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Experimental Study of Polymers Thermal Behavior by Differential Scanning Calorimetry – DSC

Georgeta Nan

Universitatea Petrol-Gaze din Ploieşti, Bd.Bucureşti, nr. 39, Ploieşti
e-mail: n_georgeta@yahoo.com

Abstract

In this paper, we study the thermal behavior of olefin polymers (polyethylene (PE), polypropylene (PP), polybutene-1 (PB-1) and some polyesters (polyethylene terephthalate (PET) and polybutylene terephthalate (PBT)) using differential scanning calorimetry (DSC). Experimental study of these polymers by DSC pursued to provide information on their thermal behavior, results that are complying with the literature data.

Key words *olefin polymers, polyesters, differential scanning calorimetry*

Introduction

Differential scanning calorimetry (DSC) is a thermal method, which is part of a group of characterization and control methods, which are based on following physical and chemical changes that a temperature-dependent material undergoes.

This technique measures the energy required to set a near zero difference between a substance and an inert material which are subject to the same temperature regimes when heated or cooled at a controlled rate.

Interpretation of DSC curves cannot be done automatically by any specific program. It takes years of experience in thermal analysis, but also the knowledge and understanding of possible reactions or transformations that may be suffered by the sample to be investigated [1-7].

Experimental Detail

In this work, to analyze the DSC curves recorded in air, was used equipment Mettler Toledo DSC 1 Star System, provided with associated software for thermal effect analysis [6].

Standard calibration was performed with indium, and the crucibles used were made by aluminium with a perforated lid. The sample was heated at the rate of 10^0C/min and cooled at the rate of 15^0C/min.

Results

Olefin polymers contain only carbon and hydrogen atoms and do not have aromatic rings. The studied polymers are: polyethylene, polypropylene and polybutene-1.

Polyethylene: analyzing the DSC heating curve (fig. 1) we observe that it is highly ordered and has an imperceptible amorphous fraction, the melting is emphasized by an endothermic peak. It is considered as the melting temperature, the temperature corresponding to the maximum peak of 137.8^{0}C.

