TGA Analysis of Injection Moulded Compounds

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***Abstract*- Thermal analysis of various materials can be done by Thermo-gravimetric analysis (TGA). TGA gives valued data which can be used to select neat and composite materials for certain applications and to determine parts-performance with better part quality. In this paper, thermal analysis of injection moulded Polypropylene-filler compounds were performed by TGA Analyzer. For TGA analysis, 3 compounds viz. Alumina compounds (PPA-series), TiO_2 (PPT-series) and micro-Marble-dust (PPM-series) compounds were made by injection moulding process with 5, 10,15 and 20 weight percentages of the fine fillers with Polypropylene (PP).**

Keywords- Thermal analysis, TGA Analyzer, Alumina, TiO2 and micro-Marble-dust

1. INTRODUCTION

TGA (Thermo-gravimetric analysis) defines rate of change in the weight of a compounds as a function of temperature in a controlled atmosphere. Thus, it is performed in constrained surroundings and shows the thermal stability and composition of compounds. TGA shows the physical, chemical and structural changes in the material and thus the thermal investigation can be done. TGA graph for different temperature of composite have generally 3 distinct stages which are thermally stable, decomposition and then the thermal degradation of material. Different specimens of white marble powder were investigated for thermal decomposition and the test were performed up to 1150^{o}C and pyrolysis reactions was observed in the composite by Meloni et al. [1].

2. MATERIALS AND METHODS

To analyse the thermal behaviour of fabricated compounds (Figure 1) Perkin Elmer Pyris -7 instrument was used. High temperature stability and degradation behaviour was observed for the compounds. Specimens were heating was done from 25^{o}C to 550^{o}C with the heating rate of 6°C /min. under nitrogen atmosphere. Alumina compounds (PPA-series), TiO_2 (PPT-series) and micro-Marble-dust (PPM-series) compounds were made by injection moulding process with 5, 10,15 and 20 weight percentages of the fillers with Polypropylene (PP). In table-1, the spcecifications of TGA analyzer has been shown: -

Table-1: Specifications of TGA analyzer

Design	A high sensitivity balance along with vertical design. A precision hang-down wire has suspended from the balance down into furnace.	
Balance	Capacity	1450 mg
	Sensitivity	0.1 µg
	Precision	0.01%
	Accuracy	0.03%
Temp. °C	Furnace Material	Platinum element
	Range	-25 to 1300 °C
	Scan-rates	0.1 to 450 °C/min

In the literature review it was investigated that following (Table -2) are the key factors which affects the TGA analysis of compounds: -

Table -2: Key factors for TGA analysis

Sr No.	Factors
1	Oxidation of composite
2	Decomposition of composite
3	Dehydration of composite
4	Crystalline level
5	Filler content

3. RESULTS

3.1 TGA of PP-Al_2O_3 compounds

In Figure 2, the TGA graph of samples having different weight % of PP- Al_2O_3 have been exhibited. It was exhibited that due to the balanced bonding of filler in PP takes place and the early degradation temp. of PP matrix increases.

According to investigation of Siengchin and Kocsis [2], it can be observed that the thermal stability of compounds depends upon the matrix as well as the wt. % of Al_2O_3 filler in the compound. It was observed that thermal stability has been improved in the compounds.

It can be noticed from the figure that the primary degradation occurs at approx. 335– 425ºC and end degradation occurs near 455−490ºC. For PP it was observed that primary degradation temperature was near 345ºC. Addition of filler Al_2O_3 to matrix by 20 wt. % moved end degradation temp. near 425ºC, which is 75-80ºC with the addition of alumina. This higher temperature degradation exhibited due to the absorbed thermal energy by compounds.

For the TGA analysis, like observation was investigated by Pedrazzoli et al. [3] as the dehydration process of alumina filler delays the compounds degradation.

Phenomenon of concentration of chain end radicals was observed by many researchers behind this thermal stability.

Figure 1 Thermo-Gravimetric Analyzer

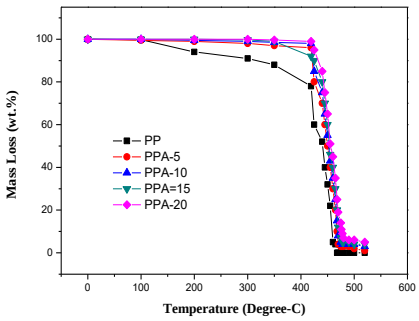

Figure 2: TGA for PP-Al_2O_3 compounds

3.2 TGA of PP-TiO_2 compounds

Figure 3 exhibits the TGA graph which examine the change in weight of TiO_2 compound with the temp. Better thermal stability can be seen in these compounds. Same behviours was observed in TiO_2 compounds as it also shows better primary degradation temperature from neat PP. In comparison to neat PP, Figure 3 also shows that end degradation temperature increased by 75ºC in 10 weights % of TiO_2 compound. Better thermal stability of all the compounds were noticed in these compounds. With the increased adhesion force at PP-TiO_2 interface better degradation temperatures was noticed by many researchers.

