a Bunsen burner, and the flame height was set to 280 mm, with the flame applied for 3 minutes. After the test, flame duration, glowing duration, and weight loss were recorded. The glucose-DAP treated samples did not sustain any flame after the 3-minute test, while DAP-treated samples retained flames for 291s, 216s, and 55s at 10.6%, 14.5%, and 19.4% DAP concentrations, respectively. Both glucose-DAP and DAP-treated samples extinguished when the flame was removed. However, the retention test indicated that prolonged heating resulted in more water-insoluble thermal condensation products, suggesting that glucose-DAP treated wood had better fire retardancy than DAP-impregnated wood.

Effect of six different phosphorus-based fire retardants (FRs) on cotton cellulose has been studied by Gaan et al [17]. The FRs used were Pyrovatex CP (PCP), Diammonium Phosphate (DAP), Phosphoric Acid (PA), Tributyl Phosphate (TBP), Triallyl Phosphate (TAP), and Triallyl Phosphoric Triamide (TPT). They found that PCP, PA, and DAP were more effective than the others at increasing the Limiting Oxygen Index (LOI) of cotton. PCP, DAP, and PA are water-soluble and were applied in aqueous solutions, while TAP, TPT, and TBP are water-insoluble and were applied in acetone.

FRs were applied at three different concentrations, and the LOI of untreated cotton was 18.5. With increasing phosphorus content, the LOI of treated samples increased. DAP-treated cotton showed the highest LOI: 24.5, 28.5, and 35.5 at 1%, 2%, and 4% concentrations, respectively. PA also showed good LOI values (24, 27, and 31) at the same concentrations.

Char formation was analysed using SEM. PA, PCP, and DAP-treated samples produced firm, intact chars, while TAP, TBP, and TPT-treated samples formed thin, brittle chars. TAP and TBP samples had completely disintegrated chars, likely contributing to their lower LOI values. The surface morphology of chars influenced the effectiveness of the FRs. PCP-treated samples showed prominent chars, likely due to both nitrogen and phosphorus content, while PA and DAP samples had less prominent char but maintained fiber integrity, possibly due to cross-linking by polyphosphoric acid. This study showed that phosphorus-based FRs improved the flame retardancy of cotton fibers during combustion [17].

Evren Terzi et al. [18] studied the fire performance of solid wood and plywood treated with MAP, DAP, and other non-phosphorus FRs. Samples were treated with 1wt% and 4wt% FRs. Solid wood was taken from the sapwood of Scots pine, and plywood veneers were from birch and larch. An 11-ply plywood was used for testing. ASTM E69 and ASTM 1354 methods were used for fire performance and cone calorimeter tests, respectively. Heat release rate (HRR) was calculated using the oxygen consumption method, and mass loss was also measured.

The main result from the fire tube test was the residual mass fraction after 10 minutes of exposure. A higher residual mass indicates better fire retardancy, with less combustible volatile production. Solid wood samples treated with 1wt% MAP and DAP showed higher residual mass than untreated samples. At 4wt%, MAP and DAP-treated solid wood had even higher residual mass fractions (0.603 and 0.439, respectively). The average HRR of solid wood samples was significantly lower than untreated ones. Total heat release rate (THR) was much lower in the 4wt% FR-loaded samples. For plywood, the peak heat release rate (PHRR) was significantly lower for both 1wt% and 4wt% treatments, and the THR for 4 wt%. MAP and DAP was notably reduced.

Fire retardant effect of a mixture of FRs in ply wood and FR mixture contain Guanylurea phosphate (GUP), Ammonium polyphosphate (APP), Pentaerythritol (PER), and Melamine were studied by Wang et al. [19]. Among which GUP and APP are phosphorous containing. Fire retardant materials mixed in water and applied in Urea formaldehyde resin (UF) – Polyvinyl acetate (PVAc) Resin. This mixture painted upon the 5-ply plywood. Total Heat release of FR painted samples significantly decreased as compared to control samples. Heat releasing process is also delayed. FR coated sample produced leage amount of ash and showed lower mass loss rate. The author states that the main mass loss took place only 6.9min exposure to the heat source. Time to ignition (TTI) is also tested. FR coated sample showed better TTI. TTI of coated sample show about 7mins, whicj is 10 times more than control samples. Carbon monoxide (CO) production delayed significantly and kept at very low level for atleast 5 mins in FR coated samples.