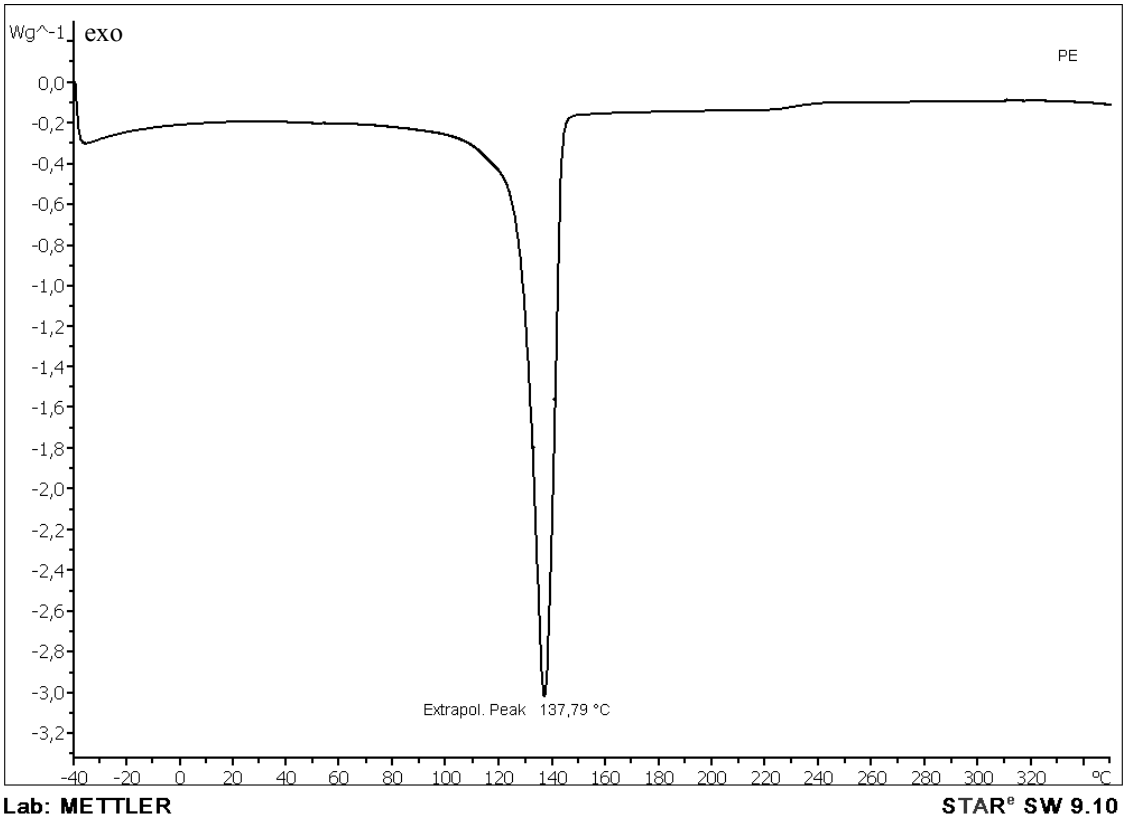

Fig. 1. DSC curve of the sample of PE - the mass of the sample was 7.87 mg, heating rate of 10^{0}C/min in the temperature range -40-350^{0}C

Further to the endothermic process of melting is an exothermic process characteristic to the decomposition process.

Polypropylene: PP sample was heated from-40^{0}C to 240^{0}C (segment 1), cooled to -40^{0}C (segment 2) and heated again at 240^{0}C (segment 3).

Looking to polypropylene DSC heating curve (fig. 2) we observe that:

In segment 1, melting is evidenced by a heating absorption zone with an endothermic peak. It is considered as the melting temperature, the temperature corresponding to minimum peak value, of about ~173^{0}C.

Segment 2 shows the presence of an exothermic crystallization "cold" peak characterized by a maximum value of ~ 121^{0}C.

In segment 3, the melting temperature value is corresponding to the minimum endothermic peak which is 167^{0}C.