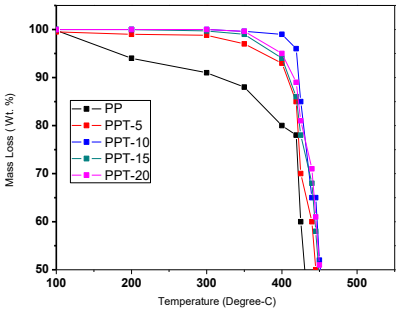

Figure 3 TGA for PP-TiO_2 compounds

Higher temperatures of 450-457.2ºC obtained showing the better thermal statbilty due to fine interfacial molecular interaction, fine grains are able to restrain the association of a polymer chain, creating the scission of molecular chains which are harder at the lesser temperatures [4]. Due to this reason, the degradation temperature of the compounds shifts to upper temperature according to researchers. The absorption of chain-end-radicals with beta-scission of molecules-radicals were the causing phenomena with this higher stability of the compounds.

3.3 TGA of PP/micro-Marble dust compounds

The thermal characteristics of fabricated PP/ micro-Marble-dust compounds has been shown in Figure 4. It indicates that as the temp. increases, the weight of these compounds decrease with the different rates [5].

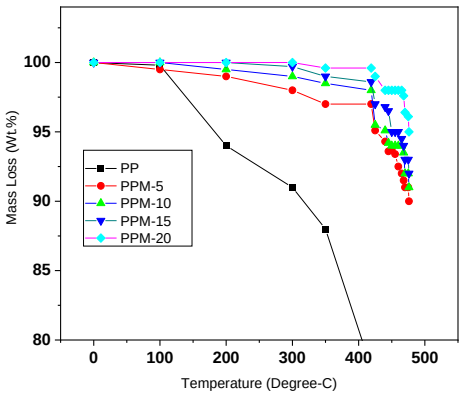

Figure 4 TGA for PP-Marble compounds

Due to dehydration procedure and the removal of unstable impurities, initially at 200°C, there was few slight losses about 0.08% in the compounds. It was observed that in the area of 350-450°C in all the graphs, the thermal degradation of PP/micro-Marble dust compounds starts for degradation. As shown in figure 4, less thermal degradation temperature was observed in 5 and 10 weight % of micro marble compounds which is comparable with neat PP.

Due to the phenomena of higher molecular char-yield, the 15 weight % marble compound exhibits greatest thermal degradation temp.

One sharp peak was noticed in range 345°C to 395°C which corresponds to the endothermic-peaks at near 355°C with weight loss of 2.5% during micro-marble compound's TGA analysis. Furhter the endothermic peak was noticed in range of 700- 880°C having micro-marble compound's weight loss near 25%. This phenomenon occurs as the evolution of CO_2 with formation of minerals CaO-MgO. According to Mehta et al. [6], the decomposition of carbonates occurs in the 2 phases, so weight loss observed in micro marble dust compounds.

6 CONCLUSIONS

The TGA of fabricated injection moulded compounds were conducted. In all the fillers, better thermal stability of compounds was noticed in comparison to neat PP. By the TGA analysis, primary degradation and end degradation temperatures of all the three compounds were determined with their possible causes regarding the molecular interface phenomenon. This study can help to design the composite product with the better thermal stability and sustainability which can be useful for industrial purpose. This study also helps in surface modification and to obtain better thermal and mechanical properties of compounds.

The study proposes a cost-effective and smart replacement of expensive fillers & materials with the use of micro-marble-dust which is currently widely available and industrial waste.

REFERENCES

[1] P. Meloni, F. Manca, and G. Carcangiu, "Marble protection: An inorganic electrokinetic approach," Appl. Surf. Sci., (2013). vol. 273, pp. 377–385.

[2] S. Siengchin and J. Karger-kocsis, "Mechanical and stress relaxation behavior of Santoprene Ò thermoplastic elastomer / boehmite alumina nanocomposites produced by water-mediated and direct melt compounding," Compos. Part A, (2010), vol. 41, no. 6, pp. 768–773.

[3] D. Pedrazzoli, V. M. Khumalo, J. Karger-Kocsis, and A. Pegoretti, "Thermal, viscoelastic and mechanical behavior of polypropylene with synthetic boehmite alumina nanoparticles," Polym. Test., (2014), vol. 35, pp. 92–100.

[4] D. Kumar, A. Srivastav, & V. Gautam, "Investigation on mechanical and thermo-mechanical properties of injection molded PP-TiO2 composites," Materials Today: Proceedings, (2021), 44, pp. 4607-4611.

[5] Ror, C. K., Tejyan, S., & Kumar, N. "Effect of marble dust reinforcement in composites for different applications: A review", Materials Today: Proceedings (2022), vol. 60, pp. 1120-1124

[6] D. Mehta, P. Mondal, and S. George, "Utilization of marble waste powder as a novel adsorbent for removal of fluoride ions from aqueous solution," J. Environ. Chem. Eng., (2016), vol. 4, no. 1, pp. 932–94