

ATACTIC POLYPROPYLENE THERMAL DEGRADATION TEST

A. Bukowski, B. Osowiecka and J. Zieliński

Institute of Chemistry, Technical University of Warsaw, Branch at Płock, Poland

Abstract

Atactic polypropylene was subject to thermal treatment the temperature range 200–260°C for 2–4 h. Polymer structure changes assessment was made by derivatograph.

Keywords: atactic polypropylene, polymer, thermal degradation

Introduction

Atactic polypropylene from polypropylene plant in Petrochemia Płock has relatively high molecular weight reaching several ten thousand what in some cases is a restriction of its application. For this reason the methods of its thermal treatment become more and more interesting. They are carried out by degradation and destruction of the polymer to obtain product of decreased molecular weight good as additive to lubricating oils, greases and waxes production.

Most of research works carried out on atactic polypropylene till now, focus on using thus by-product as an additive for bitumen [1] or lube oils [2]. Much less attention was given to the problems related to process of controlled degradation to obtain e.g. high calorific gas and fuel oil.

Experimental

Examinations of atactic polypropylene samples degradation under inert gas blanket were carried out at temperatures 200–260°C. Losses of weight of polymer were determined for qualitative and quantitative evaluation of changes which occur in the process. Process parameters were selected based on derivatographic analysis of polymer designed for test.

Examined were samples of atactic polymer forming as a by-product during industrial production of isotactic polypropylene J-400. It is a caoutchouc-like

solid containing some quantities of *n*-hexan and solvent oil. Its physicochemical properties are shown in Table 1.

Table 1 Properties of atactic polypropylene

Average molecular weight	23 000
Density/kg·m ⁻³	840
Volatile content/wt%	15.5
Ash content/wt%	0.04
Drop point/°C	123

Thermal degradation was carried out under nitrogen blanket in the laboratory glass reactor, electrically heated, equipped with a cooler, thermometers and receivers for volatile products. Samples of polymer had a weight about 5×10^{-2} kg and were weighed with 1×10^{-7} kg accuracy. Specimens were crumbled to flakes.

Assuming, that the process of degradation depends first of all on time and temperature, the examinations were carried out at: 200, 220, 240, 260°C for 2, 3, 4, 5 and 6 h respectively. Other parameters, like inert gas flow rate, heating-

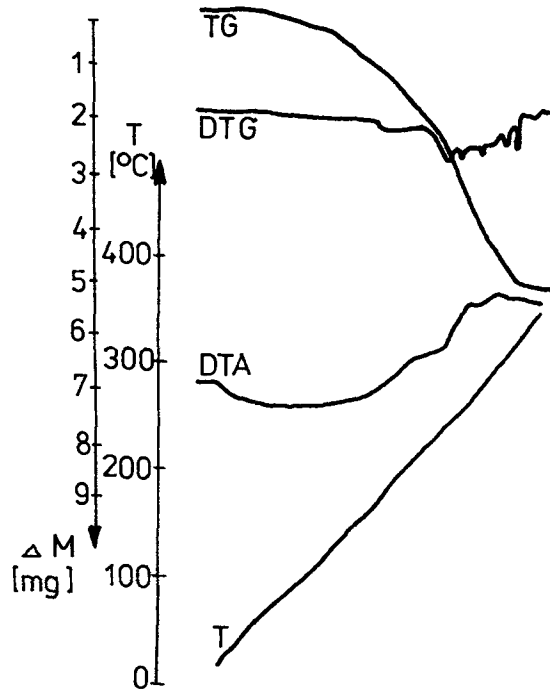


Fig. 1 TG, DTG and DTA curves of atactic polypropylene; heating rate: $5 \text{ deg} \cdot \text{min}^{-1}$; movement of band rate: 2 mm/min ; temperature range to 500°C

up rate were constant for all experiments. Having completed each test, change of colour and consistence were examined and loss of weight was determined.

Derivatographic analysis results are shown on the Figs 1 and 2. These analyses enabled determination of characteristic temperatures of structural and chemical changes occurring during heating-up of the sample. No essential changes occur up to temperature 50°C (curve TG). The evaporation of contamination like hexan and solvent oil is minimal. Shape of curve DTA gives evidence that endothermic processes run. As temperature rises in the range of 50–175°C distinctive change of the TG shape curve is noticed.

