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FIRE RETARDANT ADDITIVES OF POLYMER BASED COMPOSITES

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Abstract:- Fire hazard is now one of the vital issues in different sectors such as construction, industry, home utensils automobiles etc. The property of flame retadancy of the composites of the polymeric materials can be increased either by modification of the matrix and reinforcing phases or by offering a protective barrier by coating the composite material or by addition of suitable flame retardant additive materials. The flame retardant additive materials may be micro or nano scale sized particles, which includes inorganic hydroxide, metal hydroxides, halogen, nitrogen, phosphorus, silicon, boron based materials, clay-layered silicates and some carbon based materials. In this work we discuss about the types of fire retardant materials added to polymer composites to improve the property of fire retadancy, its effectiveness, development and application. The fire retardant materials are a blessing to our society, which prevents the dangerous fire hazards and can be effectively used as construction and automobile materials.

Keywords: *Biopolymers, Fire retradency, Nano composite. Biomolecule, biopolymer*

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I INTRODUCTION

The flame retardant additives may be defined as any substances, which on addition to a polymeric material or its composite delayed or suppresses the process of combustion and also retards the rate of the flame-spread. The flame retardant actually is not a defined term, but, however the flame retardant materials may be defined as the chemicals, which when addition, reduces the flame spread or delayed the ignition. Hence, according to the definition the addition of flame retardant materials retards the rate of combustion of the polymeric materials or their composites [1]. The flame retardant materials are any kind of additive substances, which on application impacts an enhanced property of fire retadancy performance. The additives of fire retardant may be used as coating over the surface of the polymeric materials or on uniform mixing imparts the property of flame retadancy. Although halogen based materials are used as highly effective as fire retardant additives, but on the basis of environmental point of view, the addition of these kinds of materials is now restricted and researchers are searching for new eco-friendly materials as fire retardant additives for polymer based materials [2]. Among the fire retardant materials, phosphorus, nitrogen, silicone based materials are well-known candidate materials used as additive materials to develop the property of fire retadancy for polymer based composite materials. The phosphorus−nitrogen and organophosphorus are found to beamazing fire retardant materials and produces less smoke and toxic moiety during the process of combustion. The mode of action of fire retadancy is acting either in the gaseous phase or condensed phase or in both the phases by a chemical or physical mechanism. The phosphorus based flame retardant functioning the mechanism of gaseous phase through the active radicals, which includes $PO^{\bullet}$, $HPO^{\bullet}$ and $PO_2^{\bullet}$.These radicals can able to scavenge $H^{\bullet}$, $OH^{\bullet}$ and increases the efficiency of the flame redadancy. In the condensed phase the formation of residual char serves as barriers to protect heat and the flammable O_2 gas. The N-P based materials are also exhibits excellent stability towards heat and generate less smoke as compared to the phosphorus-based additive materials. But, for development of a complete eco-friendly and sustainable fire

retardant materials more research is needed [3, 4]. Figure-1 represents different types of flame retardants

