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Progressive Deterioration of Thermal Stability of Nanofilled Polypropylene

T. Peprnicek^a; J. Simonik^a; J. Achillesova^a; I. Kuritka^b; P. Saha^b; J. Plestil^c; E. Pavlova^c

^a Department of Polymer Engineering, Tomas Bata University in Zlin, Zlin, Czech Republic ^b Polymer Centre, Tomas Bata University in Zlin, Zlin, Czech Republic ^c Institute of Macromolecular Chemistry, Academy of Sciences of the Czech Republic, Prague, Czech Republic

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Progressive Deterioration of Thermal Stability of Nanofilled Polypropylene

T. Peprnicek, J. Simonik, and J. Achillesova

Department of Polymer Engineering, Tomas Bata University in Zlin,
Zlin, Czech Republic

I. Kuritka and P. Saha

Polymer Centre, Tomas Bata University in Zlin, Zlin, Czech Republic

J. Plestil and E. Pavlova

Institute of Macromolecular Chemistry, Academy of Sciences of the Czech
Republic, Prague, Czech Republic

Abstract: This article concentrates on polypropylene stability from the point of structural and thermal characterization of PP/nanocomposites through mechanical testing, X-ray diffraction, transmission electron microscopy (TEM), and thermogravimetric analysis. The composites were prepared from powdered polypropylene (PP), stabilizer, and nano-clays both natural and organophilic using a twin-screw extruder. The results showed that the type of nano-clay affects the morphology and mechanical and thermal properties of the composite, and not only in the expected way. An extreme decrease of PP durability under isothermal load was observed. This article is presented as the first part of a PP/nanocomposites degradation investigation.

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Address correspondence to T. Peprnicek, Department of Polymer Engineering, Tomas Bata University in Zlin, Nam. T. G. Masaryka 275, Zlin 762 72, Czech Republic. E-mail: peprnicek@ft.utb.cz

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INTRODUCTION

Since the initial success of polyamide nanocomposites,^[1,2] polymer layered silicate nanocomposites (PLSN) have attracted great interest from both academic and industrial scientists. Nanocomposites are considered to be the materials of the next generation and many articles dealing with PLSN can be found in the literature. There are several polymeric matrices suitable for PLSN production; among them the following are commonly used: polyamides-PA 6, 11, 12,^[3,4] polyethylene (PE),^[5] polyvinylchloride (PVC),^[6] and poly(methyl-methacrylate) (PMMA).^[7] One of the most frequently chosen polymeric matrices is polypropylene (PP).^[8–11] PP-based plastics are of inferior quality in comparison with PE. Generally, their mechanical properties are improved by compounding with nanofillers, especially in PLSN.^[12] It is demonstrated in this article that PP nanocomposites have been developed and improved mechanical properties have been obtained in the case of the exfoliated or, at least, well-intercalated structure of PLSN.

Processes to improve nanostructured morphology are thought to be, for example, the enthalpic interaction balance between layered silicates and matrix or entropic effects.^[12] Furthermore, molecular weight^[11] and chemical modification of the clay also affect the morphology of PLSN. Well-ordered nanostructures can be easily obtained by intercalation from solution or in situ intercalation.^[12] Nevertheless, in the case of PP, these methods cannot be used. Generally, the melt intercalation method is applicable for PP but several other techniques have been developed.^[13,14] Because of the absence of organic solvents and thanks to the compatibility of this method with current industrial processes such as extrusion or injection molding,^[15,16] it is presented as versatile, environmentally friendly, and cost effective.^[17,18] A disadvantage of PP usage in the PLSN area is its nonpolar characteristic, in contrast to the polar nature of clay, which keeps its miscibility weak. Consequently, a blend of PP and maleinized PP (PP-g-MA) is used. PP-g-MA has been introduced to improve the compatibility between the polymeric matrix and clay nano-filler. PP-g-MA compatibilizer helps to bond the clay and PP matrix together due to the hydrogen bond between the polar carboxylic group of PP-g-MA and the oxygen or hydroxyl group in silicates.^[11, 19–21]

The thermal stability of PP/clay nanocomposites has been studied,^[9,10] but, generally, it represents an unsolved problem; see Pandey et al.^[22] and references therein. As far as we know, the thermal stability

of PP/nanocomposites has not been investigated by thermogravimetry (TG) in iso- and non-isothermal.