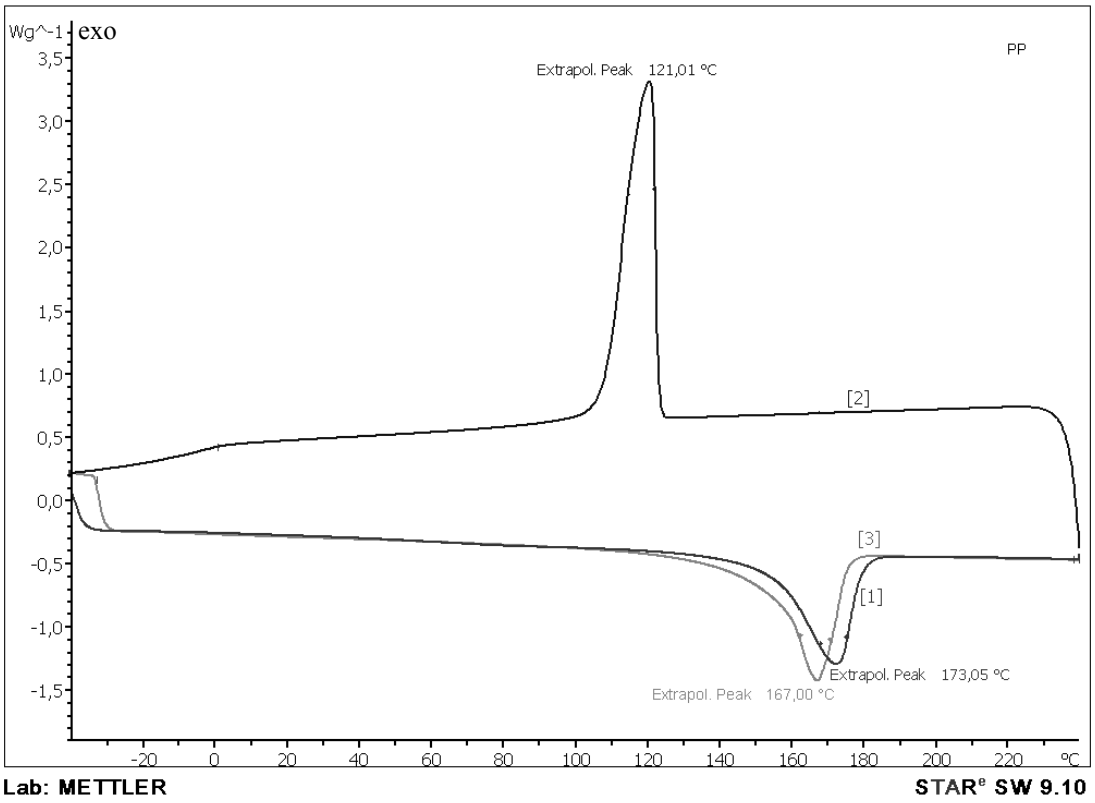

Fig. 2. DSC curves of PP sample, sample weight was 13.6 mg

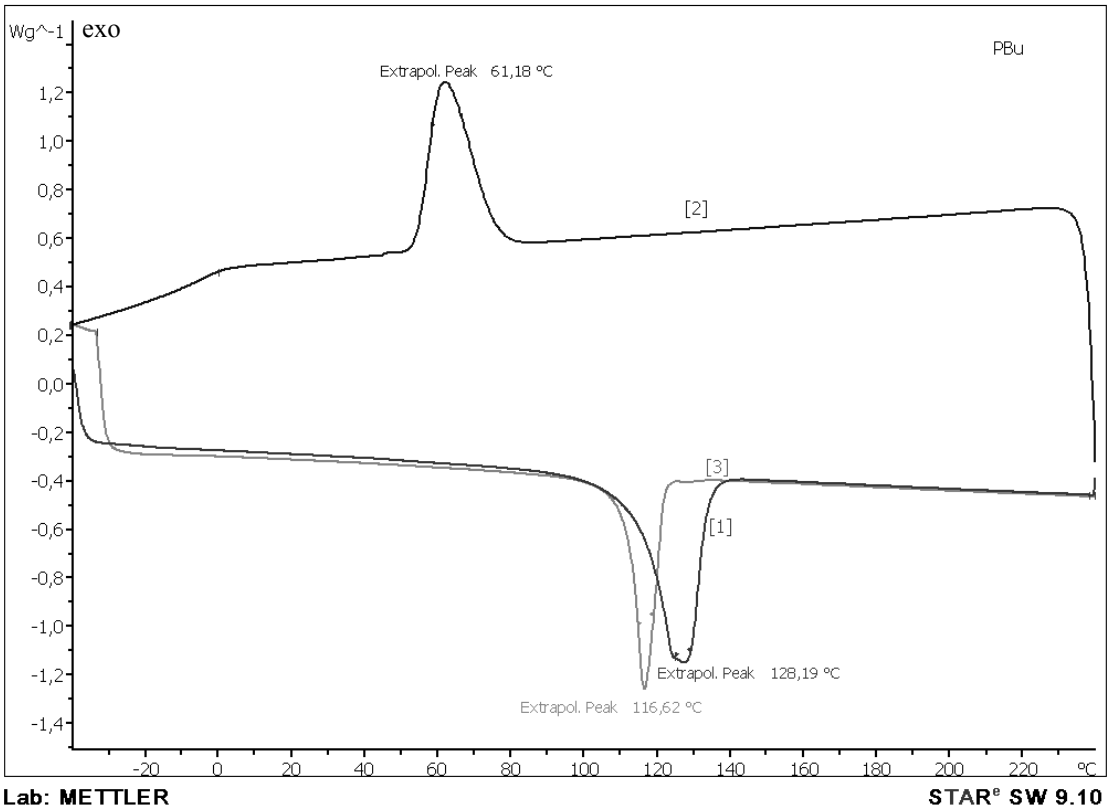

Fig. 3. The DSC curves for the PB-1 sample, the sample weight was 7.75 mg

Polybutene-1: The sample was heated from-40^{0}C to 240^{0}C (segment 1), cooled to-40^{0}C (segment 2) and heated again at 240^{0}C (segment 3).

Studying for polybutene-1 DSC heating curve (fig. 3) it is observed that:

In segment 1 melting is evidenced by a heating absorption zone with an endothermic peak. It is considered as the melting temperature, the temperature corresponding to the minim of the peak, the value is about ~128^{0}C.

By analyzing the segment 2, we observe an exothermic crystallization peak characterized by a maximum value of ~ 61.2^{0}C.

In segment 3 the value of melting temperature corresponds to the minim of endothermic peak, which is ~ 116.6^{0}C

Studied polyesters are polyethylene terephthalate (PET) and polybutylene terephthalate (PBT).