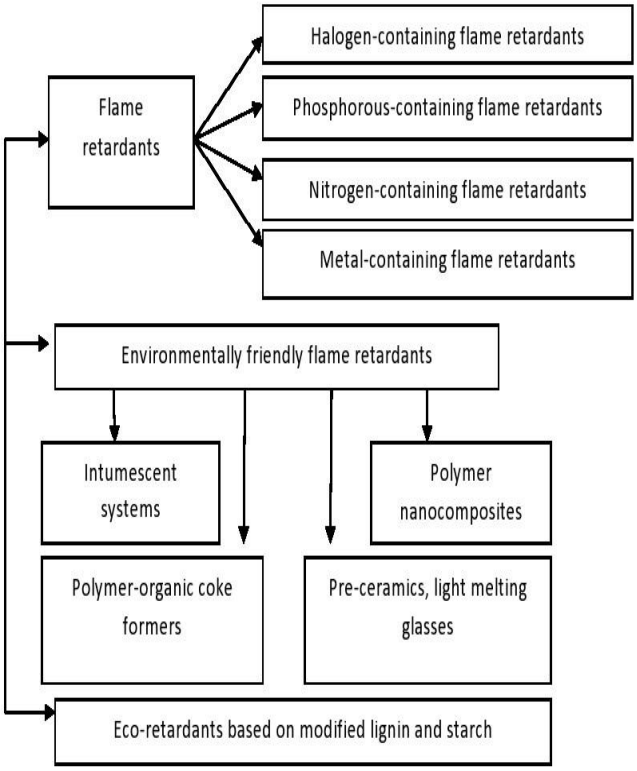

Fig.1-Types of flame retardants

Phosphorus based flame retardant additives

Phosphorus

The phosphorous based substances are used as suitable fire retardant materials either by the chemical reaction with the parent polymer chain or blending with the components of the polyurethane. It was found that red phosphorus is highly effective as fire retardant materials for polymer composites [5] In addition to red phosphorus, other phosphorus based flame retardant materials include phosphates, phosphonitrides, phosphites, phosphoric acid, phosphonates, phosphonic acid and some phosphorous compounds containing halogens are also highly effective. The mechanism of increase in the flame retardant property by addition of phosphorus based materials is similar with other polymers. The phosphorus based flame retardant materials are highly influenced the reactions carried out in the condensed phase. The mechanism of phosphorus as fire retardant materials basically occurs in the condensed phase and explained in three different stages.

- At first phosphorus converted into anhydrides of H_3PO_4 and other associated acids through the method of thermal decomposition, which functions as dehydrating agents and extract H_2O from the pyrolyzing the polymeric material with stimulating the formation of char. The formation of char is the cause of lowering transfer of heat from the flame into the condensed phase and interferes with the decomposition and heating process. [6]

- In the second stage the H_3PO_4 and other associated acids also function as a heat sink because, these acids decreases the formation of CO_2 by oxidation of oxygen and carbon leading to the inhabitation of the process of heating.

- In the third stage the acids can produce a thin liquid or a glassy protective coating within the condensed phase and cause of decreasing the diffusion of oxygen, mass and heat transfer between the condensed and gas phases. Figure-2 shows some phosphorus based flame retardant materials.

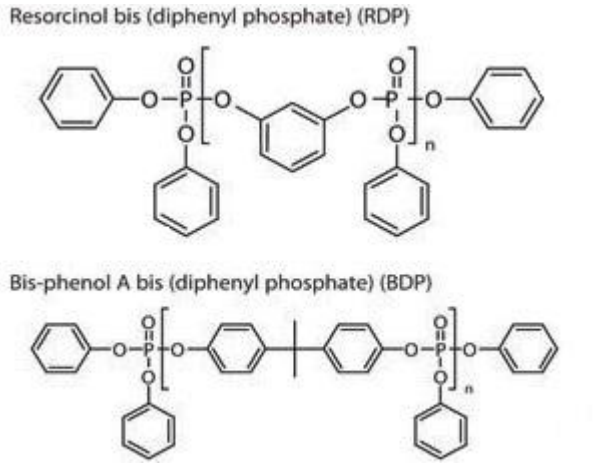

Fig. 2 Phosphorous based flame retardants

The barrier inhabits the process of oxidation of carbon to CO leading to the retardation of the exothermic enthalpy of combustion. The phosphorus based fire retardant materials are highly effective in the gaseous phase, because in gaseous phase the phosphorus compounds can be easily broken down into small species and effectively identified in gaseous phase. [7] The small species formed catalyses the formation of hydrogen molecule from hydrogen atom and decreases the energy of the flame spreading. Red phosphorus functions as good fire retardant additives for polymer based composite materials, because it does not melt during exposure to fire; rather it forms a highly protective crust without alternation of the mechanical properties. The major benefits of using red phosphorus as fire retardant additives for polymeric materials are the production of highly hazardous phosphine on reaction with H_2O . For minimization of excessive quantities of phosphine, some specific metal oxide is added as a stabilizer or stabilizing agent . It was found that Cu_2O, CdO, ZnO can effectively converts phosphine into H_3PO_4. [8] Again the effectiveness of red phosphorus can also be enhanced, if it is distributed in d-caprolactam before the initiation of

