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Effect of multi-walled carbon nanotubes (MWCNTs) on the thermal and fire properties of the polypropylene co-polymer (PPCP) based nanocomposites

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ABSTRACT

In the study, polymer nanocomposites were prepared by using polypropylene co-polymer (PPCP), maleic anhydride grafted polypropylene (MAgPP) and multi-walled carbon nanotubes (MWCNTs). In the study, MWCNTs content in the nanocomposites was varied in the range of 0-2 wt.%. Injection moulding method was employed to prepare the nanocomposites. The effect of MWCNTs on the thermal and fire properties, such as melt flow index (MFI), UL-94, smoke density, smoke visibility, limiting oxygen index (LOI) and toxicity of the in-house prepared nanocomposites was studied. From the results, it is observed that around 18% decrease is observed in the MFI of the nanocomposites with the increase of MWCNTs loading from 0-2 wt.%. The nanocomposite samples adhered to the UL-94 classification of V-2. The nanocomposites offered a smoke density value of 2.0 except the nanocomposite with 2 wt.% of MWCNT. The nanocomposites in presence and absence of MWCNTs offered a smoke visibility of Class-A. Around 8% increase is observed in LOI when the MWCNTs loading increased from 0-2 wt.%. The toxicity of the nanocomposites increased around 8.5 times in presence of 2 wt.% MWCNTs. From the findings, it can be further said that MWCNTs played a great role in the thermal and fire properties of the nanocomposites.

KEYWORDS:Nanocomposites; PPCP; MWCNTs; Melt flow index; Fire properties; Toxicity.

1. INTRODUCTION

It is known that polymers and polymer composites are widely used due to their superior characteristics such as lightness, chemical resistance, easy processing and recycling compared to metals and metallic alloys. Various types of fibers such as glass fiber, carbon fiber, and aramid; and particles such as talc, calcium carbonate and carbon black with different particle shape and sizes are introduced into the polymers in order to enhance the various properties of polymers [1]. In recent years, carbon-based nanomaterials (such as carbon nanotubes, carbon nanofibers, and graphene derivatives) have become the most important fillers for improving the polymer properties [2, 3].

Due to the carbon nanotubes (CNTs) exceptional electrical conductivity, thermal conductivity, and tensile properties, polymer-CNT nanocomposites have received a lot of scientific and industry interest in the field of nanocomposites. Additionally, CNTs have a lot of promise for shielding electromagnetic interfaces and reducing noise [4]. Many properties such as electrical conductivity, mechanical strength and thermal stability are strongly affected by the network structure which can restrain the mobility of the polymer chains in polymer-CNT nanocomposites. The extent of property improvement in CNT filled polymer composites generally depends on several factors such as volume fraction of fillers, dispersion of CNTs in polymer matrix, type of polymer employed and fabrication method etc. [5]. It is known that polymer composites with high dielectric constant play important roles in electromechanical fields such as high performance sensors, actuators, artificial muscles, as well as bypass capacitors in microelectronics and energy-storage devices [6].

Due to the combustible nature of the polymers under certain conditions, it is necessary to improve the flammability properties of polymers with the addition of nanoscale additives. Nanocomposites are particle-filled polymers where at least one dimension of the dispersed particle is in nano size [7]. Researchers have studied the flame-retardant properties of carbon nanotubes (CNTs) in polymeric matrices such as epoxy, polystyrene, polyaniline, polypropylene, and polyurethane. Polymer-CNT nanocomposites are successful at producing continuous structured networks. In PP-CNT nanocomposites that include 0.5-4.0 wt.% of CNT, a notable decrease in the peak heat release rate was noted [8].

Salih et al., [1] have studied the effect of multi walled carbon nanotubes (MWCNTs) on the mechanical, thermal and rheological properties of polypropylene (PP) nanocomposites. In the study, the nanocomposites were prepared by melt processing methods by employing extruder and injection molding. In the study, it was reported that the properties of the nanocomposites have been improved in presence of MWCNTs. Prashantha et al., [6] have studied the preparation and characterization of PP nanocomposites. In the study, MWCNTs were filled in PP polymer to prepare the PP nanocomposites. The prepared nanocomposites were used to estimate the electrical and dielectric properties. In the study, it was reported that the electrical resistivity decreased and dielectric constant increased when the MWCNTs were incorporated in the PP matrix.The reported dielectric constant was >110 to the PP nanocomposite containing 5 wt.% MWCNTs. Carvalho et al., [9] have studied the effect of carbon nanotubes on the dielectric properties of polypropylene/high-density polyethylene (PP/HDPE) nanocomposites. In the study, dielectric properties were estimated using impedance spectroscopy. It was reported in the study that the dielectric constant was increased from 3.15 (without CNTs) to 16.91 (with CNTs) at 1 Hz.

