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Differential Scanning Calorimetry and Fourier Transform Infrared Analyses of Filled Polymer Pellets

Nazim Basir Nawi[1], Saliza Asman[1*]

[1] *Department of Physics and Chemistry, Faculty of Applied Sciences and Technology, UTHM Kampus Cawangan Pagoh, Hab Pendidikan Tinggi Pagoh, KM 1, Jalan Panchor, 86400 Pagoh, Muar, Johor, MALAYSIA*.

*Corresponding Author: salizaa@uthm.edu.my
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Abstract
This study investigates the effects of filler incorporation on the chemical structure and thermal properties of polymer masterbatch pellets using Fourier Transform Infrared (FTIR) spectroscopy and Differential Scanning Calorimetry (DSC). FTIR analysis revealed that both with and without filler samples are based on a polyolefin matrix, as confirmed by the characteristic C–H stretching vibrations at 2916–2921 cm^{-1} and 2849–2855 cm^{-1}. However, the masterbatch with filler exhibited additional absorption bands within the regions of 1600–1500 cm^{-1}, 1260–1150 cm^{-1}, and 1110–1010 cm^{-1}, indicating the presence of oxygen- and nitrogen-containing functional groups associated with surface-treated fillers, compatibilizers, or dispersing agents. DSC analysis showed that both samples followed the expected thermal transition sequence of Tg < Tc < Tm. The masterbatch with filler exhibited a lower crystallization enthalpy (0.62 J/g) compared to the masterbatch without filler (0.96 J/g), suggesting reduced crystallization ability due to restricted polymer chain mobility caused by the filler particles. In addition, the melting temperature (Tm) of the masterbatch with filler was slightly lower (123.32°C) than that of the masterbatch without filler (123.65°C), indicating disruption of crystalline perfection within the polymer matrix. Overall, the findings demonstrate that filler incorporation significantly modifies the chemical structure and thermal behaviour of the masterbatch pellets while maintaining the fundamental characteristics of the polyolefin-based polymer system.

1. Introduction

This study evaluates plastic pellets with and without a filler, focusing on differences in functional groups and thermal properties, such as Tg, Tm, and Tc. The filler enhances the polymer crystallinity while simultaneously affecting the thermal degradation behaviour of the polymer through changes in polymer–filler interactions and composition. There are different types of fillers used in the industry, and each type has its own roles and purposes. Whenever it comes to studying the thermal properties of a polymer, it is important to determine the type of polymer used, such as thermosetting or thermoplastic polymers. A thermosetting polymer will turn into a liquid-solid state when its temperature surpasses its melting temperature, Tm, and then is cooled down, which is an irreversible solidification process.

Whilst thermoplastic polymer will soften and incorporate a viscous liquid state when reaching its glass transition temperature, Tg, followed by its transformation into glass or semicrystalline hard solids state after a

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cooling process. Continuous heating of thermoset polymer will result in chemical decomposition and thorough structural changes. Compared to the irreversible process of heating of thermosetting polymer, thermal melting-solidification behaviour of thermoplastics is an irreversible and limited process, which means that only a restricted number of heating and cooling cycles can be performed without any structural or functional effects such as colour and shape modifications, microstructural changes, and mechanical malfunction [1].

There are several approaches to the classification of fillers based on their different characteristics. As stated by the state of aggregation, all known fillers are divided into gaseous, liquid, and solid. By their nature, fillers are divided into organic and inorganic; according to the source of receipt - reinforcing, strengthening, neutral; by size, particle shape, and structure, which are further divided into 4 main types. The 4 main types being dispersed (powder); fibrous (fibres, threads, bundles, etc.); sheet (film) with a given structure (fabrics, paper, tapes, sheets, films, nets); volumetric (framework) with a continuous three -dimensional structure (bulk fabrics, felt, skeletal and porous frameworks) [2]. The addition of fillers has become a common method of improving the performance of composites. Therefore, there are a lot of types of fillers that are commercially available, while various other types are being studied.

