Effect of Ammonium Phosphate on the Thermal and Flammability Behaviour of Sisal/Epoxy Composite

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***Abstract-* The thermal and flammability behaviour of sisal/epoxy composite have been studied. Ammonium Phosphate (AP) has been used as flame retardant. Thermal Gravimetric Analysis (TGA), Limiting Oxygen Index (LOI) and Vertical Flammability test have been used to study the thermal and flammability behaviour of the samples. The composites were prepared by compression moulding where the materials were added by hand layup technique. Through TGA analysis, it was observed that the degradation of the sample occurs in two steps. The first step includes degradation of fibre as well as decomposition of fire retardant which forms char on the surface of the composite thus, protecting it from further weight loss. It was observed that the sample with 15 wt. % ammonium phosphate showed self-extinguishing properties and high flammability behaviour. This can also be proved by observing the limiting oxygen index of the samples which goes as high as 33. The results have revealed that on the addition of AP, leads to the improvement of the thermal and flammability behaviour of Sisal/Epoxy composite.**

***Keywords*: Limiting Oxygen Index, self-extinguishing, decomposition, ammonium phosphate, compression moulding, hand-layup.**

1. INTRODUCTION

Natural fibres are made from living organisms like plants and animals. Natural fibre holds several advantages over synthetic fibres. Synthetic fibres are usually made of plastic and hence when they burn severe dripping occurs whereas natural fibres are cheaper, easily available, has low density and shows fire resistance and water absorbing properties. But due to organic nature of natural fibres, the composites derived from them have poor thermal resistance (Rakotomalala et al., 2010). Flammability of a material can often limit its use, and smoke generation properties of composite materials are important factors that determine the end use of the products. The flammability of a composite is often found to be different from the flammability of the constituent materials. Fire retardant resins can improve the fire performance of natural fibre reinforced composites. However, the additives used to improve fire resistance can have a detrimental effect on the processing techniques and on mechanical performance (Fan et al., 2017). Fire resistance can be achieved with the incorporation of various flame retardant mainly metal oxides, metal hydroxides, halogen-based and phosphorous based flame retardant (Yang, 2017). Phosphorous containing inorganic flame retardants have been widely used due to their advantages such as non-volatility, non-thermal stability, long lasting effect, corrosive gas generation and low toxicity when compared to natural fibres.

Dorez et al., 2013 used ammonium polyphosphate as fire retardant on different natural fibres and bio-composites and observed that minimum fibre content is required to form a fire protective layer and the addition of APP as fire retardant agent results in significant changes for thermal and fire degradation of PBS. Khalili et al., 2017 used expandable graphite (EG) on natural fibre reinforced epoxy composite. It was observed that the flammability and thermal resistivity of the composite was remarkably improved by the inclusion of EG particles. Kalali et al., 2017 tried to improve the flame retardancy of ammonium polyphosphate retardant by combining it with phytic acid modified layered double hydroxide. It was observed that the flame retardancy improved due to the formation of a thermally stable char layer which prevented heat transfer. Kong et al., 2018 used copper phenylphosphate nanoplates with epoxy resin. It was observed that the fabrication of copper phenylphosphate improved the dynamic mechanical performance of epoxy nanocomposites, which may be due to the reinforcement effect of copper phenylphosphate with rigid structure.

In the present work epoxy based composite is prepared on incorporation of sisal fibre. In order to improve the flammability of the composite, ammonium phosphate has been added (5 to 15 wt. %). Further, the effect of ammonium phosphate on the mechanical and thermal behaviour of the composite samples has also been examined.

2. Experimental details

2.1. Materials

Sisal fibres were procured from the local market of Ranchi, Jharkhand (India). Araldite AW106 standard epoxy resin and its hardener HV953 IN were procured from Huntsman Advanced Materials Americans Inc., USA. Di-ammonium hydrogen orthophosphate was obtained from Thermo Fisher Scientific India Pvt. Ltd.

2.2. Manufacturing of composite samples

Epoxy and hardener are mixed in the ratio of 1:0.8. Sisal fibre is cut into small pieces and then dried to remove the dirt and the moisture content from the fibre. Then the fibre was mixed with epoxy resin in fixed proportions. Later, the fire retardant was also added in different proportions (5 to 15 wt. %). The sample was then prepared by hand layup technique and then compression moulded to produce sheets. Then the sheets were cut to produce samples for various different tests.

2.3. Material Characterization Thermogravimetric Analysis

This test measures the amount and rate of change in the mass of a sample as a function of temperature or time in a controlled atmosphere. DTG-60 model of Thermogravimetric analyser is used in Nitrogen atmosphere at the flow rate of 50 ml/min and heat rate of 10 ^{o}C/ min.