Kashiwagi et al., [7] have studied the thermal and flammability properties of nanocomposites prepared from polypropylene and multi walled carbon nanotubes. In the study, MWCNTs content was varied from 0.5-4.0 % by weight. Flammability properties of the prepared nanocomposites were measured using a cone calorimeter in air and a gasification device in a nitrogen environment. In the work, it was reported that the peak heat release rate was significantly decreased at a MWCNT content of 1 wt.% in the nanocomposite. It was also reported that the thermal stability of the nanocomposites increased in presence of MWCNTs.

Yuan et al., [10] have studied the preparation and characterization of the polypropylene composites. The effects of graphene content (0.5-2.0 wt.%) on the composite flammability and fire behavior was studied. The authors observed that in presence of graphene in the composites decreased the release of heat and smoke. The UL-94 classification was reduced from V-0 to V-2 with graphene loading up to 2 wt.%.

Stanciu et al., [4] have studied the preparation and characterization of nanocomposites prepared with PP and MWCNTs. The nanocomposites were prepared by keeping the MWCNTs loading in the range of 1-5 wt.%. Various properties such as Thermal, Rheological, Mechanical and Electrical Properties of the prepared nanocomposites were estimated in the study. In the study, it was reported that the properties of the nanocomposites improved in presence of MWCNTs and its loading. Cabello-Alvarado et al., [8] have studied the effect of CNTs and TiO_2 nanoparticles on the thermal, mechanical, flammability, and electrical properties of PP nanocomposites. In the study, the nanomaterials TiO_2/CNT were mixed in the ratio of 1:2 using melt extrusion process; and the mixed nanomaterial was used in the preparation of PP nanocomposites at different loadings of 1-10 % by weight. The prepared PP-TiO_2/CNT nanocomposites were used to characterize various properties. In the study, it was reported that the thermal stability of the nanocomposites increased with the increase of nanomaterial loading. The flammability properties of the nanocomposites were estimated with a cone calorimeter. In the study, it was reported that heat release rate was decreased to the nanocomposites with the increase of nanoparticles loading from 5-10 wt.%. Rajesh Kumar et al., [11] have studied the effect of MWCNTs on the thermo-mechanical, thermal and electrical properties of MAgPP/PP nanocomposites. In the study, it was reported that the properties of the composites enhanced in presence of MWCNTs.

From the literature, it is observed that many studies are available mostly on the thermal, mechanical, rheological, electrical and fire properties of the nanocomposites prepared with various polymers (like polypropylene) in presence of CNTs or MWCNTs. It is further observed from the literature that in presence of CNTs or MWCNTs, the properties of the nanocomposites improved. However, to the best of our knowledge, the literature pertaining to the thermal, mechanical, rheological, electrical and fire properties of polypropylene co-polymer (PPCP) based nanocomposites in presence of fillers such as MWCNTs and maleic anhydride grafted polypropylene (MAgPP) is not disclosed in the open literature. Therefore, the objectives of the present study are to prepare the PPCP based nanocomposites in presence of MWCNTs and MAgPP; and also to evaluate their thermal and fire properties.

2. Materials and Methods

2.1. *Materials*

Polypropylene co-polymer (melt flow index = 9 g/10 min at 230 °C) was purchased from Reliance Polymers (Mumbai, Maharashtra, India). The MWCNT-PP master batch contains 20 wt.% of MWCNTs procured from Hyperion Catalysis International (Cambridge, USA). Maleic anhydride grafted polypropylene (melt flow index = 110 g/10 min at 190 °C) was procured from Plus Advanced Technologies Private Limited (Gurugram, Haryana, India).

2.2. *Preparation of composites*

The nanocomposite preparation and characterization process is shown in Figure. 1.