In this article, seemingly conflicting results obtained by TG investigation of PP/nano-clay composites are explained. They demonstrate the importance of isothermal testing in thermal stability data evaluation. Furthermore, inert atmosphere used in the experiments allows the examination of thermal degradation without the influence of oxygen. Consequently, the primary degradation processes dominate. The results of thermal study are corroborated with the prepared composite morphology measurements, i.e., X-ray and TEM analysis and with tensile strength tests.

EXPERIMENTAL SECTION

Materials

In this work, montmorillonite (MMT) obtained from Southern Clay Products (Cloisite[®]) and Süd-Chemie (Nanofil[®]) was used as nano-filler. Its silicate layers are approximately 200 nm long and 1 nm thick, and the interlayer spacing between stacked layers, denoted d_{001} , is about 1 nm.^[23,24] Two different types of MMT were used: natural type, labeled Cloisite[®] Na⁺, without chemical treatment, cation exchange capacity (CEC, determined by titration by using an ammonium acetate method) 92.6 meq/100 g clay,^[25] which displays a polar character, and two organophilic MMTs, denoted Nanofil[®] 5 and Nanofil[®] SE 3000, which are purpose-modified with distearyldimethyl-ammonium chloride for use in polyolefin matrices.^[26] Powdered polypropylene (PP) (Moplen HF 500 N, Basell, The Netherlands), which is distributed without stabilizing agents, was chosen as a polymeric matrix. Consequently, Irganox B 225 obtained from Ciba, (Switzerland) was used as a polypropylene stabilizer. The PP-g-MA, Excelor 1015, was applied as the compatibilizer between PP matrix and the clay.

Processing of Nanocomposites

For the preparation of PP/nanocomposites, a specific method was used. First, PP powder with a proper amount of 0.1 wt% of stabilizer was mixed. Clay in the amount of 2, 5, and 7 wt% was added into the prepared mixture and homogenized using a laboratory mixer. The last step of preparation was adding of PP-g-MA, in the shape of pallets. Final blend of powders and PP-g-MA pellets was processed in a corotating twin-screw extruder, type MP 19–25 TC from AVP Baker, melted down,

Table I. Samples and their labels

Sample	Matrix	Nano-clay	Clay content [% wt]
Neat PP	PP	—	0
Na ⁺	PP	Cloisite [®] Na ⁺	7
Nano5	PP	Nanofil [®] 5	7
SE3000	PP	Nanofil [®] SE 3000	7

and finally pelletized. The temperatures alongside the twin-screw extruder varied from 170°C (under the hopper) to 190°C (die temperature). The screw speed was 350 rpm and the melt temperature was 200°C. Neat PP samples were prepared under the same conditions as composite materials to obtain a proper reference polymeric material with the same thermal and flow history.

In the following, the nanocomposites are labeled according to the type of a nano-filler. Main characteristics of the samples are given in Table I.

Mechanical Tests

Mechanical properties were tested using an Instron 8871. Tensile data were obtained at the speed of the moving clamp of 100 mm/min in a static testing mode. Testing was carried out according to test standard CSN EN ISO 527-1, 527-2.

X-Ray Diffraction Measurement

X-ray diffraction (XRD) patterns of the samples were obtained using an HZG 4 diffractometer, from Freiberg Präzisionsmechanik, with back monochromator and Cu anticathode (step 0.01°, step time 20 s). Special attention was paid to the low 2θ region, from 1.4 up to 10 $2\theta^\circ$, for the accurate determination of d_{001} , for the nano-clay denoted as the d-spacing. Original fillers were analyzed by the use of another device of exactly the same type with the same test conditions (except the minimal 2θ angle set) as powder samples.

TEM Analysis

Transmission electron microscopy (TEM), was performed on a JEM 200CX (JEOL) device at 100 kV. Ultra-thin cross sections prepared on a special cutting device, Leica Ultracut Uct ultracyromicrotome, at the

sample temperature of -110°C and the knife temperature of -50°C . The TEM scans were digitized by means of a computer-controlled digital camera DXM1200 (Nikon).

TGA Analysis

The thermal behavior of the samples was studied by using a thermogravimeter, Setaram SETSYS Evolution 1200. The samples were examined under inert atmosphere of He (5.5 purity, SIAD TP); the gas flow was $30\text{ cm}^3/\text{min}$ at a pressure of 101 kPa for all experiments. A platinum crucible was used for sample weights of about 8 mg. Temperature growth rate was $20^{\circ}\text{C}/\text{min}$ in the range from the ambient temperature up to 1000°C for non-isothermal analysis. An arbitrary temperature of 426°C was chosen for isothermal testing of material stability. It means that each sample was heated at a temperature heating rate of $20^{\circ}\text{C}/\text{min}$ until the temperature reached 426°C . Then an isothermal period took several hours. The temperature control ensured programmed temperature overshooting of only about 0.5°C during temperature stabilization.

