Investigation on development of fire resistant high performance polymeric nano composite film

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Abstract
This investigation emphasizes on developing polymeric films with high performance thermoplastic polymer that can improve flame retardancy. The study focuses on Polyetherimide (PEI) that has relative advantages over other engineering materials because of their low weight, corrosion resistance, design flexibility and improved fire-resistant properties. A comparative study was carried out on characteristics of films developed with PEI on reinforcing different fire-retardant fillers such as Ag-CNF, Magnesium hydroxide, Borax, Red phosphorous. Films of PEI, PEI with other fillers were fabricated by solution casting process with Dimethylacetamide (DMAc) as a solvent. Characterization techniques were employed to know the thermal, mechanical and fire-retardant properties. It was evident that there was an increase in LOI values for samples made with reinforcing Mg(OH)2 as filler on comparison with plain PEI films. Thermogravimetric (TGA) characterization studies revealed that there is minimal increase in thermal stability for reinforced PEI films compared to plain PEI films. It is confirmed from the results of Differential Scanning Calorimetry test that the glass transition temperatures were high for films of PEI- Mg(OH)2 films, which in turn is an indication for thermal stability. Fourier transform infrared spectroscopy was performed to know about the behavior of the functional groups.

1. Introduction
The aerospace industry ranks among the world’s largest manufacturing industries in terms of people employed and value of output. Since the birth of aviation, designers have continuously struggled to improve the lift to weight ratios of aircrafts. In the early stages aluminum was the most preferred material in the aerospace sector because of its lightweight and strength. Aluminum wasn’t suited for the skin surfaces as their property retention at high temperatures is low. Steel was also tried as a potential replacement for aluminum but it was observed that it was three times heavier even though it had a higher strength and higher stiffness. Titanium and super alloys were also employed. In comparison to properties of composites, its substitute still has been evolved. This innovative idea of introducing composites in this industry enabled the designers to overcome the hurdles.

Composites are typically materials that are made up of two or more phases designed to display the combination of best characteristics of the components used. The introduction of composites made a breakthrough in structural applications because of their reduced weight. This further enhances the fuel efficiency of the aircraft when developed with composites. Composites also exhibit properties like high specific strength, high thermal stability, high impact resistance and high corrosion resistance. Hence these properties make composites a dominant material for aerospace industry.

Composites are mainly classified into metal matrix composites, polymeric matrix composites and ceramic matrix composites. Components in the aircraft structure are made mainly from polymeric matrix composites. Fiber-reinforced polymer composite materials are fast gaining ground as preferred materials for construction of
aircrafts and space crafts [1]. The polymers used in an aircraft are mainly thermosetting polymers with the reinforcement of different fibers. At present Carbon Fiber Reinforced Epoxy composite and Glass Fiber Reinforced Epoxy composite are used in the aerospace industry. Epoxy being a thermosetting polymer has high strength to weight ratio and is easy for manufacturing. On comparison thermoplastic polymers hold higher thermal stability and are recyclable in nature than the thermoset thus improving the performance. However, there is very limited literatures are available till date on high temperature thermoplastic polymeric composite for structural applications and other related high performance applications such as fire resistivity, structural integrity, radiation resistivity etc.

Among the resins used in aerospace industry, epoxy is widely used polymer. Epoxy, a thermosetting polymer exhibits good wettability, high mechanical strength, and good electrical insulating properties. Ease of availability and manufacturing has made as a prominent resin among other polymeric resins. In spite of having all these properties, they exhibit low fire resistivity. Therefore, scope of high temperature resistant thermoplastic polymers in terms of its fire resistance open new direction of research and gradually more emphasize will be given on application of high temperature resistant thermoplastic polymers. Among these high temperature thermoplastic polymers, polyether ether ketone (PEEK), polyether imide (PEI), polysulfone (PUS) appears to be highly promising.

The necessity of fire resistant coatings has been increasing in the last few decades to forbid fire accidents avoiding casualties. Majority of the accidents are either due to engine fire, cabin fire or due to other hidden fire as stated in Mouritz (2006) [2]. When an aircraft catches fire, there is catastrophic failure within a very short duration of time. The immediate effects are smoke and fumes that debilitating the crew. Fire resistant materials are basically added or applied to the materials in order to slow or prevent the start or growth of fire. Fire-resistant fillers/fire-resistant coatings act as a barrier by reacting with the fire through distinct mechanisms thus causing minimal damage to the structure.