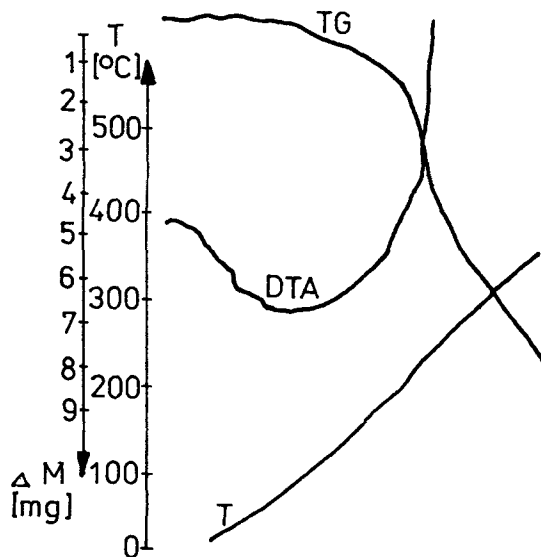


Fig. 2 TG and DTG curves of atactic polypropylene; heating rate: 10 deg·min⁻¹; movement of band rate: 5 mm/min; temperature range to 500°C

This is an indication that polymer melts and evaporation continues. In the range of 170–210°C degradation processes predominate over evaporation and reaction switches to exothermic one. Processes running over 300°C are exothermic which testify further polymer degradation.

Polymer sample weight changes (Figs 3 and 4) show that in the tested range of temperatures contamination evaporation and degradation occur. Losses of weight caused by evaporation amount to 19 wt%.

Heating-up atactic polypropylene at 260°C for 6 h leads to considerable reduction of average molecular weight.

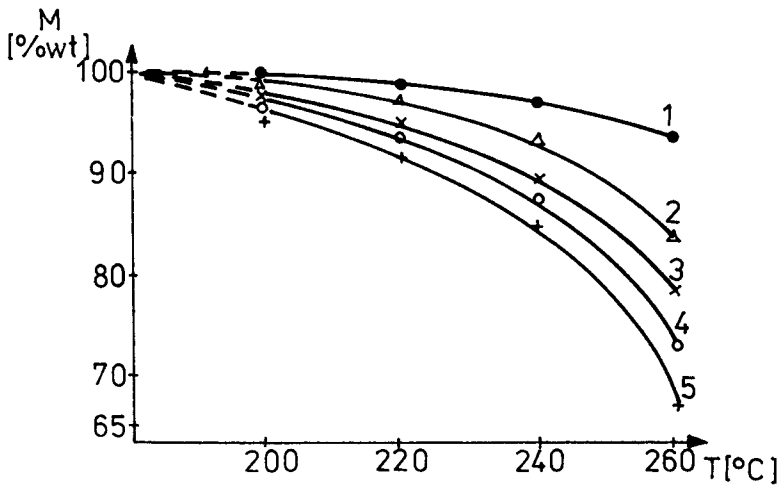


Fig. 3 Relative loss of atactic polypropylene sample weight as a function of temperature: 1. degradation time 2 h; 2. degradation time 3 h; 3. degradation time 4 h; 4. degradation time 5 h; 5. degradation time 6 h; M [wt%] - relative loss of polymer weight

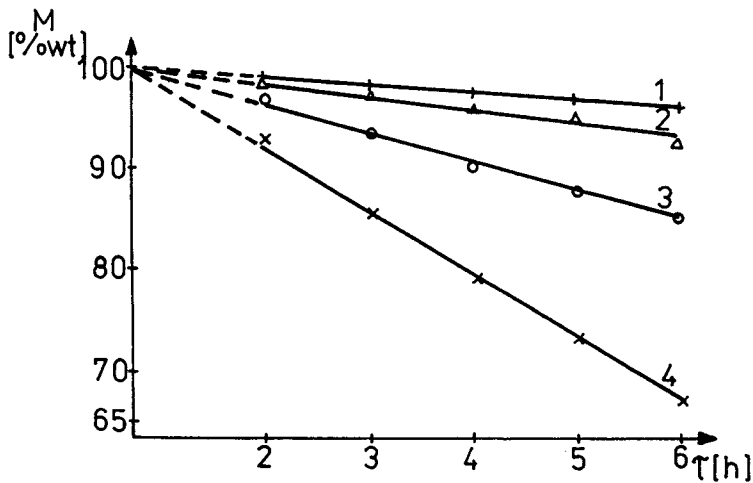


Fig. 4 Relative loss of atactic polypropylene sample weight as a function of degradation time: 1. degradation temp. 200°C; 2. degradation temp. 220°C; 3. degradation temp. 240°C; 4. degradation temp. 260°C; M [wt%] - relative loss of polymer weight

Conclusion

Atactic polypropylene when heated-up at temperature 240–260°C for 6 h undergo thermal degradation. Triple decrease of molecular weight is possible under these conditions. Inert gas atmosphere prevents disadvantageous oxidation

reactions. When samples of atactic polypropylene are subject to thermal treatment, presence of considerable quantities of *n*-hexan and solvent oil should be taken into consideration. They are completely separated out at the temperature 220–240°C.

References

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- 2 T. Milczarska, A. Bukowski, *Przemysł Chemiczny* 55 (1975) 1, 21.

Zusammenfassung — Ataktisches Polypropylen wurde für 2–4 Stunden einer Wärmebehandlung im Temperaturbereich 200–260°C unterzogen. Mittels Derivatograph wurden Strukturänderungen des Polymers abgeschätzt.