At present, polymers are widely used due to their high specific strength, good overall performance, and ease of processing into complex shapes. Nevertheless, many polymers still suffer from poor thermal and mechanical stability, limited chemical resistance, and low operating temperatures. To enhance their properties, fillers are commonly incorporated into polymer matrices [3]. Despite extensive research on polymer composites, there remains a lack of detailed understanding regarding the effects of fillers on the thermal behaviour and structural characteristics of polymers. Hence, further studies are necessary to clarify the influence of fillers on polymer crystallinity and degradation behaviour.

2. Materials and Samples

The instruments used in the analysis were Fourier Transform Infrared (FTIR) Spectroscopy and Differential Scanning Calorimetry (DSC). Only two materials were used to prepare the samples, which are plastic filler and polymer pellet, which are used to make plastic models.

2.1 Material Selection

A commercial putty filler was used in this study. Such fillers are generally employed for repairing holes and cracks up to 5 cm deep. The filler, supplied in powder form, was mixed with water at a 2:1 powder-to-water ratio to form a lump-free paste prior to application [4]. The putty filler used was purchased online and consisted of two mixing paste components, black and white in colour. The polymer that was used to make the sample is called polymer masterbatch, which can be bought in pellet form. A masterbatch is a blend of rubber and one or more compounding ingredients. The compounding ingredients must be in higher concentrations than they would normally be present in a complete compound. Master batching is used for accuracy and efficiency in dispersion during mixing, in order to avoid adding accelerators, antioxidants, pigments, sulphur, and peptizing agents in small quantities [5]. An alternative definition for masterbatch is a concentrated mixture obtained by the distribution of colours and additives into a polymer by heat treatment, particularly a high shear mixing extruder. The mixture will then be cooled, cut, and formed into granules via a pelletizer.

2.2 Sample Preparation

To prepare the samples for both FTIR and DSC analysis, the first step is to mix equal amounts of the “mixing paste” for 3 to 5 minutes until the two are combined into one colour. Then, a mixed-form filler, which is a pinch of the filler, was taken to cover a single masterbatch polymer pellet. For FTIR, two masterbatch pellets were coated with the putty filler to be crushed into fine powder, while the other two were not. For DSC, on the other hand, only two masterbatch pellets are required, in which one masterbatch pellet is coated with the putty filler while the other one isn't. The coated masterbatch pellets are then left for 4 hours for hardening and another 24 hours for complete solidification. One coated masterbatch pellet is then cut into several small pieces, with only the pieces being used for the sample for DSC analysis. Fig. 1 shows the plastic pellets and putty fillers used in this analysis.

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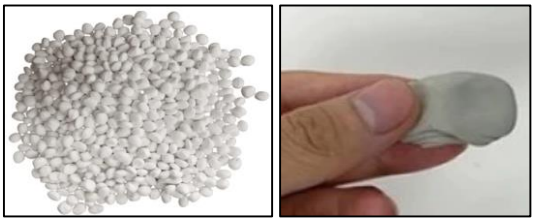

(a) (b)

Fig. 1 (a) Plastic pellets as the "Masterbatch Pellets"; (b) Putty filler used (5 - 6 mm in size)

3. Results and Discussion

3.1 FTIR Result

The graphs of both samples, which are masterbatch pellets with filler and without filler, are represented by Fig. 2(a) and Fig. 2(b), respectively. Based on Fig. 2(a) and Fig. 2(b), both spectra show strong absorption bands at the wavelengths of 2916–2921 cm^{-1} and 2849–2855 cm^{-1}. These characteristics are attributed to C–H asymmetric and symmetric stretching vibrations of aliphatic hydrocarbon chains. These bands confirm that the carrier resin in both samples is a polyolefin-based polymer, such as polyethylene (PE) or polypropylene (PP), which is typical for masterbatch formulations.