An attempt is made to understand polymeric composite and its fire-resistant property. Studies from Bar (2015) [3] proves that flame retardancy behavior of thermoplastic polymeric composite can be improved by providing protective fire-resistant coating or through improvising the constituents i.e. matrix and the reinforcement. Different fillers have different flame retardant mechanisms to enhance flame retardancy.

Jeencham (2010) [4] has reported his research on polypropylene composites using fillers like zinc borate, magnesium hydroxide, ammonium polyphosphate and combination of these three fillers and studied its characteristics. Halogenated monomers, resins, and additives cause problems with smoke toxicity, making them unsuitable for many naval and transportation applications [5]. Zhang (2012) [6] works focuses on reinforcing on ammonium polyphosphate and silica on wood-fiber/polypropylene composites. Nachtigall (2006) [7] stated that addition of metal hydroxides is best for flame resistance properties.

In this investigation, the high temperature thermoplastic polymer such as Polyetherimide (PEI) has been used. PEI is a high-performance engineering thermoplastic polymer whose creep resistance over long term allows it as a replacement for metals in structural applications. Various nano fillers such as carbon Nano fibers, silver coated carbon Nano fibers, Magnesium hydroxide, Borax and Red phosphorous were dispersed into the PEI matrix. The other features of PEI have enabled its usage in wide applications like medical, automotive, electronics. Its properties of good flame resistance and high thermal stability has proved it to be a polymer that can be used for fire resistant coatings, but till date minimal research work has been reported on fire retardancy of PEI.

The fillers such as carbon Nano fibers, silver coated carbon Nano fibers, Magnesium hydroxide, Borax and Red phosphorous are essentially are essentially used to increase fire resistivity of the polymer. Therefore, detail thermo-mechanical characterization has been carried out after dispersing these nano fillers into PEI matrix. It was also taken into account that they have high decomposition temperatures and high tensile strength even at elevated temperatures.

The aim of the study is to develop a high temperature fire resistant thermoplastic films which will be subjected to thermal and mechanical characterizations.

2. Experimental

2.1. Material selection

2.1.1. Polyether imide (PEI)

Polyether Imide (PEI) is an amorphous, high performance thermoplastic polymer possessing properties like flame retardancy, high mechanical strength and thermal resistance was procured from Ultem, SABIC. The density of PEI is 1.27 gm cm⁻³. The heat distortion temperature and Limiting oxygen index (LOI) is 200 °C and 47% respectively. The chemical formula for PEI, (C₃₇H₄₅O₆N₂)ₙ. The chemical structure of PEI is given in figure 1.
2.1.2. Dimethylacetamide

DMAc being colorless, water-miscible, high boiling liquid is used as a high-grade solvent in organic synthesis. PEI being a polar compound has a very good solubility in DMAc. DMAc was acquired from The Precision Scientific co.

2.1.3. Fillers

(i) Silver coated Carbon Nano Fiber: The high thermal and mechanical property offers CNF to be a potential replacement over the conventional fillers used till date. CNF were procured from Nano shell. In order to attain a homogeneous dispersion of nano fibers, to augment the interfacial bonding and to make use of the excellent properties of reinforcements in the matrix material surface, modification of CNF was done by coating silver on it by sputtering process.

(ii) Magnesium hydroxide: Mg(OH)$_2$ is a mineral filler that acts a fire retardant through heat sink mechanism i.e. heat absorption through endothermic decomposition. It decomposes to form magnesium oxide (MgO) with the release of water. It breaks down endothermically forming water vapors, diluting the radicals in the flame, while the residue of the magnesium oxide forms a protective layer. The decomposition temperature of Mg(OH)$_2$ is 300 °C–320 °C. We obtained Mg(OH)$_2$ form from The Precision Scientific co.

\[
\text{Mg(OH)}_2 \rightarrow \text{MgO} + \text{H}_2\text{O}
\]