polymerization reaction. The addition of red phosphorus decreases the molecular weight of the polymer with the simultaneous increase in the efficiency of fire retardant of the phosphorus to optimum level. The presence of phosphorus exhibits the synergistic action in combination with halogen based substances and enhances the property of fire retradency of Polymers and optimum result is obtained, if the molar ratio of halogen and phosphorus is in the ratio of 1:1. The thermal decomposition of red phosphorus produces P_4, which on reaction with HCL and O_2 forms PCl_3, which is a popular flame inhibitor. Some synergistic effect also been observed due the presence of some metal oxides. The presence of MgO is the cause of production of higher quantity of H_3PO_4, which is the cause of enhancing the rate of charring on combustion with the surface of the polymer. V_2O_5 also effective similar to red phosphorus, which promotes the oxidation process of phosphorus and forms $V_3(PO_4)_5$, and act as catalyst for char formation at the polymer surface. Besides V_2O_5, other metal oxides like MoO_3 and WO_3 can also be used as co-additives in phosphorus, which is less advantageous. [9] Phosphorus normally exhibits the property of fire retradency only in case of polymeric materials containing oxygen. On the action of heat, phosphorus converted into white phosphorus (volatile), which gradually diffuses from the surface of polymeric material towards the combustion surface and oxidized into the derivative of H_3PO_4. Hence, at the surface of the polymeric material H_3PO_4 functions as a good char-forming agent and restricts the access of oxygen and volatilization of fuel. But, still the mechanism of interlinking between the polymer composite and red phosphorus is not still completely clear.

Phosphorus based organic materials

The organophosphorous is found to be a very good fire retardant additive for polymer composites. The organophosphorous based polyols are observed to be more suitable as compared to the nonreactive additives. The foams synthesized from such kind of polyols are capable of retaining the property of fire retardancy even after aging. The trivalent phosphorus including phosphines, phosphites, phosphonites and phosphinites shows less thermooxidative stability, hence, considered as an excellent fire-retardant additive applied for polymer composite. [10] The phosphonates and phosphine oxides exhibits less loss of weight as compared to other fire retardant materials due to the creation of a layer of polyphosphate in char, which has capacity of delivering higher resistance towards the volatility and diffusion of the fuel, which leads to the mechanical stability of synthesized char. The chlorinated aliphatic phosphites are more effective among the phosphate polyols. The major benefits of phosphate polyols are that hydrolysis of these compounds are also be carried out even in the presence of a little quantity of water. The dimethylphosphite and hydroxyethyl phosphate allowed to react with the trimethylol propane or pentaerythritol to get phosphate polyols, which contains more than 20% phosphorus. Phosphites instead of phosphine oxides are the mostly organophosphorous materials resistance to of heat and oxidation. But, however, due to their comparatively high cost the application of these as flame retardant materials for polymer is normally avoided. Most of the phosphites applied in the polymer are finally transformed into phosphonates. [11] The dimethylmethyl phosphonate (DMMP) and aminomethyl phosphonate have been effectively used as additive material for the fire retardant to the polymer composites. It was found that 25% phosphorus present in the DMMP, where 8% is needed for the formulations of rigid foam. The material triethyl phosphates and diethylethyl phosphonates are also efficiently used as fire retardant additives for polymer based materials. Similarly, the substances like diethylpropane phosphonate and dimethylpropyl phosphonate are free from halogens and used successfully as fire retardant additives for urethane foams. The substances like trixylene phosphate, triphenyl phosphate, isopropylphenyldiphenyl phosphate and tricresyl phosphate may be used as fire retardant material in some cases of formulations of rigid foam. About 15% phosphonate was needed as fire retardant material to incorporate 1.5–2.0% of P into the finished foam. [12] The phosphorus–sulphur additives including P4S10, P4S3 and P4S7 can be utilized for fire retardant material. It was found that approximately 6% Sb, 2%P, 13% Cl and 10% Br are required to made the composite materials having non-combustible in nature. The tri-isocyanates and di-isocyanates mainly phosphoryl tri-isocyanate are normally utilized to incorporate phosphorus inside the Polymer matrix. The phosphorus based fire retardant materials of the polymer composite are highly retarded, because of the halogens associated in it and shows synergistic effect with phosphorus. Normally a concentration of 1.5% of phosphorus is necessary for production of an effective fire-retardant without halogens. The requirement of phosphorus may be reduced up to 0.5 % by the addition of 4–7% bromine and 1.0% by addition of 10–15% chlorine. [13] Hence the synergistic systems of phosphorus–bromine are found to be more effective as compared to the systems of phosphorus–chlorine although both are effective for noticeable reduction if can be used on association with Sb_2O_3.