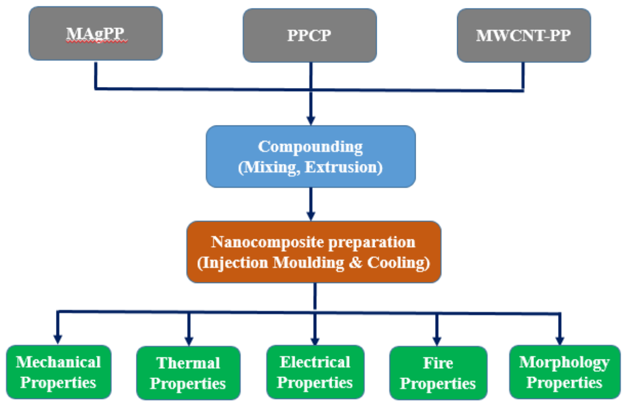

Figure 1. Preparation and characterization methodology of PPCP/MAgPP/MWCNT nanocomposites.

In the process, required quantities of PPCP pellets, MWCNT-PP master batch along with MAgPP compatibilizer were added into the extruder. The extrusion process was carried out at a temperature of 190-230 °C; and mixed through the action of the two counter-rotating blades at a rotor speed of 80 rpm. After that, the extruded product was cut into pellets and dried at room temperature. Then, the pellets were fed into an injection moulding machine (Japan Steel Works, India) that was operated at a temperature of 190-210 °C, speed of 100 rpm and holding pressure of 300 bar to prepare the nanocomposite (NC) specimens. The prepared specimens were cooled to room temperature. Finally, the nanocomposite samples were used to estimate various properties. Table 1 shows the composition of the composites used in this study. A control sample, i.e., a polypropylene co-polymer along with MAgPP sample, was also prepared using a similar procedure.

Table 1. The chemical composition of the in-house prepared nanocomposites.

Sample No.	PPCP (wt.%)	MAgPP (wt.%)	MWCNT (wt.%)
NC-1	98.0	2.0	0.0
NC-2	97.5	2.0	0.5
NC-3	97.0	2.0	1.0
NC-4	96.5	2.0	1.5
NC-5	96.0	2.0	2.0

2.3. Characterization of the in-house prepared nanocomposites

2.3.1 Melt flow index test

The melt flow index (MFI) of the nanocomposite was estimated by adopting ASTM D1238 method. The specimen can be pellets/powder/material that can fit into 10 mm; and it is fed into the barrel without any noticeable gaps and the temperature was attained. The temperature to be attained was taken according to the standards based on the material used. As the temperature was gained accordingly, the setup was set aside for 30 minutes and piston is placed. Similarly, load was applied on the piston according to the standard. On the application of the load, resultant products were collected at equal intervals. The weights of the products were noted. Two samples for each specimen were taken and the average weights of the sample were considered for the calculation of the melt flow index.

2.3.2 Flammability test

To classify the materials into V-0, V-1, V-2; the UL-94 test is conducted by using a Bunsen burner and holder. To prepare the specimen, the material was cut into rectangular shape (125 mm × 12.5 mm). The specimen was fixed vertically by using a holder. The test was then performed as the burner was introduced with flame, of height 1.5 inch (38.1 mm), tilted at an angle of 45°. The specimen was heated for a duration of 10 seconds and then the flame was turned off. Based on the burning characteristics and time of the specimen, the material was classified according to the observations given in Table 2.

Table 2. Material classification based on flammability test.

Classification	Observation
V-0	If the specimen is self-extinguishing (i.e. will not burn after the flame is removed, after 10 seconds).
V-1	If the specimen burns for 30 seconds and stops burning.
V-2	If the specimen burns totally in less than 30 seconds.

2.3.3 Smoke density test

Smoke density test was performed to determine the amount of smoke given by a material which is burning by considering ASTM D2843 method. To perform the test, the specimen was cut into the required size (125 mm ×12.5 mm). The specimen was ignited and the smoke evolved while burning was introduced into the smoke density chamber where a light beam was allowed to pass through it. The smoke density was then calculated by measuring the amount of light scattered by the smoke using a photo sensor; and the readings were noted.

2.3.4 Smoke visibility test

To determine the deterioration of visibility due to the smoke released by the combustion of material, the smoke visibility test is done by the smoke visibility testing apparatus which consists of a chamber. The chamber provides a provision for insertion of the sample, burning of the sample, a bulb and a lux meter where the readings are shown. The ULC 564 standard procedure was followed to perform the test. The specimen was cut into the specified size (110 mm × 125mm). The specimen was inserted in the slot given where it was burnt for 30 seconds. After 30 seconds, while the smoke was developing, reduction in intensity of light was observed and the readings were noted through lux meter at regular intervals for 5 minutes. Based on the values achieved, the materials were graded accordingly.