RESULTS AND DISCUSSION

Mechanical Testing

All the data from mechanical testing are summarized in Table II, where σ_y and σ_f are tensile strength at yield and failure and δ is standard deviation. The results show that the improvements in mechanical characteristics, modulus E , are in tenths of percent and are not significant. As suggested, the value of tensile modulus increases with clay loading. The highest growth was obtained for the PP matrix Nano5. The increase reached almost 20% in comparison with the pure PP matrix. Odd filled matrices exhibit improvements of 10% and 15% for filler SE 3000 and Na^+ ,

Table II. Mechanical properties of the prepared materials and relative standard deviations δ of the respective values

Sample	E [Mpa]	δ [%]	σ_y [Mpa]	δ [%]	σ_f [Mpa]	δ [%]	Δl [mm]	δ [%]
Neat PP	803	7	38	4	27	15	8.6	64
Na^+	929	9	40	8	35	7	3.6	9
Nano5	954	10	36	3	30	7	4.7	35
SE3000	887	6	38	3	22	21	7.8	50

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respectively. In contrast to the previous findings, different results were found in the case of tensile elongation at failure, Δl . It is known that the filled matrices possess lower value of tensile elongation at failure, caused by the filler addition into the system, which, consequently, loses its elasticity. Accordingly, the sample denoted as SE3000 had the best value, with 90% of elongation at failure, compared with the pure PP matrix. Remaining samples exhibit lower magnitude of elongation at failure: Nano5 54% and Na^+ 42%, respectively. Similar data were found for other concentrations of the fillers. High stiffening effect obtained for filler Na^+ is a surprising result. It is thought that chemically treated clays should provide better results than non-treated Na^+ s, but this was not the case.

X-Ray Diffraction

The X-ray diffraction (XRD) measurements are used as a nondestructive method allowing insight into the nanocomposite structure. The diffraction patterns for original nanofillers are shown in the upper window in Figure 1 together with diffraction patterns of prepared nanocomposites in the lower window in Figure 1. The filler Cloisite[®] Na^+ shows only one peak in its XRD spectrum at $7.2 \ 2\Theta$ degrees. No intercalation peak appeared in the XRD spectrum of material Na^+ below $7 \ 2\Theta$ degrees, and only one peak typical for source material can be found at $7.8 \ 2\Theta$ degrees shifted to higher diffraction angles. This may be explained by the lack of interaction between the polymer matrix and the clay. However, the broadening of the peak and/or the decrease of its relative intensity indicates some structural change that may be affected by a loss of water during compounding at the elevated temperature. This explanation is in agreement with the shift of peak maximum to higher 2Θ value, which means that the distance between silicate layers of the filler is smaller.

Nanocomposite materials with organophilized clays display a minor or major level of intercalation/exfoliation. Nanocomposite sample SE3000 possesses two high well-resolved peaks in the XRD spectrum lying in region typical for intercalated Cloisite[®]. The first peak is of low intensity and it is centered at about $7.1 \ 2\Theta$ degrees, the second peak is centered at $4.9 \ 2\Theta$ degrees, and the third one is at around $2.7 \ 2\Theta$ degrees (lower graph in Figure 1). The first two peaks show only moderate changes in comparison with the peaks at 4.7 and $7.1 \ 2\Theta$ degrees present in the original SE filler (upper graph in Figure 1). Thus, the nanofiller SE 3000 inside the polymeric matrix keeps not only a high level of intercalation by molecules of the modifier but also is intercalated by the polymer. Well-developed sharp peaks indicate that the filler is only moderately disaggregated and that almost no exfoliation can be expected. The sample denoted as Nano5 has better degree of exfoliation. Three wide