(iii) Borax: Sodium tetra borate decahydrate (Na$_2$B$_4$O$_7$.10H$_2$O) forms an insulating layer when exposed to fire. This boron-based compound on decomposition gives sodium metaborate (Na$_2$B$_4$O$_7$) and water molecules. The sodium metaborate on further decomposition gives boric oxide, which reacts with water to form boric acid that acts an insulating layer. The melting point of sodium tetra borate decahydrate is 743 °C. We acquired borax from The Precision Scientific co.

\[
\text{Na}_2\text{B}_4\text{O}_7.10\text{H}_2\text{O} \rightarrow \text{Na}_2\text{B}_4\text{O}_7 + 10\text{H}_2\text{O} \rightarrow 2\text{NaBO}_2 + \text{B}_2\text{O}_3
\]

(iv) Red Phosphorus: Red phosphorus is a harmless substance with extremely low toxicity. On adding small amount of red phosphorus with the flammable resin, the resins burning rate decreases. The melting point of Red phosphorus is 590 °C. The flame retarding effect is based on the formation of phosphoric acid. Together with the organic polymer, a char is created which prevents the propagation of the flame. The compound was obtained form from The Precision Scientific co.

\[
\text{C}_m\text{H}_m + \text{O}_2 \rightarrow \text{CO}_2 + \text{H}_2\text{O} + \text{C}(\text{soot})
\]

\[
\text{P} + \text{O}_2 + \text{H}_2\text{O} \rightarrow (\text{HPO}_4)_n\text{(Phosphoric acid)}
\]

\[
(H\text{PO}_4)_n + C_m\text{H}_m \rightarrow \text{Char}
\]

Table 1 shows the melting and decomposition temperatures of different fillers used.

2.2. Methods

2.2.1. Surface modification by sputtering

Surface modification of carbon nanofiber was carried out through sputtering process to enhance the dispersion. Sputtering is a technique by which a thin film of a material is deposited on a given surface. Silver as the target material and the substrate as CNF were placed in a vacuum chamber. A high vacuum was applied in the chamber such that the target and the substrate are attached to the cathode and anode respectively. Argon gas was used as the sputtering gas, which was ionized for creating plasma. This ionized gas bombards the target (silver) and sputters of the material onto the substrate (CNF).
When examined by a Transmission electron microscope (TEM), there was predominant agglomeration of CNF in polymer as observed in figure 2. So, there was a necessity of surface modification to attain better dispersion of filler. Hence, through sputtering process silver was coated on surface on CNF, whose TEM image as showed in figure 3 showed less agglomeration which enhanced the dispersion rate. Thus, Ag-CNF was chosen to be the filler reinforced with PEI.

| COMPOUND            | Melting point (°C) | Decomposition temperature (°C) |
|---------------------|--------------------|---------------------------------|
| Polyether imide     | 346                | 520                             |
| Magnesium hydroxide | 350                | 300–320                         |
| Borax               | 743                | 1575                            |
| Red phosphorous     | 590                | 240                             |
2.2.2. Preparation of basic PEI film
A solution of 5 grams of PEI was mixed with 45 grams of DMAc and was kept for stirring on a magnetic stirrer controlled at 100 °C and 600 rpm for 10 h. The resulting yellow colored homogeneous solution was transferred to a Petri dish. The Petri dish was kept in vacuum chamber $10^{-2} - 10^{-3}$ mbar. Then the yellow transparent film was taken out from the Petri dish by immersing it in water.

2.2.3. Preparation of PEI with different fillers
The reinforcement of fillers in PEI was carried out at different weight ratios as mentioned in table 2. Initially, for each percentage the mentioned amount of PEI was mixed with DMAc in a magnetic stirrer at 600 rpm and 100 °C for 10 h. According to the weight of PEI dissolved, the different ratios of filler was reinforced in the yellow homogeneous solution in a magnetic stirrer at 600 rpm and 100 °C for 8 h. The solution was then transferred to a Petri dish and was kept in oven maintained at 100 °C for 16 h. The film was then taken out by immersing the Petri dish in water having the thickness of 0.2 mm.

3. Characterization

3.1. Differential scanning calorimeter (DSC)
DSC technique was employed to understand the shift in glass transition temperatures. The test was done for basic PEI film and PEI film with different reinforcements by DSC Q20 V24.10. 5 g of the sample was taken in a DSC holder and was heated at a rate of 10 °C min$^{-1}$ from 0 °C to 400 °C.