Effect of Resorcinol di phenyl phosphate (RDP) as flame retardant in Zinc oxide coated kenaf fiber – Poly lactic acid (PLA) composite were studied by Zhou et al. [20]. RDP dissolved in chloroform and ZnO coated kenaf was added in to the solution. Stirring is done for 30mnts so that the RDP would be absorbed on the ZnO coated kenaf sufficiently. PLA pellets were added in to the mixture and injection moulding is used for making suitable sample for different tests. FR composites show better thermal performance than pure PLA. LOI and UL 94 tests also show improved results for ZnO coated kenad RDP system. All samples receive V-0 level. 15wt% RDP loaded samples show LOI 0f 31.2 as compared to pure PLA with LOI 20. By adding ZnO coated kenaf is added, LOI is slightly decreased.

Izran et al. [21] studies flame retardant properties of particle board made with kenaf fiber particles. Kenaf particles were treated separately with 8% and 10% solutions of MAP, DAP and a mixture of boric acid , GUP, phosphoric acid. Among these FRs show best result for fire retarding effectiveness followed by MAP, DAP. Phosphoric acid treated samples show ignition time of 120s, DAP and MAP treated samples show 100s. the untreated samples have an ignition time of just 50s. Chindaprasirt et al [22] analysed the effects of diammonium phosphate (DAP) on the properties of wood flour/expanded polystyrene waste composites. The DAP modification improved fire retardancy of the composite, but it also increased its water absorption.

Matko´ et al. [23] also investigated the flame retardancy of polyurethane/cellulosic composites using DAP as a flame retardant for the cellulosic component. A polyurethane/wood flake composite had a LOI value of 23% and a polyurethane/corn shell composite had a LOI value of 20%. When DAP was used as the flame-retarding agent, the composites had LOI values of 30% and 29%, respectively. UL 94 ratings improved from H-B (no flame retardant) to V-0 (DAP). Diammonium phosphate ((DAP), (NH4)2HPO4) is a condensed phase flame retardant that has been used for a long time as a nondurable flame retardant for cellulosic textile.

Nitrogen containing Fire Retardants

Major nitrogen containing FR additives include melamine and compounds containing melamine eg; melamine cyanurate, melamine polyphosphate, melamine poly (zinc/ammonium) phosphate, melem etc. there are several N – P materials like APP which shows effect of both N and P in fire retarding action [9].

Melamine is thermally stable product having a melting point of 345°C. On sublimation I absorb significant amount of energy and helps to decrease the temperature. At higher temperature melamine decomposes and eliminate ammonia which dilutes oxygen and combustible gases and also produce residues in condensed phase namely, melam, melem and melon. Melamine can also generate thermally stable salts with strong acids like melamine cyanurate, melamine phosphate , and melamine polyphosphate. Melamine and melamine salts are characterized by various flame retardant mechanisms. Upon heating melamine salts dissociates and form melamine volatile [10].

Nitrogen based FRs works in both gaseous and condensed phase. In gas phase, nitrogen based molecules released which are stable and helps to remove and decrease flammable volatiles from burning area. And in condensed phase complex nitrogen compounds are produced which are capable of producing char. The reaction mechanism of melamine is given:

$$\text{Melamine} \xrightarrow{\text{Heat}} NH3 \xrightarrow{O_2} N2 + H2O \qquad [9]$$

Several FRs like APP, DAP wich contains Nitrogen along with P is works on the synergic effect of both nitrogen and phosphorous.