In the masterbatch pellets with fillers in Fig. 2(a), additional absorption features are shown in the 1600–1500 cm^{-1} region. This wavelength corresponds to C=O stretching and NH_3^+ symmetric bending vibrations. These peaks indicate the presence of functional additives or surface-treated fillers such as coupling agents or dispersants, which are introduced to improve filler–polymer compatibility. However, these bands are weak or absent in the masterbatch pellets without fillers. This suggests a lower concentration of oxygen- or nitrogen-containing functional groups. The fingerprint region (1300–900 cm^{-1}) shows a stark contrast between the two samples. It also shows stronger and more complex absorption bands around 1260–1150 cm^{-1} and 1110–1010 cm^{-1}, which belong to C–O–C stretching vibrations. These peaks may be attributed to ether or ester-related functional groups originating from filler surface treatments, dispersing agents, or other additive systems used to improve filler dispersion and stability within the polymer matrix.

Meanwhile, masterbatch pellets without filler in Fig. 2(b) show only weak C–O–C bands. In the low-wavenumber region (900–500 cm^{-1}), Fig. 2(a) displays sharp bands 872, 828, and 712 cm^{-1}. These indicate N–H out-of-plane bending and O-C-N deformation modes. These vibrations further prove the presence of inorganic or organo-modified fillers. As opposed to Fig. 2(a), Fig. 2(b) shows significantly reduced intensity in this region. This proves the absence of filler added into the masterbatch pellets. The FTIR results show that while both samples share the same polyolefin carrier matrix, the presence of filler introduces additional functional groups and deformation bands specifically in the fingerprint and low-wavenumber regions. Fig. 2(a) shows a simpler spectrum dominated by hydrocarbon vibrations that reflect a clean polymer matrix with minimal additive content. These findings confirm the successful addition of filler into the masterbatch pellets and its detectable influence on the chemical structure.