3.2. Thermo gravimetric analysis (TGA)
TGA characterization technique was done for basic PEI film and PEI film with reinforcements by TGA Q600 V20.9. The purpose was to study about the decomposition temperature of the solvent, polymer and the filler in a composite. 5 g of the sample was taken in a TGA holder and heated at a rate of 10 °C min$^{-1}$ from room temperature to 1000 °C.

3.3. Fourier transform infrared spectroscopy (FTIR)
In order to understand the chemical characteristics of the sample on addition of fillers i.e. to know the changes caused due to new functional groups. FTIR spectra for samples were recorded using Nicolet iS10 spectrometer with transmission spectra in the range of 4000–4000 cm$^{-1}$ and with a resolution of 4 cm$^{-1}$.

3.4. Tensile test
UTM machine of standard ASTM D-638 was used to know about the tensile strength of the samples. The film was loaded at the rate of 50 N min$^{-1}$.

3.5. Limiting oxygen index
LOI test is done to know about the percentage of minimum oxygen concentration that is required for combustion of a material. This test was done under the standard ASTM 2863.

4. Results

4.1. Differential scanning calorimeter
DSC is a thermo analytical technique in which difference in the heat flow is measured as a function of temperature that helps in determining the glass transition temperature. This test was carried out on basic PEI film and on PEI with reinforcements of CNF, Ag-CNF, borax, magnesium hydroxide and red phosphorus. The data was collected from the DSC system and graphs were plotted using excel. The graphs were consolidated into a single graph for comparison of basic PEI film with other reinforcements and are plotted in figure 4.

From the obtained results it was evident that there was a prominent change in the glass transition temperature (Tg) with the addition of different fillers. Tg is the temperature at which the amorphous material
changes from a hard and relatively brittle glassy state into a rubbery state, after which the material loses its stability. Glass transition temperature of basic PEI is 178.6 °C, however, when silver coated CNF is dispersed in PEI, glass transition temperature decreases to 126.4 °C. Glass transition temperature increases significantly when magnesium hydroxide (Mg(OH)₂) filler is dispersed in PEI as evident from figure 5. In this case the recorded glass transition temperature is 218 °C. In a particular DSC graph, the value of Tg is interpreted from the first shift from the obtained plot and figure 5 depicts the Tg of the samples.

The Tg value of Mg(OH)₂ is 218 °C, which is higher than the one without any filler reinforcements. It is concluded that the Tg value for Mg(OH)₂ is greater in comparison with other fire-retardant fillers used. Other fillers show a reasonable Tg but they tend to loss its property at an earlier temperature. Magnesium hydroxide possesses high decomposition temperature which proves that when used as reinforcement imparts good dimensional stability even at elevated temperatures.

4.2. Thermogravimetric analysis (TGA)
This thermal analysis is performed to know about the materials thermal stability by determining its decomposition temperature at various phases. In this technique the mass of substance given for test is monitored as a function of temperature. The samples weight is measured until a temperature of 1000 °C.

From the weight loss, the thermal stability of the material can be inferred. Figure 6 demonstrates TGA plots of PEI, Rphos dispersed PEI, MgO dispersed PEI, Borax dispersed PEI and AgCNF dispersed PEI. It is observed that there are three transitions in the graph that indicates the decomposition of solvent, polymer and filler. Once the decomposition temperature of the polymer reaches, there is considerable decrease in weight loss of the polymer. In table 3 the weight of the sample given and the weight loss are stated.
The onset of decomposition of PEI without reinforcement is at a faster rate compared to that of PEI with reinforcements. The TGA plot reveals that Mg(OH)$_2$ results weight loss at 215 °C which is due to endothermic release of water, as stated in heat sink mechanism. PEI-AgCNF thermally decomposes at around 201 °C which is 6.9% less than magnesium hydroxide. Though borax has ten molecules of water and its mechanism of boric oxide as an insulating layer, it isn’t able to withstand its properties at higher temperature on comparison with magnesium hydroxide. Among all the fillers the maximum weight loss at high temperature is faced by plain PEI. So, it can be concluded that the material with minimum weight loss at higher temperatures i.e. the material with highest thermal stability is PEI reinforced with Magnesium hydroxide. It maintains its stability till 532 °C with minimum weight loss.