Polyethylene terephthalate: the sample was heated from-40^{0}C to 340^{0}C (segment 1), cooled to-40^{0}C (segment 2) and heated again at 340^{0}C (segment 3).

Analyzing PET DSC heating curve (fig. 4) it is observed that:

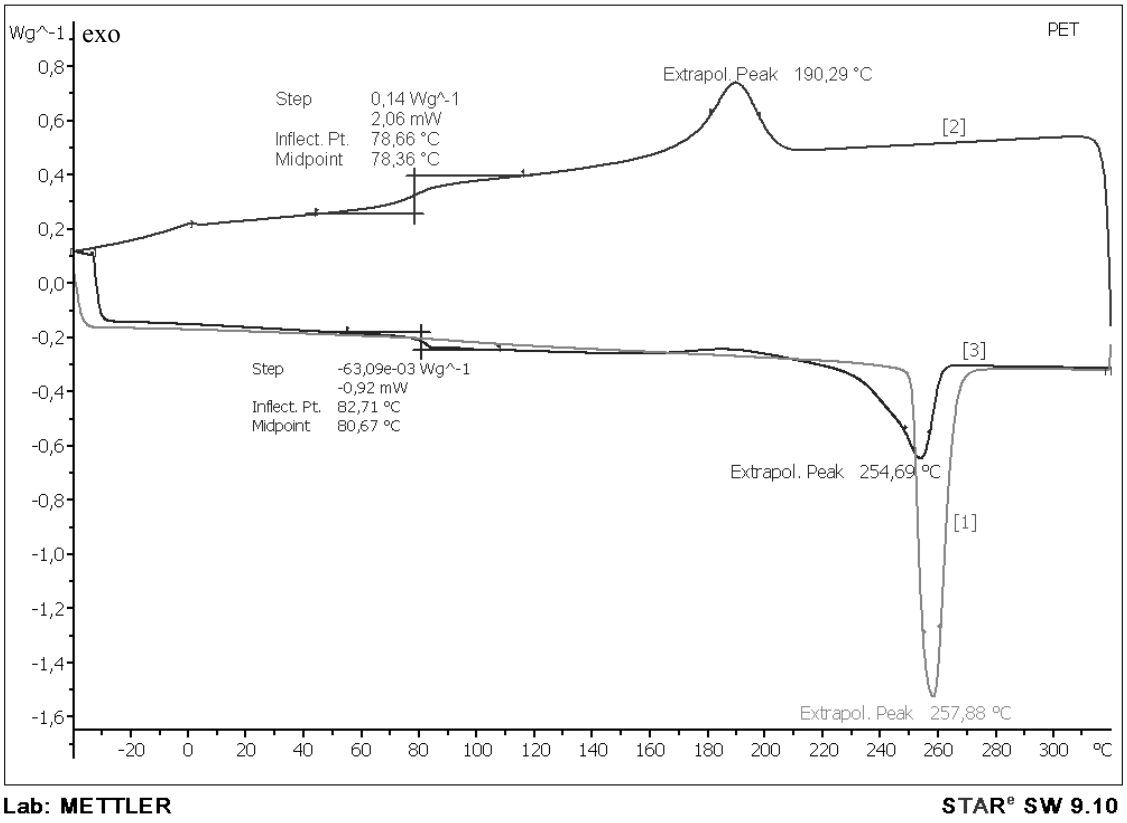

Fig. 4. DSC curves of PET sample, sample weight was 14.51 mg

In segment 1 melting is evidenced by an absorption zone with an endothermic heat peak. It is considered as the melting temperature, the temperature corresponding to minimum peak value, being ~ 257.8^0 C.

Analyzing segment 2, we see either the presence of an exothermic crystallization peak characterized by a maximum value of ~ 190,3^0 C and also the glass transition characterized by a temperature T_g = 78.3^0 C.

Analyzing the segment 3, we see that the melting temperature corresponding to the minimum of endothermic peak is ~ 254.7^0 C and that the glass transition is characterized by the temperature

T_g = 80.7^0 C. So we can conclude that PET is a polymer strongly ordered, that by heating or cooling became partially amorphous (T_g).