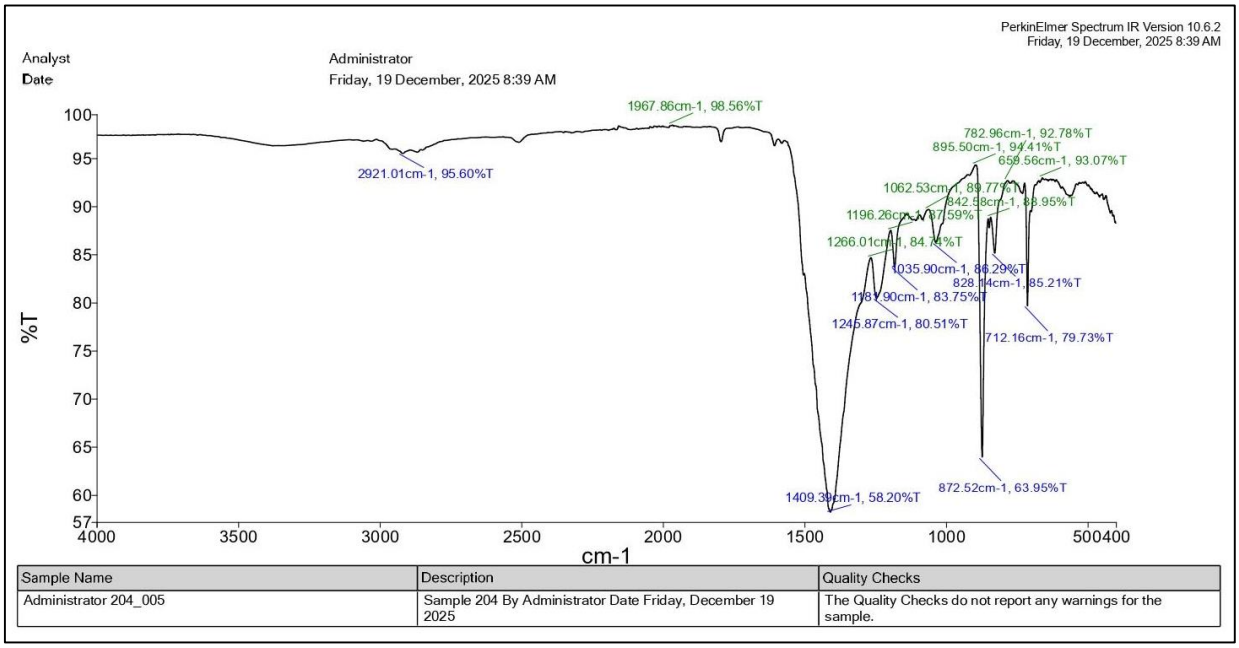

Fig. 2 (a) FTIR result of masterbatch pellets coated with filler

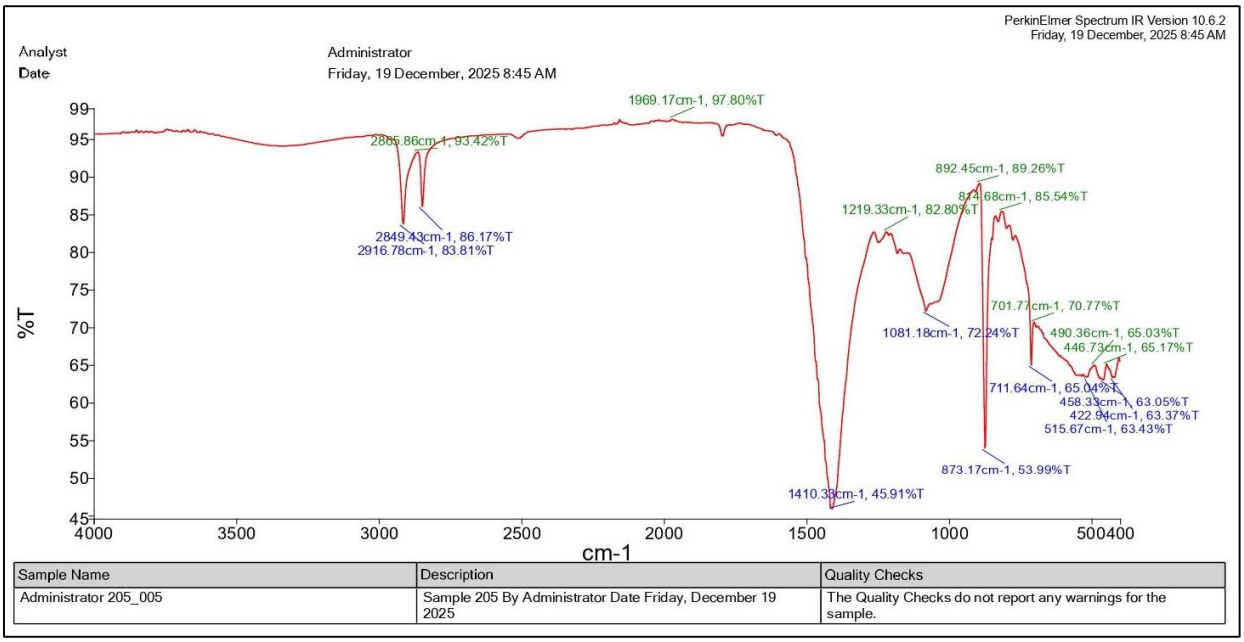

Fig. 2(b) FTIR result of masterbatch pellets without filler

3.2 DSC Result

Based on Fig. 3(a) and Fig. 3(b), both samples exhibit three important thermal transition regions that follow the expected order of Tg < Tc < Tm. The glass transition temperature (Tg) occurs at a lower temperature region and is represented by a slight baseline shift associated with increased segmental mobility of the polymer chains. Following Tg, the samples exhibit cold crystallization behaviour (Tc) at approximately 103.33–103.99°C, indicating the rearrangement of amorphous polymer chains into more ordered crystalline structures during heating [6-8]. The DSC thermograms of both masterbatch pellet samples exhibit similar thermal transition behaviour, indicating that both materials are based on a similar polyolefin polymer matrix. This similarity suggests that the incorporation of filler does not significantly alter the fundamental thermal transition sequence of the polymer system.