Si Containing Fire retardants

Silicone materials have been produced commercially since the beginning of the 1940s. Over the past 60 years, silicone materials have grown into a billion-dollar industry, and are used in many applications in civil engineering, construction building, electrical, transportation, aerospace, defence, textiles, and cosmetics industries. Silica containing materials also provide better thermal resistant properties. Silicons provide slow burning rate and low emission of toxic smokes. Silicons exposed to elevated temperatures provide under oxygen provide silica residue. The shielding effect provide helps for the development of silicon based fire retardants. Silica residue acts as a insulating blanket which is a barrier between fire and composite material [24]. Li et al. [25] studied effect of a silicon contining FR, aminopropyltriethoxysilane in cotton fiber.

Mineral containing (Metalic oxides, hydroxides and hydrocarbonates) FRs

Metals reacts with oxygen to form metallic oxides. These are used for improving thermal stability and morphological structure of char residue eg; magnesium oxide , zinc oxide , ferric oxide.

Metal hydroxides mineral FR additives with combination of hydroxides an carbonates. These are used low smoking FR additives and are low cost and abundantly available in nature. This type of FRs works through endothermic reaction and release water molecules eg; Aluminium trihydroxide (ATH), Magnesium di-hydroxide (Mg OH2), Antimony trioxide (Sb2O3).

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

$2Mg(OH) \rightarrow 2MgO+2H2O$ [9]

One drawback of the mineral additives is that, they should add in higher content to get better fire-retardant efficiency. And Hydroxycarbonates are not much studied for the fire retardant activity [26].

Similar to metal hydroxides hydroxycarbonates like calcium and magnesium hydrocarbonates undergo endothermic decomposition on fire. They release CO2 and H2O in the gas phase which dilutes the combustible gases in the flame and the metal oxide helps to create charred layer making barrier between composite and flame.

$Mg_5(CO_3)4(OH)_2 . 4H_2O \rightarrow 5MgO + 4CO_2 + 5H_2O$ [27]

Effect of magnesium hydroxide in Flax – polypropylene composite were studied by El-sabbagh et al. [28]. Polypropylene matrix reinforced with 30 and 50wt% of flax fiber. Magnesium hydroxide at different levels on composite, 0,20,30 wt% is taken for study. LOI increased to 27 for 50wt% flax – 30wt% MgOH2 composite. For 20% loading of FR in 30 and 50wt% flax fiber, LOI increased to 21.1 and 23.3, but the increase is not significant. But none of the compound succeeded UL94 test as per ASTM D 635. Sample 30wt% MgOH and 50wt% flax showed a slow rate of burning.

Fire retardant effect of TiO2 and ZnO in wood were explored [29]. Coating of FRs in wood synthesised using a facile one-pot hydrothermal method. Wood particle dipped in the FR solution in an autoclave. Cross section images of wood with SEM showed wod surface including vessels are covered by FR mixture. Time to ignition (TTI) for untreated sample show 10s and for FR treated sample show 37s. heat release rate (HRR) showed difference in treated and untreated samples. HRR curve for treated sample was lower that treated samples, and the burning rate intensity and heat release were slowed and decreased. Mass loss rate (MLS) curves also showed better result for added FRs. MLS curve of treated samples was always lower than untreated samples. It suggests that less weight decreased with burning time. Less weight decreased means lower the heat produced.

Fly ash and red mud were used to increase the fire resistant properties of sisal and jute fiber – polyester composite [30]. Fly ash and red mud are industrial wastes from coal and aluminium industry respectively. The main constituents of Fly Ash are Silicon dioxide (SiO2), Aluminium Oxide (Al2O3) and Calcium Oxide (CaO). Red Mud contains Fe2O3, Al2O3, TiO2, CaO, SiO2, Na2O. both the materials contains several mineral oxides which in general known for fire retardant activity. Fire ignitability behavior of red mud/fly ash composite ndicates that these are not easly ignitable. Fire propogation index shows that FR loaded composite are better in comparison with MDS, polystyrene and wood.