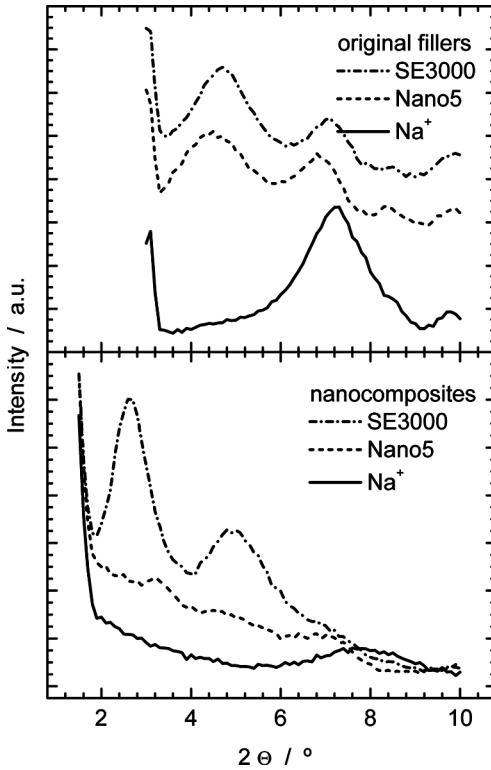


Figure 1. X-ray diffraction plot of nanocomposites.

and low-intensity peaks are observable in the XRD spectrum. Intercalation by the polymer, disaggregation of the layered structure, and, in addition, a certain level of exfoliation can be predicted, as peaks at 6.8 and 4.5 2θ degree almost vanish after incorporation into the composite. The material Nano5 exhibited the best morphological state determined by XRD, which is further confirmed by TEM.

TEM Analysis

TEM photographs in Figures 2, 3, and 4 show the so-called true morphological state of the nanocomposites. On the other hand, one should take into account that only a small area can be observed by TEM.

The sample denoted as Na⁺ with nano-filler without chemical modification possesses the worst level of intercalation/exfoliation (see Figure 2). In this sample, big agglomerates around 2 μm can be found and almost no intercalation can be seen. This observation from the TEM

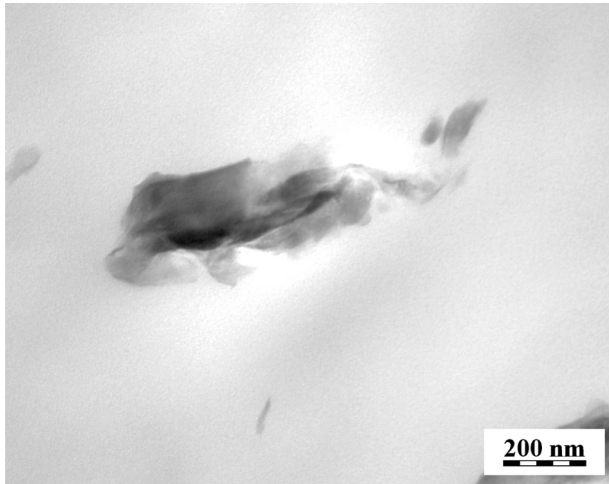


Figure 2. TEM photo of Na^+ nanocomposite.

photograph is in contrast to the results from mechanical testing where these samples do not display such a weak interaction between the polymer matrix and nano-clay. Nevertheless, the result from TEM is supported by XRD measurements. The nano-clay Na^+ is distributed in the polymeric matrix only in agglomerates without any intercalation or exfoliation. As a result, only the peak that belongs to the pure Cloisite[®] Na^+ appeared (see Figure 1).

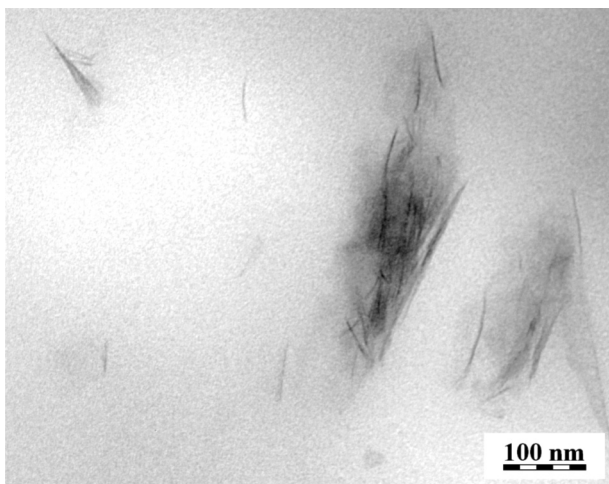


Figure 3. TEM photo of Nano5.

