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Experimental study of polymers thermal behavior by differential scanning calorimetry – DSC

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Abstract
This study investigates the thermal behavior of selected polymers ethylene-co-propylene and hydrogenated isoprene-co-styrene using Differential Scanning Calorimetry (DSC). Polymer samples were subjected to controlled heating and cooling cycles, and the heat flow associated with thermal events was recorded. DSC analysis revealed distinct thermal transitions corresponding to polymer-specific structural characteristics. The results demonstrated that semicrystalline polymers exhibited clear melting and crystallization peaks, while amorphous polymers showed only a glass transition. The study highlights the sensitivity of DSC in detecting subtle thermal events and provides insights into the thermal stability and phase behavior of polymers, which are critical for processing and application design. The findings contribute to understanding how polymer composition and morphology influence their thermal properties and potential performance under varying thermal conditions.

Keywords: Polymers, DSC, experimental study

Introduction

Differential scanning calorimetry is a thermoanalytical method that measures the difference in the amount of heat required to increase the temperature of a sample and a reference – maintained at the same temperature throughout the experiment – as a function of temperature [1, 3]. This technique provides qualitative and quantitative information on the physical and chemical changes that occur in the sample to be analyzed during heating and that involve endothermic or exothermic processes or changes in heat capacity. On the one hand, chemical reactions can be studied, with the determination of kinetic parameters: activation energies and pre-exponential factors, kinetic constants and reaction orders [4, 6], and on the other hand, the characteristic properties of the samples such as melting, crystallization and vitrification of polymers [7].

The characteristic appearance of a DSC curve is shown in figure 1.

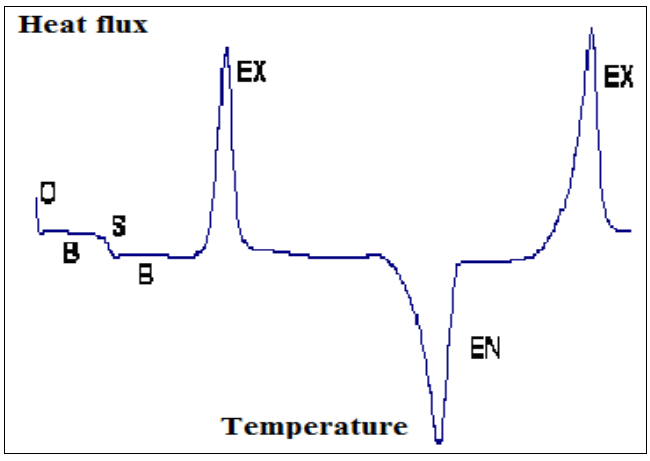

Fig 1: Appearance of a DSC curve

The notations on the curve have the following meanings: O – compensation of the heat capacity of the crucible with the sample and its contents and of the crucible with the reference and its contents; B – baseline, whose position is dependent on the heat capacity of the sample; S – gradual decrease of the baseline, which represents the change in the heat capacity of the sample unaccompanied by a change in enthalpy; EX – exothermic effect; EN – endothermic effect.

The DSC method allows the determination of the heats of reactions that occur in the analyzed temperature range (usually from room temperature to approximately 600°C, depending on the company supplying the device).

The substance is placed inside a small metal crucible, the nature of the material from which the crucible is made depending on the process for which it is used: physical transitions or thermal effects. Since an exothermic effect releases a considerable amount of gas, in order to avoid destroying the crucible, it is practiced to use crucibles with perforated lids or, in some cases, crucibles without lids. Closed crucibles are used for highly volatile substances, to prevent evaporation. The thermal effects that occur during heating of the sample appear as deviations from the baseline and are dependent on the amount of energy supplied to the sample, which may be lower or higher compared to the energy supplied to the reference material. From the evaluation of the exothermic and endothermic peak areas, the values corresponding to the heat of reaction or the physical transition involved are obtained [87, 88].

The DSC curve allows not only the characterization of a physical or chemical process as exothermic or endothermic, but also the identification of the types of transitions that occur in the sample. If several peaks, endotherms or exotherms, appear on the diagram, they are related to the endotherm peak that corresponds to the melting of the substance, if it is crystalline. When there is an overlap of peaks, subsequent experiments must be carried out under conditions in which either the sample mass or the heating rate are modified, in order to achieve a good resolution of them. By using increasingly lower heating rates, a better reproducibility of the signal is obtained. With increasing heating rate, the exotherm peak and its starting temperature move towards higher temperature values, while the endotherm peak remains at the same value.