Flame-retardant effect of magnesium hydroxide in polypropylene and sawdust/rice husk composites were explored by M.Sain et.al [31]. Flame retardancy was assessed using horizontal burning and oxygen index tests in this study. The synergistic effect of boric acid and zinc borate with magnesium hydroxide was also explored. Samples with 25% flame retardant loading showed a 50% improvement in flame resistance. A 20:5 ratio of magnesium hydroxide to zinc borate resulted in faster burning than composites containing only magnesium hydroxide. The study suggests that magnesium hydroxide exhibits a stronger endothermic flame-retardant effect compared to other additives. Replacement of magnesium hydroxide with these flame retardants reduces its effectiveness. Oxygen index results also indicated that higher magnesium hydroxide content led to higher LOI, with no synergistic effect from boric acid/zinc borate.

Kozlowski et al. [32] studied fire retarding effect of vermiculate in particle board made of hemp shives and urea formaldehyde as adhesive. Vermiculite is a mixture several minerals, a group hydrated aluminium-iron-magnesium silicates, and undergoes expansion on heating. Vermiculite is having high thermal insulation parameters. Non-combustible vermiculite products do not emit smoke or toxic fumes and make no hazard to the environment. Vermiculite applied in surface layer by mixing it with adhesive. HRR, THR, pHRR, TI, MLR, and smoke generation expressed as extinction coefficient and specific extinction area (SEA) are analysed. Every result except SEA show better result compared to control sample. TI of FR added composite show 14mns while control sample show just 33 secs.

Effect of Alumina trihydrate in natural fiber epoxy composite as a fire retardant material was studied [33]. Eucalyptus bleached fibers (EBF) fibers are used with epoxy matrix to produce the composite. ATH loaded in 5, 10 and 15wt.%. effect of APP is also studied, as synergic effect with ATH. Char residue formation increased as the amount of ATH loading increased. But increasing ATH concentrations deteriorates the mechanical properoties.

Gallo et al. [34] studied effect of ATH in Thermoplastic starch (TPS). Researchers also studied effect of adding coconut fiber (CF), such that the amount of ATH is replaced and its effect in the fire retardant properties. ATH decomposes endothermically into water and inorganic residue. Replacing a part of ATH with coconut fibers shows improved flame retardancy. TI, HRR etc tests were carried out. ATH loaded in 40wt.% and in another samples 10wt% of the ATH replaced with CF (30wt% ATH). TPS show LOI of 18.2 were TPS/ATH and TPS/ATH/CF show 24 and 24.7 respectively. Time to ignition (TI) for TPS was 23+-2 and same of TPS/ATH is 34+-2 TPS/ATH/CF is 38+-2 , which clearly shows improved flame retarding property. No dripping was found in both FR loaded samples. Heat release rate also showed better results for FR loaded samples. pHRR curves shows TPS/ATH, TPS/ATH/CF have significant reduction compared to control samples, 45,44.5% repectively. Increased residual mass production seen in TPS/ATH/CF material wich helps to protect the material from fire. Authors suggest that synergistic combination of ATH and CF shows promising results which helps for reduction in ATH, and replacing that part with CF.

Studies contected to analyse the effect of a hydroxycaronate, Hydrotalcite ((Mg4Al2(CO3)(OH)123H2O)0.5) in Ethylene vinyl acetate copolymer [35]. Other additives like magnesium hydroxide, Aluminium hydroxide, boehmite are added separately and results are evaluated. Results shows that 50wt% hydrotalcite loaded samples show slowest heat release rate and lowest evolved gas temperature.

Nano metric particle containing Fire retardants (nano clay, CNT, graphene)

Conventional flame retardants such as halogen based retardants have been widely used as these are having great fire retardant properties. But these FRs also produce some corrosive fumes, organic pollutants such as dioxins, furans etc. alternative method for halogen FRs, are used to avoid such problems. These alternatives include halogen free flame retardants such as ATH, magnesium hydroxide. But these FRs should be loaded in high quantity for satisfactory fire retardant effects. Research in nano metric particles for fire retardant properties aim to overcome the problems of traditional fire retardants. [25]

Nano metric particles helps for improvement of not only fire resistance properties but also thermal and mechanical properties. They allow considerable reduction in loading content as the interfacial area between the polymer and nanofiller is greatly increased. These particles can be classified as:

Layered materials : Nano clays, Layered silicates

Fibrous materials : Carbon nano tubes (CNT), sepiolite etc.