4.3. Fourier transform infrared spectroscopy
FTIR Spectrum reports the considerable information regarding presence of new functional groups on the surface of the material. Figure 7 shows the FTIR graph of plain PEI, PEI-AgCNF, PEI-Mg(OH)$_2$, PEI-Borax, PEI-Red Phosphorous plotted in origin software in a single graph for comparison. There are no significant changes observed in the FTIR plot because the amount of filler present is too low on comparison with the base material, PEI. As there are no changes found in the FTIR plot it can be summed up that there are no new functional groups formed.

4.4. Tensile test
Tensile strength of basic PEI film and with the reinforcement of different fillers such as Ag-CNF, magnesium hydroxide, borax and red is carried out and the results are presented in bar chart image in figure 8. The filler reinforcement concentration of 5% was included in each the sample made.

The tensile strength of basic PEI film developed was found to be 45 MPa. When Ag-CNF is dispersed in PEI, tensile strength reaches up to 53 MPa. This is because Ag-CNF increases load bearing capacity of the PEI films with local strengthening and resulting in higher tensile strength. Similarly when borax, Mg(OH)$_2$ and red phosphorous are dispersed in PEI, tensile strength increases to 48 MPa, 51 MPa and 49 MPa respectively.

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**Table 3. Weight loss of films from TGA.**

| Material         | Weight given (grams) | Weight lost (grams) | Percentage loss (%) |
|------------------|----------------------|---------------------|---------------------|
| PEI              | 1.43                 | 0.848               | 59.32               |
| PEI-AgCNF        | 1.65                 | 0.948               | 57.47               |
| PEI-Mg(OH)$_2$   | 1.24                 | 0.68                | 54.9                |
| PEI-Borax        | 3.103                | 1.7345              | 55.9                |
| PEI- Red Phosphorus | 2.074            | 1.124               | 54.2                |
4.5. Limiting oxygen index

Limiting Oxygen Index test was carried out for basic PEI film and PEI reinforced films and the results are shown in bar graph image in figure 9. Examining the results, it is concluded that reinforcement of different fillers escalated the LOI values to a commendable extent than the plain PEI film. With the reinforcement of Mg(OH)$_2$ it was observed that LOI value increased 55.5% of that of basic PEI film. This is because of the heat sink mechanism followed by magnesium hydroxide which had a higher decomposition temperature evident from the DSC results.

Figure 7. FTIR Spectroscopic images of the film.

Figure 8. Tensile strength of the films.

Figure 9. Limiting oxygen index of the films.
5. Discussion

Objective of this investigation is to increase fire resistance properties on PEI nano composite films. Therefore, selection of appropriate nano fillers was highly desirable. The flame-retardant characteristics of films made by reinforcing fillers were better than the characteristics exhibited by the plain PEI film. The filler’s mechanism to extinguish the fire is an important factor. In this research, four different fire-retardant fillers were reinforced with polymer to know the best outcome possible.

From the studies in LeVan (1984) [8] knowledge of different fire-retardant mechanisms followed by different materials were acquired. The paper also discusses about the test methods used for determining fire retardancy, the various formulations used to make wood fire retardant, and the research needs in the field of fire retardancy.

The investigations from Mohan (2017) [9, 10] emphasized on materials for aerospace applications with polyetherimide, high performance thermoplastic polymer that has the capacity to retain its thermal and mechanical properties. With the reinforcement of nano fillers PEI showed a substantial decrease in structural weight yet having better thermo-mechanical properties than basic PEI. This is because individual fillers are essentially more stronger than polymer matrix and therefore, due to reinforcement of fillers tensile strength of the polymer increases.

Carbon nanofibers are high-aspect ratio graphitic materials possessing unique physical properties such as high strength, low density, tunable morphology, metallic conductivity. Thus, reinforcement of nano fillers with PEI enhances the thermal properties. Klein (2008) [11] has evaluated the techniques used for modification of the surface of carbon nanofiber for enhancing their functionality to perform extensively in various fields. Thus, surface modification was done on CNF through sputtering process, by coating silver on it. The surface modified Ag-CNF showed better dispersion than CNF, so Ag-CNF was taken to be reinforcement.