Phosphonitrides

It is an iminophosphazene having extremely cross-linked structure and synthesized by prolonged action of heat on aminophosphazene and eliminates NH_3. The product of this kind of substances have the general chemical formula $(PN_2H)_m$ and also termed as phosphasm. Since, this kind of substances are highly thermally stable, therefore on incorporation into the

polymers enhances the property of flame retardancy. The efficiency or effectiveness of phosphasm as fire retardant additives are almost comparable to red phosphorus. Both phosphasm and red phosphorus exhibits the property of raise in LOI from 16.5 to 23.0 in the presence of 1.0% phosphorus, but relative to red phosphorus, phosphasm is found to be beneficial because of its free from release of phosphine and light colour. [14] Again, the efficiency of the property of flame retardant can be enhanced, if it can combine with zinc borate and phenol–formaldehyde resin. The Polymer composites may become fire retardant material if the normal polyols from it is replaced by reactive phosphonitride or substituted polyol including as tetrakis (hydroxymethyl) phosphonium chloride, which serves as a pre-reactor in addition to polyol. Phosphorous oxynitride is a material containing both nitrogen and phosphorus and is also significantly effective as a material for increase in the property of flame retadancy.

Inorganic phosphates

The mon, di and tri $(NH_4)_3PO_4$ are soluble in water and never dissolve in the components of urethane, whereas the polymer composites comprising these phosphates are stable in humid aging and possesses better fire retardancy. The total involvement in running water, tri-ammonium, di-ammonium and mono-ammonium phosphates may be derived from the composite maetrials and the flame produced from it almost same. The solubility in water was drastically reduced by using ammonium polyphosphate (APP) of high molecular weight. [15] The polymer having APP are shows good self-extinguishing property after 15days of dipping in the running water. APP provides a solid-phase mechanism, which produces less smoke than any halogen-based fire retardant additive substances. The APP produced with melamine cyanurate shows an excellent retardation of rate of heat release and loss of weight both in rigid and flexible polymeric materials. The urethane foams on treatment with organophosphorous (liquid) fire retardant additives and solid APP as fire retardants loses 15 and 10% from their original weight respectively on testing with vertical bar flammability test. APP also be used as coating material for preventing fire due to its ability to act as catalyst in char forming the organic materials and generate a intumescent protective char. [16] The addition of 5% APP and 25% pyromellitic anhydride in polymer composite produces a new material, which on combustion generates 28% char along with moderate amount of smoke. These are some inorganic materials including alumina trihydrate (ATH), $ZnCO_3$, MnO_2, $MnCO_3$, $CaCO_3$, As_2O_5, SbO_3 $CaSO_4$ and $(NH_4)_2CO_3$ also effectively used as flame retardant coadditives along with APP.