Particulate materials: Nano scale particulate additives; POSS, spherical silica nano particles Etc. [10]

Incorporation of low amounts (~10 wt.%) of organo modified nanoclay in a polymer matrix creates a protective layer during combustion. During heating, the viscosity of a molten polymer/layered silicate nanocomposite decreases with increasing temperature. This facilitates the migration of clay nanolayers to the surface. Heat transfer promotes thermal decomposition of an organo modifier and creates strongly protonic catalytic sites onto the clay surface, which can catalyse the formation of a stable char residue. The accumulation of clay on the surface of a material, limits the volatilization of combustible degradation products, as well as the diffusion of oxygen in to the material [36].

The main fire retardancy mechanisms in polymer/silicate layered nanocomposites may be the formation of a barrier against heat and volatiles by migration of clay nanolayers towards the material surface, followed by char formation together with increased melt viscosity for exfoliated nanocomposites. These mechanisms may modify the fire properties of nanocomposites differently depending on the fire test applied. In general, incorporation of nanoclays retards and reduces the peak heat release rate (PHRR), but does not lower the total heat release (THR). This may mean that almost all of the fuel released is combusted, hence almost no change in THR.

Carbon Nano Tubes (CNTs) are an interesting alternative to the use of conventional flame retardants and even for nanoclays. It has been studied that low content (<3wt%.) of CNT loading in several polymers (PP,PS,EVA etc) shows enhanced flame retarding properties. There are two different types of CNTs, Single walled nanotubes (SWNTs) and multi walled nanotubes (MWNTs). SWNTs have small diameter(1-2nm) as compared to MWNts (10-100nm). Studies of polymers with CNTs shows that it helps for increasing time to ignition (TTI), and peak heat release. Different factors affect the flame retardant properties of polymeric material nanocomposites: nanotube dispersion, nanotube loading rate, mean size of nanotubes and large aspect ratio. Large aspect ratio can be lead to the formation of efficient and compact layer.[36]

Guo et al. [37] investigated the effect of nano clay on flame retardancy of extruded metallocene polyethylene-wood fibre nanocomposites. By incorporating 5 wt% nano clay into the wood fibre composites, it was found that char was formed during burning and hence retarding the flame and preventing the fire from spreading. The burning rate was lower than that for the composites without nano clay

Deka and Maji [38] prepared wood polymer nanocomposites by using polymer blend (consisted of HDPE, low-density polyethylene (LDPE), PP and PVC) and wood flour with the co-incorporation of nanoclay and titanium oxide (TiO2) nanopowder via solution blending method. Poly (ethylene-co-glyciidyl methacrylate) (PE-coGMA) was used as the compatibilizer. The disappearance of nanoclay characteristic peak indicated the formation of exfoliated structures in the nanocomposites. It was noticed that both the mechanical and thermal properties of the nanocomposites showed improvement after the addition of compatibilizer and wood fibre into the polymer blend.

Subasinghe et al. [39] reported the synergistic effect of halloysite nanotubes (HNTs) and MMT nano clay on flame and mechanical properties in an intumescent flame retardant PP-kenaf nanocomposites.

FIRE RETARDANTS IN BAMBOO

As a lignocellulosic-construction material fire safety is well explored for bamboo with different techniques and chemicals. Some of the chemicals Boric-Borax treatment are well known and popular.[40]

Yu et al. [40] Studied Boric acid/Borax treatment of bamboo for its fire retardant improvement. Boric acid, Borax, mixture of Boric acid and borax are loaded in the bamboo fiber. Bamboo filaments immersed in 100°C for 2h in different solutions separately. Compared to untreated samples, the effect of ingredients in the boric acid and/or borax formulas were quite different during the combustion process. Borax displayed better performance for restraining the heat release rate than boric acid, while for the total amount of heat release, the result was vice versa. The excellent synergistic effect could be achieved by the mixture of boric and borax with the reasonable proportion (Boric Acid : Borax = 1:1)

Flame retardant properties of Laminated bamboo lumber (LML) using Phosphorus-nitrogen-boron flame retardants are studied by Jin et al. [41]. LBL treated with Monoammonium phosphate (MAP) – Boric acid/borax compounds (SBX) possessed a high fire resistance, improved total release by 55.0%. Monoammonium phosphate and boride compounds exhibited a good effect of catalytic charring.