Hull (2011) [12] has investigated about the endothermically decomposing mineral fillers, such as aluminum or magnesium hydroxide, magnesium carbonate, or mixed magnesium/calcium carbonates and hydroxides which are in heavy demand as sustainable, environmentally benign fire retardants. They reduce the flammable content of the material by three quantifiable fire-retardant effects: heat absorption through endothermic decomposition; increased heat capacity of the polymer residue; increased heat capacity of the gas phase through the presence of water or carbon dioxide. Studies of Tang (2013) [13] shows that the increase of magnesium hydroxide concentration could significantly improve the flame-retardant performance of the unsaturated polyester resin. These characteristics of mineral fillers were the main motivation for choosing magnesium hydroxide as filler.

Lili Yu (2017) [13] has evaluated the effects of different boron fire retardants on the heat release and smoke release of bamboo filaments and untreated samples. The results showed that on comparison with untreated samples, the fire retardancy of samples with boric acid or borax effectively reduced the heat and smoke release from bamboo filament. Borax displayed better performance for restraining the heat release rate than boric acid, so the form of sodium tetraborate decahydrate was preferred as the reinforcement. Savas (2017) [14] has reported that micro capsules red phosphorous exhibits better barrier effect of residue in condensed phase and formation of active radicals in gas phase. It also shows enhanced mechanical properties. From Savan’s research it can be inferred that red phosphorous is a active fire retardant filler than can suitable for various applications.

The filler to polymer ratio gave positive outcomes till a certain extent. With increase in filler ratio after 7.5% weight of PEI, the films failed [15]. So, optimization ratio of filler-polymer was considered to be 2.5–7.5. From the results of Differential Scanning Calorimeter, the highest glass transition is found to PEI reinforced with Mg(OH)2. Thermogravimetric analysis has helped to determine the decomposition temperature, which is found to be maximum for PEI-Mg(OH)2. This shows that the film is more thermally stable for PEI reinforced with magnesium hydroxide. The FTIR results clearly show that there are no functional groups formed on plain PEI and PEI with other reinforcements. Though a comparative study was done in tests such as DSC, TGA and LOI; there weren’t any appreciable changes in the tensile strength of films with reinforcement of different fillers. LOI, being a major characteristic for fire resistant was found to be exceeding for the films with reinforcement of magnesium hydroxide than the conventional LOI value of the materials used in aircrafts.

From this research it can be inferred that the flame retardancy characteristics can be significantly improved by addition of fire retardant fillers in thermoplastic polymers. From the results obtained mineral filler magnesium hydroxide exhibited higher thermal properties when reinforced with PEI than the other reinforcements. The limiting oxygen index test contributed in proving that magnesium hydroxide to be the best filler that can be used for fire resistant coating.

This investigation was at its preliminary stage however, the results obtained were proved to be satisfactory for the application as materials with fire resistivity. This can be implemented in various fire-resistant coating that is used in day to day life. Other test such as smoke emission test and cone calorimetry can be suggested to get the extended idea about other characteristics behavior of the material. Surface modification of the polymer prior to
making nano composite could enhance better interfacial interaction between the polymer and nano fillers and could result higher thermo-mechanical properties leading to more comprehensive applications applications.

6. Conclusion

• The Tg of PEI-Mg(OH)2 was found to be 22% higher than plain PEI film.
• On comparison with all other reinforcements, Mg(OH)2 reinforced PEI possess higher thermal properties.
• LOI was highest when Mg(OH)2 was dispersed into PEI matrix. The zenithal value of LOI obtained is 28%, which is for PEI-Mg(OH)2.
• There weren’t any appreciable changes observed in FTIR and Tensile strengths. Minor increase in tensile strength was observed due to reinforcement of nano fillers.
• From all the characterization techniques done, Magnesium hydroxide proves to the best reinforcement as a fire retardant compared to other fillers.
• Polyether Imide (PEI) when reinforced with Magnesium hydroxide Mg(OH)2 showed the best reinforcement in respect of its fire resistivity in compared to other fillers.

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