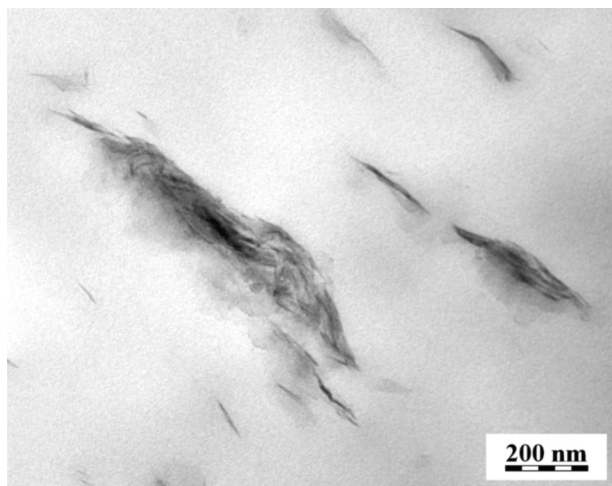


Figure 4. TEM photo of SE3000 sample.

The next two samples with chemically modified clay show a much better level of intercalation and exfoliation. For both nanocomposites, a high level of exfoliation, where lamellas are distributed in the polymer matrix with a few agglomerates of 200 nm, can be found. The TEM photos agree with XRD results where a better level of intercalation/exfoliation was thought to appear for the sample labelled Nano5 (see Figure 3). For this sample, smaller agglomerates with better clay layer separation are seen. In addition, the intercalated state is also found in these agglomerates. In the case of the sample denoted SE3000, bigger agglomerates were observed (Figure 4). However, the agglomerates are intercalated and, moreover, some exfoliated lamellas can also be found in this case.

The obtained data support the presumption that intercalation and exfoliation can easily be reached by using proper chemically treated clays and the modifier PP-g-MA as well. Furthermore, this behavior may be predicted from mechanical testing where the nanocomposites with modified clays possess increased modulus in comparison with the pure PP matrix.

TGA Analysis

No consensus about an improvement or decrease of thermal stability is reported for PP/clay nanocomposites in inert atmospheres (see Bertini et al.^[27] and references therein). Such a confusion might be caused by

an established use of inappropriate evaluation procedures. Hence, the prepared samples were tested under both non-isothermal and isothermal mode in order to clarify this question.

The results of non-isothermal TGA experiments are shown in Figure 5. The lower graph window shows the weight loss in dependence on temperature, and the upper graph shows the weight loss rate as a function of temperature. The inflection points on integral TGA curves correspond with the minima marked in the upper graph. Table III summarizes the evaluation of thermal stability based on various temperature criteria applied for non-isothermal TGA experiments. The temperature corresponding to the initial 2% ($T_{0.02}$), 5% ($T_{0.05}$), and 10% ($T_{0.10}$) weight loss, and the temperature of maximum weight loss rate (T_{inflex}) were chosen to test the reliability of temperature onset evaluation. Obviously, the use of each thermal stability criterion leads to different ranking of material stability. The most explicit example is the composite material Nano5, whose stability varies from the best to the worst quality (from

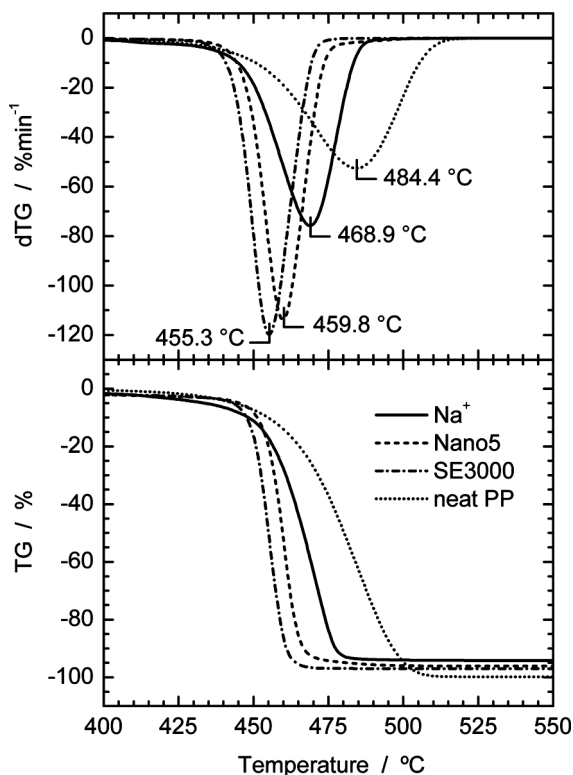


Figure 5. Non-isothermal analysis of prepared materials; for description see text.