Studies shows that thermally modified Bamboo Laminate can give Fire retardancy [42]. Samples were treated at 140oC for 3hr in this research. heat treated bamboo strips impregnated with phenolic formaldehyde adhesive, and pressed using hot press. Both radial and tangential layered structured are made. Heat treatment improved HRR, THR properties of laminated bamboo.

Lin et.al [43] used the bamboo slices, which treated with melamine and phytic acid using layer-by-layer (LBL) assembly technology to improve their flame retardancy properties. The morphology and chemical composition of untreated and treated bamboo slices were measured by scanning electron microscopy equipped with energy dispersive X-ray analysis and Fourier transform infrared spectra. The results showed that two-dimensional melamine–phytate (MP) nanoflakes were successfully formed and deposited on the bamboo surface. The deposition of the MP coating caused the earlier degradation of the bamboo to form char, according to thermogravimetric analysis. The peak heat release rates of the treated bamboo slices were reduced by more than 28% compared to those of the untreated ones. The MP coating promoted the formation of thermally stable char, which was responsible for the significant improvement in f lame retardancy. Besides, the char layer with excellent thermal resistance performed a vital role in suppressing flame spread.

Novel Fire retardants

Apart from commonly using and traditional fire retardants, some new materials emergered for fire retardant action. Like halogenated fire retardant were replaced by phosphorous, metal oxides, nano clays etc, there is new category of fire retardants called, Natural Fire retardant additives.[9]

Chitosan: Chitosan is a natural and abundantly available renewable biodegradable polymer obtained from the fully or partially deacetylated form of chitin, which is extracted from exoskeleton of living organisms such as fungi (cell walls), crustaceans (shrimps and crabs) and cuticles of insects; moreover, it is a non-toxic, highly biocompatible organic polymer. Chitosan chemically is an amino polysaccharide with multi hydroxyl groups and potential to be used in carbonization. Hence, chitosan has the capacity to promote the formation of char during combustion. Due to this unique property of chitosan, it is being used as a natural FR additive to polymer matrix and intumescent flame retardant in polymer composites. In combustion chitosan, can produce char layer on the surface of the polymer materials which acts as heat insulator or thermal shield to suppress the fire propagation. Florez et al. [44], Hu et al. [45] studied effect of chitosan as fire retardant in various materials.

Egg shell: On the application of heat calcium carbonate present in the egg shell decomposes into CaO and CO2, carbon dioxide does not support combustion because it is good fire extinguisher The more quantity of egg shell additive is incorporated into the polymer matrix, the more quantity of CaCO3 in the composite and less tendency of the composite to burn. The considerable amount of availability with unique composition and structure makes egg shell a potential source and it is efficiently useful as bio-filler reinforcement material as well as FR additive for polymer nanocomposites. Prabhakar et al. [46] studied effect of egg shell as a fire retardant. Another study is from Igwe et al. [47] about egg shell in polypropylene.

Lignin: Lignin is from plant constituent around 20 - 30 by wt%, chemically polyphenolic and structurally amorphous. Its degradation temperature is around 450ºC in nitrogen environment and leave stable dense crosslinked char, increases with increasing temperature up to 900º C. lignin can also be incorporated in polymeric materials to improve the thermal properties because its cross-linked structure with phenolic groups is capable of generating the high char yield after decomposition. Moreover, improvements in thermal properties and flame retardant properties of lignin based composites can be strengthened by chemically modifying lignin with phosphorus and nitrogen elements. Liu et.al [48] studied effect of lignin based Flame retardant in polypropylene and wood composite.

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Pradeep K Kushwaha: Conceptualization, Resources, Supervision, Writing – review & Editing.

Both authors have read and approved the final manuscript and agree to be accountable for all aspects of the work.

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The authors declare no financial or non-financial competing interests that are directly or indirectly this related.

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