From the DSC diagrams, it is possible to evaluate: the starting temperature of the process and the temperature of the minimum of the endotherm peak, respectively of the maximum of the exotherm peak. From the evaluation of the peak areas and by using calibration curves, the heats of

reaction are obtained. The evaluation of the characteristic temperatures and heats of reaction is done by deconvolution of the exothermic curve with the decomposition into the component stages of the overall process, considering for each stage a certain kinetic model and thus determining the kinetic parameters (pre-exponential factors, reaction orders, activation energies) [8, 12].

In the present work the DSC method was used to determine the thermal degradation of copolymers used as viscosity modifiers for SAE 10W oil.

Materials and methods

The ethylene-co-propylene and hydrogenated isoprene-co-styrene copolymers used being elastomers, the DSC method was used for thermal decomposition and the DSC diagram in air atmosphere, which was recorded over the temperature range 40-600ºC using the CAHN DSC 550 apparatus, uses a lower heating rate of 10ºC/min.

Fig 2: Differential scanning calorimetry CAHN DSC 550

Results and discussions

The mechanism of degradation of the ethylene-co-propylene copolymer:

1. In the temperature range 0-270^{0}C the copolymer is thermally stable, so there is no danger of its degradation at the operating temperature of an engine.
2. Between 252 and 380ºC degradation occurs with the elimination of propylene.
3. Between 380 and 470ºC degradation continues with the elimination of ethylene.

The following composition can be approximated for the copolymer: about 45% propylene and about 55% ethylene.

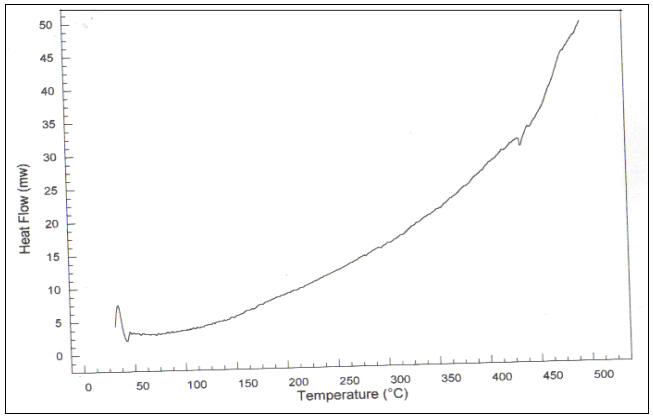

Fig 3: DSC diagram in non-isothermal mode of the ethylene-co-propylene copolymer recorded with the CAHN DSC 550 apparatus in the temperature range 40-600ºC

The DSC diagram recorded over the temperature range 40-600ºC, represented in figure 3, indicates a slight shift of the temperature range over which the reaction occurs towards values lower by about 10ºC than that obtained from the thermogram, explainable by the fact that the heating rate was lower (10ºC/min).

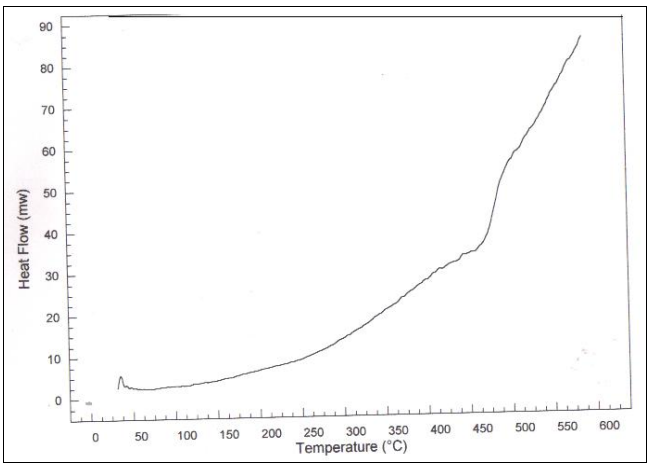

Fig 4: DSC diagram of hydrogenated isoprene-co-styrene copolymer over the temperature range 40-600ºC

The initial decomposition temperature of hydrogenated isoprene-co-styrene copolymer is 231ºC, which means that although it is less thermally stable, it is still stable enough to be used as an additive for improving the viscosity index. The mass losses thus increase with increasing temperature: 231-275ºC – 2.23%, 275-400ºC – 92.77% and residue – 5%.

Conclusions

Taking into account the mass losses in figure 3 – 42.78, respectively 52.16% - the following composition can be approximated for the ethylene-co-propylene copolymer: about 45% propylene and about 55% ethylene.

The initial decomposition temperature of the hydrogenated isoprene-co-styrene copolymer being 252ºC for the first and 231ºC for the second, and the maximum operating temperature of an engine is, under normal conditions, 100ºC, and under severe conditions 150ºC).

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