Table III. Thermal stability evaluation based on non-isothermal TGA

Sample	Degradation temperature [°C]							
	$T_{0.02}$		$T_{0.05}$		$T_{0.10}$		T_{inflex}	
Na ⁺	407.4	2.	433.4	4.	448.4	3.	468.9	2.
Nano5	394.1	4.	445.4	1.	451.2	2.	459.8	3.
SE3000	396.8	3.	443.1	3.	447.6	4.	455.3	4.
Neat PP	427.1	1.	443.8	2.	454.9	1.	484.4	1.

Values of degradation temperature evaluated according to four criteria (see text) are presented with decimal numbers; the stability order within the column is marked by the bold numerals.

first to fourth order according to thermal stability) depending on the choice of evaluation criterion. Therefore, the thermal stability assessments based on non-isothermal criteria must be handled with caution, especially the evaluation using only a single TGA curve recorded at one heating rate for each nanocomposite material.

The TGA curve describes the weight loss; thus only the final stage of material degradation connected with the volatilization of the degraded polymer matrix and, at a certain level, the filler calcination is observable while previous phases of thermal degradation remain virtually invisible. As various processes may influence the shape of the TGA curve, the onset temperature evaluation is not unambiguous. Finally, the use of the isothermal tests sheds light on the problem and gives a reliable ranking of stability, as exemplified in Figure 6. The upper graph window shows the full time scale of the isothermal experiment, and the lower graph window zooms on the area marked in the upper graph by the narrow rectangle. The dashed arrow marks the start of the isotherm after reaching 426°C. The temperature of 426°C was chosen from the interval given by the extremes in onset temperatures to allow a comparison of the stabilities of the prepared materials. At this temperature, the degradation rate is the highest for the sample of neat PP (about 2% min⁻¹), and degradation rates of nanocomposites are below 1% min⁻¹. Surprisingly, the most stable material is unmodified PP compound, which is fully degraded after 2000 s. The Na⁺ composite degrades in approximately 300 s, and the nanocomposites appeared to be the least stable materials under the isothermal condition test, as they showed degradation span within 150 to 200 s. Once the material starts to evolve gaseous products of degradation, the deterioration proceeds much faster in the presence of nano-clay than it does without the filler. The sample denoted SE3000 appeared to be the less stable sample, with total degradation time more than one order of magnitude smaller than that of neat PP. These

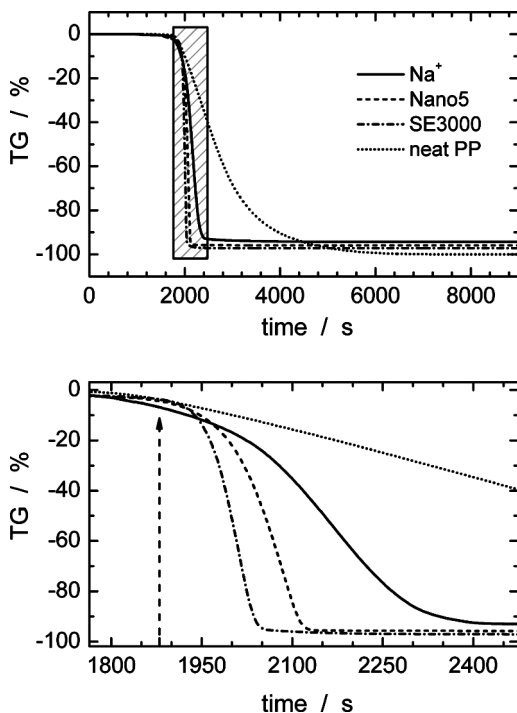


Figure 6. Isothermal analysis of prepared materials.

results correlate well only with stability evaluation based on T_{inflex} , but this quantity cannot be accepted as a sufficient criterion for thermal stability assessment without further consideration.

CONCLUSIONS

Our study reveals the importance of isothermal stability tests of the examined PP/nano-clay nanocomposites that were successfully prepared. Nano5 material exhibited improved mechanical properties but all nano- and composite materials had markedly lower thermal stability than the neat polymer. The progressive deterioration of the nanocomposite material was observed under the isothermal load. However, the use of commercially available sophisticated nanofillers, which leads to formation of exfoliated nanocomposite structures, resulted in materials with lower thermal stability. Therefore, increased attention has to be paid to the thermal stability improvements claimed only by the evaluation of onset temperature obtained by non-isothermal analysis. Isothermal experiments must be carried out to properly evaluate the thermal stability

of the prepared materials. Further studies are required to describe kinetics and explain the origin of observed phenomena in examined PP/nano-clay materials. A new approach to stabilization of PP in clay nanocomposites is needed to make them viable for potential applications.

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