2.3.5 Limiting oxygen index test

To determine the limiting oxygen index (LOI), the oxygen index tester was used and analysis was carried out. ASTM D2863 method was adopted to perform the test. The specimen was cut into rectangular size (125 mm × 12.5mm). The specimen was held vertically in the centre of apparatus column. The flow valves were set to introduce the desired concentration of oxygen into the column. The top of the specimen was ignited with the help of a lighter. The specimen was required to burn in accordance with the set criteria. The criteria was determined based on the burning time and rate of burning. The concentration of oxygen was adjusted to meet the criteria and the test was repeated until the critical concentration of oxygen was obtained; then the readings were noted.

2.3.6 Toxicity test

To determine the toxicity index of the material, the toxicity test was conducted by using the Asian Toxicity Tester machine. NCD 1409 standard was used to perform the experiment. The specimen required for the test was cube sized 4 grams of material. The prepared specimen was then placed inside the chamber where it was burnt by using methane gas at 1150 ±50 °C for 3 minutes. The mixing fan was then switched on for 30 seconds so that the gases released would maintain the same concentration throughout the chamber. Then immediately, the gas detection tubes were inserted to pump the specified gases out from the chamber. The concentrations of individual gases were noted and the analysis was carried based on the concentrations of gases obtained. Initially the whole process was carried without using the specimen to ensure that the chamber was completely empty before the actual sample was introduced. If any gases were detected, their concentrations were noted and used during the calculation of the toxicity index.

3. Results and Discussions

3.1 Effect of MWCNTs loading on melt flow index (MFI) of the nanocomposites

In the study, the effect of MWCNTs loading on the MFI property of the PPCP nanocomposites is studied by varying the MWCNTs loading in the range of 0-2 wt.%; and the obtained results are shown in Table 3. From the data, it is observed that the MFI of the nanocomposites decreased with the increase of MWCNTs loading in the nanocomposites. Around 18% decrease is observed in the MFI of the nanocomposites with the increase of MWCNTs loading from 0-2 wt.%. As a measure of flow property, MFI's decline reveals a rise in melt shear viscosity as a result of interactions between CNT-CNT and between CNT-polymers [12-14].

Table 3. Effect of MWCNTs loading on MFI index of the nanocomposites.

Sample No.	MWCNTs loading, (wt.%)	MFI (g/10 min), at 230 °C/2.16 kg load
NC-1	0.0	10.38
NC-2	0.5	10.00
NC-3	1.0	9.54
NC-4	1.5	9.12
NC-5	2.0	8.50

3.2 Impact of MWCNTs content on flammability property of the nanocomposites

The impact of MWCNTs content on the flammability property (flame retardant property) of the PPCP nanocomposites is studied by considering the MWCNTs content in the range of 0-2 wt.%. One of the most used flammability tests for measuring the relative flammability of plastic is the UL-94. The impact of MWCNT content on UL-94 of the nanocomposites are shown in Table 4.From the results, it can be seen that all the nanocomposite samples adhered to the UL-94 classification of V-2, which denotes that burning ends within 30 seconds on a component allowing for drips of vertical flammable plastic. Further it can be said that the addition of MWCNTs in the nanocomposites has no significant impact on the flammability (flame retardancy) property under present experimental conditions.

Table 4. Impact of MWCNTs content on flammability property of the nanocomposites.

Sample No.	MWCNTs content, (wt.%)	Flammability property (UL-94)
NC-1	0.0	V-2
NC-2	0.5	V-2
NC-3	1.0	V-2
NC-4	1.5	V-2
NC-5	2.0	V-2

3.3 Effect of MWCNTs loading on smoke density characteristic of the nanocomposites

Theeffect of MWCNTs quantity on smoke density characteristic of the PPCP nanocomposites is studied with MWCNTs quantity in the range of 0-2 wt.%. This test gauges how much smoke emits by a burning or smouldering material. The material is tested when it begins to smolder and again when a flame source is added. The smoke density rating ranges from 0 (no smoke generated) to 800. A rating of 450 or less is required for the majority of finishes and materials. The impact of MWCNT content on smoke density of the nanocomposites are shown in Table 5. From the findings, it can be seen that all the nanocomposite samples offered a smoke density value of 2.0 except the nanocomposite with 2 wt.% MWCNT. The study revealed that at high MWCNT content (2 wt.%), the smoke density increased slightly due to the carbon content associated with MWCNTs.

Table 5. Effect of MWCNTs loading on smoke density characteristic of the nanocomposites.