Polybutylene terephthalate: the sample was heated from-40^{0}C to 340^{0}C (segment 1), cooled to-40^{0}C (segment 2) and heated again at 340^{0}C (segment 3).

Analyzing PBT, DSC heating curve (fig. 5) we observe that:

On segment 1, melting is highlighted by a heating absorption zone with a bimodal endothermic melting peak, that can occur due to reorganization and premelting phenomenon. It is considered as melting temperature, the temperatures corresponding to the minimum peak values, the values are ~ 214.6^{0}C and ~ 230.4^{0}C.

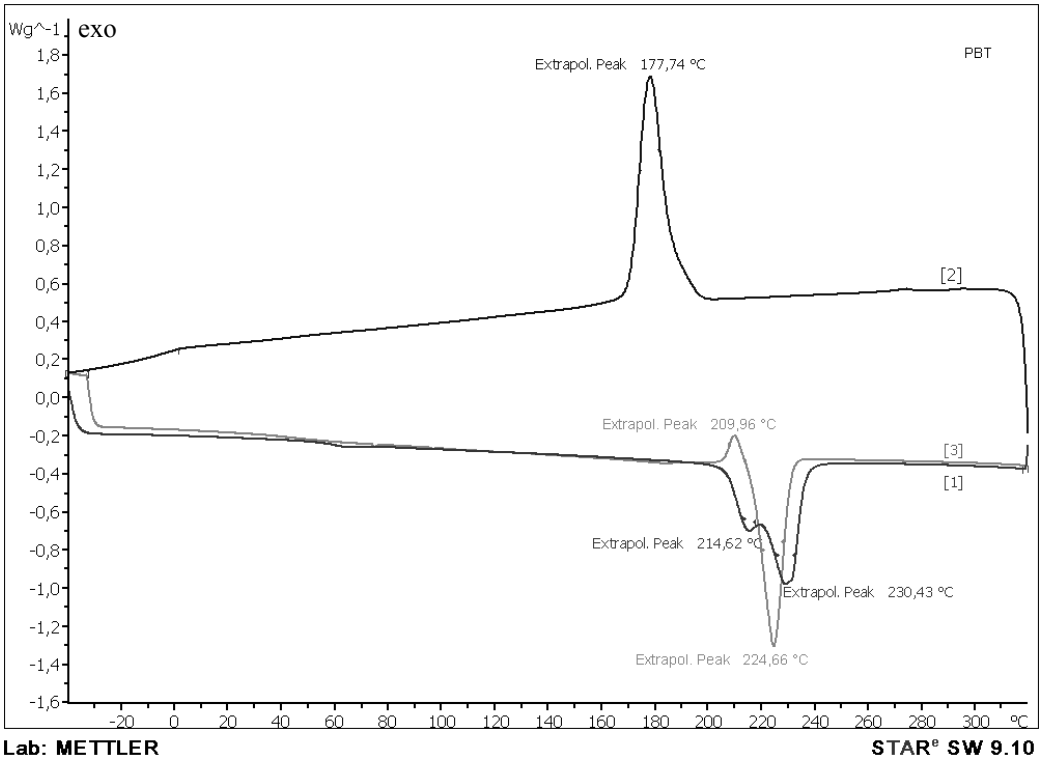

Fig. 5. DSC curves of PBT sample, sample weight was 17.01 mg

On segment 2 is observed an exothermic crystallization peak characterized by a maximum value of ~ 177.7^0 C.

On segment 3 we observe a crystallization temperature of ~209.9^{0}C, followed by the polymer melting. Melting temperature value corresponding to the minimum endothermic peak is de ~224.7^{0}C.

Conclusion

DSC characterizations of PE, PP, PB-1, PET and PBT have brought information on the thermal behavior of those materials, results that are complying with the literature data [8].

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Studiul experimental al comportării termice a unor polimeri prin calorimetrie diferențială de baleiaj – DSC

Rezumat

În aceasta lucrare se studiază comportarea termică a unor polimeri olefinici (polietilena (PE), polipropilena (PP), polibutena-1 (PB-1) şi a unor poliesteri (polietilentereftalat (PET) şi polibutilentereftalat (PBT)) folosind calorimetria diferențială de baleiaj (DSC).
Studiul experimental al acestor polimeri prin DSC a urmărit să aducă informații asupra comportării lor termice rezultate care sunt în acord cu datele din literatură.