Halogen based flame retardant additives

Although a number of variety kinds of halogen based product materials are identified as flame retardant additives for polymeric compounds, but the efficiency of materials containing halogens are in the order of I >Br>Cl>F. The iodine and fluorine based materials are practically being not suitable because fluorine forms a strong interaction or bond with carbon, whereas iodine forms a very weak bond because of little interaction with carbon. From the two remaining halogen, bromine is comparatively more suitable than chlorine owing to its weaker interaction with carbon, which makes it more effective in interfering in the process of combustion. [17] Bromine and chlorine can have used individually or in combination with some metal salts, metal oxides of synergistic nature, phosphorus-based substances and high-char forming agents used effectively as fire retardant materials. There are three potential mechanisms are suggested for halogen based substances as flame retardant materials for polymer composites as follows.

- Production of free-radical chain terminating agents possessing less energy.
- The formation of char via dehydrogenation reactions
- The synthesis of hydrogen halide (HX) as a protective layer, which is non-combustible and serves as a barrier between the condensed phases and fuel gas

Normally the halogen based fire retardant additives are found to be less effective as compared to the phosphorus based additives [18]. The major drawback of the halogen based fire retardant additives is, these are highly susceptible towards hydrolysis, therefore the foam produced is completely excluded with water. Another demerit of it is their capability of forming corrosive combustion substances.

Chlorinated substances

The chlorinated cycloaliphatics and chlorinated hydrocarbons are the two major fire-retardant materials containing chlorine used effectively in commercial and industrial sectors. The major benefits of using chlorine based compounds as flame retardant materials is that, it influences adversely the foam generating characteristics and offers maximum extent of the fire retardancy. The cycloaliphatic chlorine based substances are normally stable up to temperature of 260 ^{0}C and widely used as an effective fire retardant material. The compound hexachloroendomethylene tetrahydrophthalic acid is the chlorine based material, which are highly used as flame retardant for polymers. It is normally the reaction between tetrachlorophthalic acid or hexachlorocyclopentadiene with maleic anhydride having 35%

chlorine, which is produced due to Diels–Alder reaction. The other different chlorinated materials associated with polymer composte are epichlorohydrin interlinked with hexachlorometaxylene, pentachlorophenol and chlorinated form of poly-isobutylene. Again, the property of fire retardant of the material can be developed by adopting suitable synergist. The proper selection of synergist normally prepared from SbO_3, ZnO, Fe_2O_3 and $Zn_3B_2O_6$. The halogen and antimony in the molar ratio of 3:1 was found to exhibit optimum synergistic performance. Approximately about 20% chlorine is needed to develop non-combustible polymeric material, if Sb_4O_6 is added in appropriate amount and finally it was found that 3.8% chlorine and 4.4% Sb_4O_6 are suitable for fire retardant foam. Again, the addition of 5.9% Sb_4O_6 decreases the necessity of chlorine to 2.4% in the compound chlorinated polyether polyol to develop PUF having same performance of fire retardant properties. [19] The organic compound 3,30- dichloro-4,40-diamino diphenylmethane on addition with polymeric material exhibits excellent property of fire retardancy and that materials are highly suitable for manufacturing of motor parts.

Brominated substances

Since, the lower concertation of bromine is needed in comparison to chlorine based materials, therefore bromine based materials are treated as superior than chlorine based material on the basis of property of fire retardant. Again, the mechanical and physical properties of bromine based fire retardant materials are less influencing than the chlorine based materials. The bromine based fire retardant materials can be easily incorporated as both additive and reactive fire retardants. The two major classes of bromine based fire retardant materials are cycloaliphatics and aliphatic compounds. [20] The cycloaliphatic bromine based materials shows higher fire retardant materials in comparison to aliphatic bromine based materials, because the materials offer a better impact of fire retardancy to polymeric materials. The Polymer, if added with the nonreactive bromine based additives, 8–10% bromine based product materials synthesizes self-extinguishing foams, but 12–14% bromine produces non-burning foams. Again, it was found that aliphatic bromine based materials are found to be more beneficial than the aromatic bromine based materials. Pentabrominated diphenyl ethers can be more effectively utilized as a suitable fire retardant material for various polymeric materials and their composites. [21]