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The masterbatch pellets without filler show a crystallization enthalpy of 0.96 J/g, whereas the filled masterbatch pellets exhibit a lower enthalpy of 0.62 J/g, suggesting that the presence of filler restricts polymer chain mobility and reduces crystal growth during heating [9-11]. Different observations between the filled and unfilled masterbatch pellets indicate a greater degree of crystallization. In addition, the filled masterbatch shows a slightly lower melting temperature (Tm) than the unfilled sample, implying that the presence of filler may disrupt the crystalline structure and reduce crystalline perfection within the polymer matrix.

At higher temperatures, both samples display a distinct melting transition (Tm) at approximately 123–126°C. The masterbatch pellets without filler exhibit a melting temperature of about 123.65°C, while the filled sample shows a slightly lower melting temperature near 123.32°C. This reduction in Tm may be attributed to the disruption of crystalline perfection caused by the incorporation of filler particles. Therefore, the filler not only affects crystallization behaviour but also slightly reduces the thermal stability and melting characteristics of the polymer matrix [12-14]. Overall, the DSC analysis demonstrates that filler incorporation influences the thermal behaviour of the polymer by reducing crystallization ability and slightly lowering the melting temperature, while maintaining the overall thermal transition characteristics of the polyolefin-based masterbatch system.

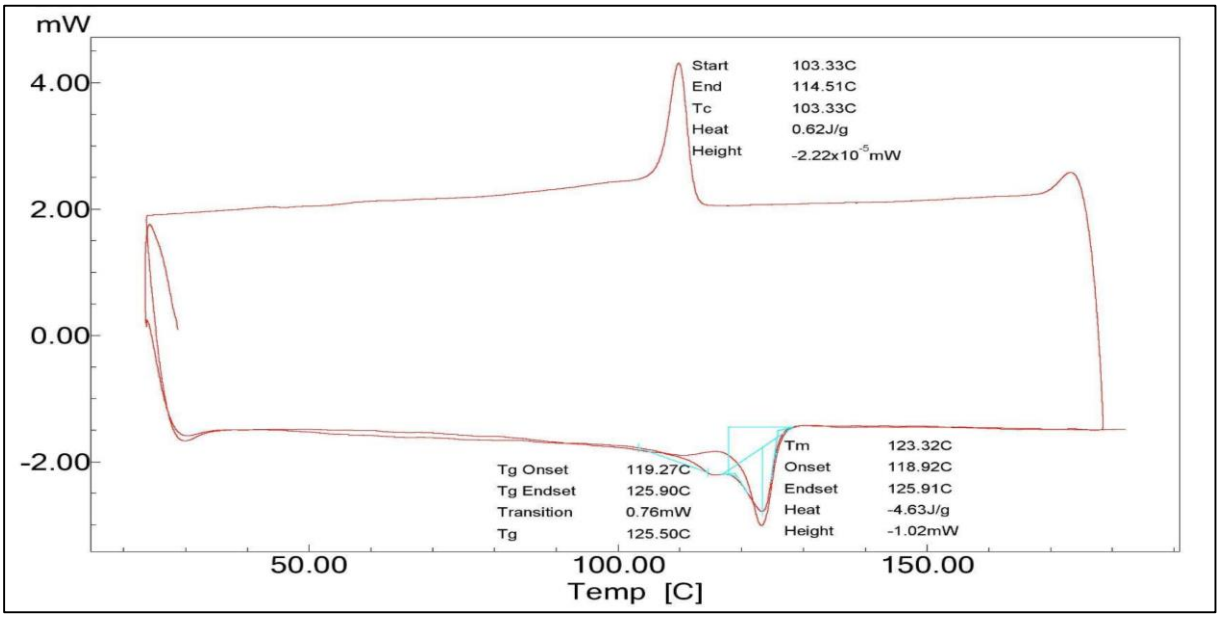

Fig. 3(a) DSC result of masterbatch pellets with filler

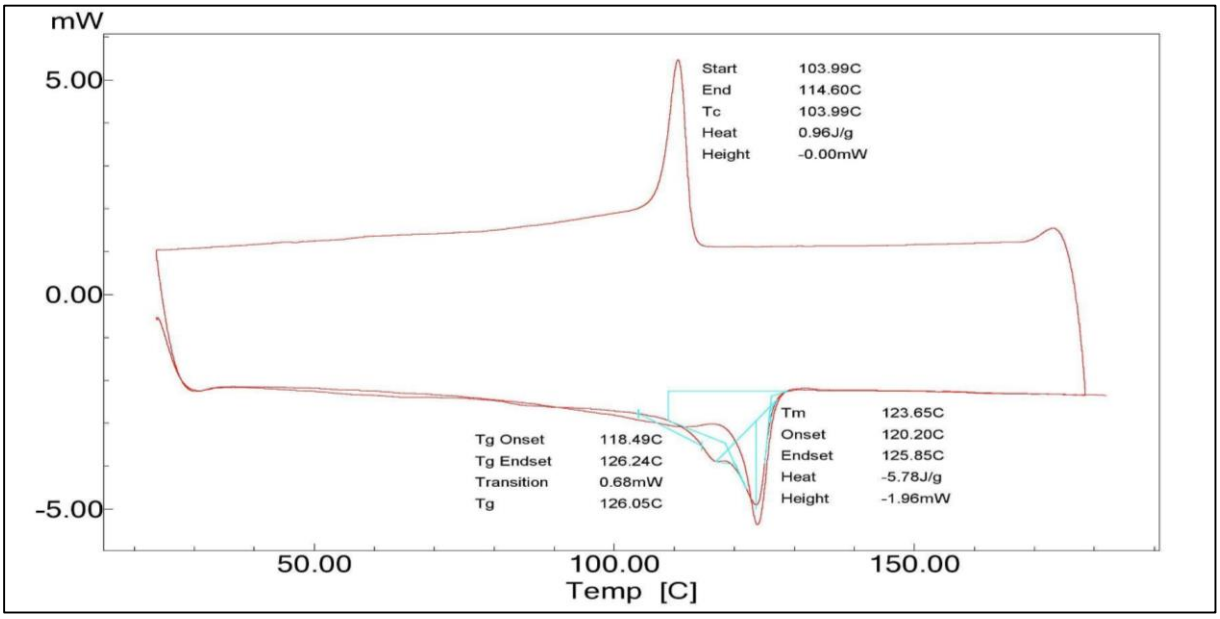