Flammability Test Limiting Oxygen Index:

The samples were cut according to the UL 94 standard on a 5" x 1/2" (12.7 cm x 1.27 cm). The samples were clamped on a metal clamp and were subjected to flame for 10 s. After it was left to burn on its own and the time was noted accordingly. According to the total time required to burn the LOI was allotted.

Vertical Flammability test

The samples were cut according to the UL 94 standard on a 5" x 1/2" (12.7 cm x 1.27 cm).The samples were clamped on a metal clamp and were subjected to flame for 10s. After it was left to burn on its own and the time was noted accordingly

3. RESULTS AND DISCUSSION

Thermogravimetric Analysis

Thermogravimetric analysis is a measure of thermal analysis in which the samples decomposition is studied over time as the temperature changes at a fixed rate. In this, the mass loss in carefully observed as the temperature rises.

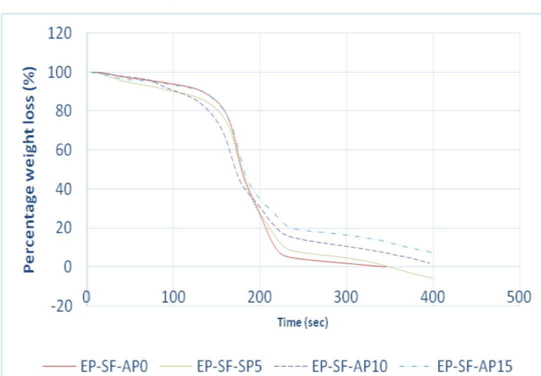

Fig 1: TGA graph of composite samples

All the material decomposition takes place in two steps as shown in Fig. 1. Addition of ammonium phosphate to the sisal/epoxy composite accelerates the first step of decomposition (below 200 ^{o}C). Hence, the first range of decomposition is not only because of sisal fibre but also because of the decomposition of flame retardant, releasing NH_3 and H_2O (Zhang et al., 2018; Statheropoulos and Kyriakou, 2000). The second part basically shows thermal insulation properties and hence, the degradation has been shifted to higher temperature. The mechanism can be understood in this way that ammonium phosphate interacts with the composite, leading to generation of volatile compounds on degradation. This forms a protective layer around the polymer matrix which decomposes to form char and thus protecting the polymer matrix. Thus, we can observe that on the addition of flame retardant the residual left in the end increases. Similar effect was observed by Zhang et al., 2018.

Flammability Test

Limiting Oxygen Index (LOI) is used for the determination of fire self-extinguishing property of epoxy- sisal/ ammonium phosphate composites. Table 4 gives the LOI of all the composite samples.

Table 4: Limiting Oxygen Index for the composite samples

SAMPLE	LOI
EP-SF-AP0	23
EP-SF-AP5	28
EP-SF-AP10	31
EP-SF-AP15	33

It can be seen that EP-SF shows LOI of 23 as the sisal fibre being a flammable material. On adding ammonium phosphate the LOI goes as high as 33 which is better than the one observed by Zhang et al., 2018. So it can be concluded that ammonium phosphate can effectively catalyse the composites to form char.

Table 5: Vertical flammability test for the composite samples

SAMPLE	TIME (sec)	OBSERVATION
EP-SF-AP0	120	Dripping occurs
EP-SF-AP5	150	Dripping occurs
EP-SF-AP10	160	Dripping occurs
EP-SF-AP15	extinguishes	The sample self-extinguishes It is classified as V-0.

The rating of sample under UL94 flammability test depends upon the burning time and the dripping phenomena. The burning time determines whether it is V-0, V-1 or no grade. The dripping phenomena distinguish between V-2 and V-1 grade. If the dripping flame ignites the cotton at the bottom then its V-2 grade (Wang et al., 2012).

4. CONCLUSION

The effect of AP on sisal/epoxy composite was studied. The TGA graph shows that the decomposition of flame retardant leads to the formation of a layer around the polymer matrix which acts as a protective layer and prevents further weight loss of polymer. This can also be proved by observing the limiting oxygen index of the samples which goes as high as 33. Thus, we can conclude that as the flame retardant content increases, the flame retardancy of the composites increases.

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There are no conflicts of interest between authors. There is no involvement of human or animal cell in this work. All the co-authors have seen the final manuscript and agreed with the submission to the journal. The manuscript has not been published elsewhere. The article is not consideration under any other journal in full or in part. No data or figures have been fabricated or manipulated. The finding agencies (NPIU/AICTE New Delhi, India) have been duly acknowledged. All the authors agree to transfer copy right to SN Applied Science's. All the further responsibilities will be undertaken by the corresponding author.

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