Nitrogen-based materials as additives

The major nitrogen based materials, which are effectively used as additive to enhance the property of fire retardant, are melamine, urea, ammonium biborate, dicyandiamide and ammonium pentaborate. The mechanism of the property of fire retardant of melamine is generally explained in three steps. Melamine undergoes sublimation at a temperature of 25^0C, therefore the insertion of melamine into polymeric materials liberates some gaseous components. The concentration of 30 wt. % of melamine is enough to form self-extinguishing polymeric materials. The addition of melamine not only increases the property of flame retardancy, but also reduces the quantity of smoke formation. If melamine used as fire retardant material in liquid form, such as tris(1,3-dichloroisopropyl) phosphate and tris(2-chloropropyl) phosphate, then it shows excellent property of fire retardancy. The efficiency of fire retardancy of the melamine enhances, if it is added in combination with halogen-phosphorus based additives. [22] The use of 3% concentration of halogen- phosphorus additives exhibits equivalent effect on the performance of fire retardancy of polymeric materials as the use of approximately 20% melamine. Urea exhibits better fire retadancy property as compared to melamine in case of polymer composites. Dicyandiamide and urea can be used individually or on combination with melamine as fire retardant additives.

Silicon-based materials as additives

Silicones are normally noncorrosive in nature and do not contain any halogen atom in it, on combustion it generates less quantity of smoke. The O-Si bond treated as the backbone almost equivalent to material resistance to elevated temperature like quartz, sand and glass. The strong bond Si-O is the cause of impacting the stability of the material against elevated temperature and the characteristics of the material is being almost unchanged even in a range of temperature. [23] The addition of siloxane substances into the polymeric maetrials offers the increase in the properties of fire retadancy and thermal stability. The silicone based materials added the property of fire retadancy in two methods

- As a fire-retardant additive
- Insertion into the backbone of the polymeric materials

The silicate-clay nanocomposites reduce the peak rate of heat release in the cone calorimeter experiment. The most popular and widely used silicones as flame retardant materials are polydimethylsiloxane. The LOI value of PUFs is 20.8, if siloxane present in it is off about 7.5% and again its value is further raised into 29.8 if the concentration of siloxane is increased to 50%. [24] Th Si-Br based substances shows the properties of higher fire retadancy as compared to the Si-P combination. Silicones on incorporation with the platinum, magnesium carbonate, fumed silica, zinc stearate, quartz and magnesium stearate are highly effective for enhancing the property of flame retradency. The dibromoneophenyl diol, silicone along with the polymer exhibits the LOI value of 25.7 and it again decreased to 21.0, if the

combination changed into siloxane–phosphorated diol. The silicone based materials functions as a fire retardant additive due to the formation of more amount of char residue. The silicon based materials is the cause of increase in the pyrolytic char and resistance in the oxidation of char.

CONCLUSION

Since the use of halogen based compounds exhibits highly toxic impact to our ecosystem, therefore its use is banned in various sectors, but the chlorinated and brominated substances are serves as highly effective additive materials to enhance the property of fire retadancy of the polymeric materials. The nitrogen based flame retardant additives such as metal phosphinates and melamine polyphosphate shows good fire retardant characteristics for polymer based composite materials. The phosphorous based additive materials is now identified as a good fire retardant materials with comparatively lower rate of loading in spite of the mode of combination. The phosphorus based flame retardant materials normally functioning in both condensed and gaseous phase. Some silicon and clay based materials also be effectively used as fire retardant additives. Now scientist are searching for a new eco-friendly and sustainable flame retardant additives to prevent the risk of fire hazard and minimization of environmental pollution.

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