Fig. 3(b) DSC result of masterbatch pellets coated without filler

4. Conclusion

In conclusion, the FTIR and DSC analyses confirm that both masterbatch pellet samples are based on a similar polyolefin polymer matrix, most likely polyethylene (PE) or polypropylene (PP). However, the incorporation of filler introduces additional functional groups and structural modifications, particularly in the fingerprint and low-wavenumber regions, indicating the presence of surface-treated fillers, compatibilizers, or dispersing agents that enhance filler–polymer interactions. Furthermore, the DSC results show that both samples follow the expected thermal transition sequence of Tg < Tc < Tm, although the filled masterbatch exhibits lower crystallization enthalpy and a slightly reduced melting temperature compared to the unfilled sample. This suggests that the filler restricts polymer chain mobility, disrupts crystal growth, and reduces crystalline perfection within the polymer matrix. Overall, the incorporation of filler successfully modifies the chemical structure and thermal behaviour of the masterbatch pellets while maintaining the fundamental characteristics of the polyolefin-based polymer system.

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Conflict of Interest

The author declares that there is no conflict of interest regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Nazim Basir Nawi;* ***data collection:*** *Nazim Basir Nawi;* ***analysis and interpretation of results:*** *Nazim Basir Nawi;* ***draft manuscript preparation:*** *Nazim Basir Nawi, Saliza Asman. The author reviewed the results and approved the final version of the manuscript.*

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