Sample No.	MWCNTs content, (wt.%)	Smoke density, (%)
NC-1	0.0	2.0
NC-2	0.5	2.0
NC-3	1.0	2.0
NC-4	1.5	2.0
NC-5	2.0	3.0

3.4 Effect of MWCNTs loading on smoke visibility of the nanocomposites

The effect of MWCNTs loading on smoke visibility characteristic of the PPCP nanocomposites is studied by using MWCNTs quantity in the range of 0-2 wt.%. The impact of MWCNT content on smoke visibility of the nanocomposites are shown in Table 6. From the findings, it is observed that all the nanocomposite samples in presence and absence of MWCNTs offered a smoke visibility of Class-A. The study revealed that the addition of MWCNTs and its loading in the nanocomposites has no adverse effect on the nanocomposite smoke visibility property.

Table 6. Effect of MWCNTs loading on smoke visibility characteristic of the nanocomposites.

Sample No.	MWCNTs loading, (wt.%)	Smoke visibility
NC-1	0.0	Class - A
NC-2	0.5	Class - A
NC-3	1.0	Class - A
NC-4	1.5	Class - A
NC-5	2.0	Class - A

3.5 Effect of MWCNTs loading on limiting oxygen index of the nanocomposites

The effect of MWCNTs loading on limiting oxygen index (LOI) of the PPCP nanocomposites is studied, and the observations are presented in Table 7. The results indicate that the LOI value of composite without MWCNT is 26%; and it is increased when mixed with various weight percentages of MWCNTs in the composites, which indicates a reduction in ignitability. Around 8% increase is observed in LOI when the MWCNTs loading increased from 0-2 wt.%. The data further shows that MWCNT helped to increase slightly the flame retardant property of the PPCP nanocomposites by taking a longer period for the nanocomposite to burn completely.

Table 7. Effect of MWCNTs loading on limiting oxygen index of the nanocomposites.

Sample No.	MWCNTs loading, (wt.%)	LOI, (% O_2)
NC-1	0.0	26.0
NC-2	0.5	27.0
NC-3	1.0	27.0
NC-4	1.5	27.0
NC-5	2.0	28.0

3.6 Effect of MWCNTs loading on toxicity index of the nanocomposites

The effect of MWCNTs loading on toxicity index of the PPCP nanocomposites is studied by adding MWCNTs in the range of 0-2 wt.% in the nanocomposites. The effect of MWCNT loading on toxicity index of the nanocomposites is shown in Table 8. From the results, it is observed that the toxicity value of the nanocomposite in absence of MWCNTs is lower in comparison with the nanocomposites prepared in presence of MWCNTs. The toxicity of the nanocomposites increased around 8.5 times in presence of 2 wt.% MWCNTs.

Table 8. Effect of MWCNTs loading on toxicity property of the nanocomposites.

Sample No.	MWCNTs loading, (wt.%)	Toxicity index
NC-1	0.0	0.19
NC-2	0.5	0.47
NC-3	1.0	0.81
NC-4	1.5	1.19
NC-5	2.0	1.62

4. Conclusions

In the study, polymer nanocomposites were prepared by using polypropylene co-polymer (PPCP), maleic anhydride grafted polypropylene (MAgPP) and multi-walled carbon nanotubes (MWCNTs). In the study, MWCNTs content in the nanocomposites was varied in the range of 0-2 wt.%. Injection moulding method was employed to prepare the nanocomposites. The effect of MWCNTs on the thermal and fire properties, such as melt flow index (MFI), UL-94, smoke density, smoke visibility, limiting oxygen index (LOI) and toxicity of the in-house prepared nanocomposites was studied. From the results, it is observed that around 18% decrease is observed in the MFI of the nanocomposites with the increase of MWCNTs loading from 0-2 wt.%. The nanocomposite samples adhered to the UL-94 classification of V-2. The nanocomposite samples offered a smoke density value of 2.0 except the nanocomposite with 2 wt.% of MWCNT. The nanocomposite samples in presence and absence of MWCNTs offered a smoke visibility of Class-A. Around 8% increase is observed in LOI when the MWCNTs loading increased from 0-2 wt.%. The toxicity of the nanocomposites increased around 8.5 times in presence of 2 wt.% MWCNTs. From the findings, it can be further said that MWCNTs played a great role in the thermal and fire properties of the nanocomposites.

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Conflict of interest statement

Authors declare that they do not have any conflict of interest.

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