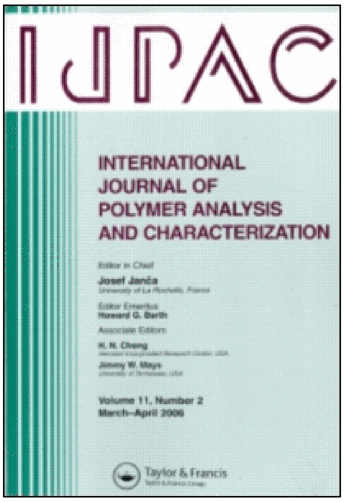

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ANIONIC NYLON 6/ZINC COMPOSITE MATERIALS: EVALUATION OF THERMAL AND MECHANICAL BEHAVIOR

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This study deals with the experimentally investigated effect of zinc (Zn) particles on the thermal and mechanical properties of nylon 6/zinc composite materials. Nylon 6 and nylon 6 containing 6.0, 8.0, 12.0, 15.0, 20.0, and 25.0 wt.% of unmodified zinc particles, metal-polymer composite materials, were prepared by in situ anionic polymerization of ϵ-caprolactam (CL) in the presence of metallic fillers using rotational molding technique. The characteristics, namely melting temperature, degree of crystallinity (first and second heating scan) and crystallization temperature, crystalline structure, thermal stability, and mechanical properties (flexural modulus, flexural strength, and notched Izod impact strength), of the nylon 6/zinc composites were determined for each sample. Different DSC, ATG, wide-angle X-ray diffraction, and mechanical measurements were performed. The results related to nylon 6 composites were compared with those obtained for pure nylon 6.

Keywords: Anionic nylon 6; Mechanical properties; Nylon 6/zinc composites; Thermal analysis

INTRODUCTION

The incorporation of inorganic particulate fillers into polymers has long been an interesting topic of scientific research and industrial applications due to its potential to promote improvement in some physical properties of the resultant polymer composites such as stiffness, modulus, mechanical strength, electrical and thermal conductivity, temperature performance, and dimensional stability.[1–3]

Aliphatic polyamides, also known under the generic name nylons, are very versatile engineering thermoplastics that encompass a broad range of uses in many fields such as textile fibers, packaging, implants, membranes, biosensors, and automotive, electrical/electronic, and consumer applications.[4–8] The attractive properties and the semicrystalline character of nylons stem from intra- and interchain hydrogen bonds constituted between amide groups separated by methylene repeated unit with variable lengths. The hydrogen bonds formed intra-sheet between anticlined, extended chains and those binding inter-sheet isoclined, pleated chains are specific to α- and γ-crystalline phase, respectively.[8,9] The γ-crystalline

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form seems to be favored by changing of the homogeneity in the structure of nylons as a result of slow crystallization, heating, copolymerization, or incorporation of other compounds (i.e., inorganic fillers). Among various aliphatic polyamide matrices, nylon 6 has been one of the best choices for preparation of various types of composite materials.[10]

Nylon 6 can be synthesized by ring-opening polymerization of ϵ-caprolactam via hydrolytic and anionic polymerization.[11] Through hydrolytic polymerization, nylon 6 is conventionally produced by heating at much higher temperature of 250°C, under pressure with a catalytic amount of water (5–10%) and nylon salt or aminocaproic acid as an initiator for about 12–24 h.[11] In contrast, anionic polymerization of ϵ-caprolactam can be carried out at the rather low temperature of 150°C in the presence of a strong base as initiator. At the same time, an activator is usually added to the ϵ-caprolactam to speed up the activation step, and therefore the anionic polymerization is much faster than the hydrolytic reaction. The hydrolytic polymerization of ϵ-caprolactam is the most important for commercial production of nylon 6, while high molecular weight polymer with high degree of crystallinity can be obtained via anionic polymerization.[12] It is well known that nylon 6 prepared from anionic ring-opening polymerization of ϵ-caprolactam exhibits both desirable properties (high strength, stiffness, and resistance to nonpolar solvents) due to a high degree of crystallinity,[12] and undesirable properties (poor low-temperature toughness, high moisture sorption, dimensional instability).[8,13–15] Many deficiencies of nylons can be overcome by formulation of the system containing metal particulate as fillers. As a matter of fact, it is well known that composite materials based on polymer and metal filler show improved physical and mechanical properties. The first include enhanced thermal, electrical, and dielectric properties. The type and the volume fraction of the metal fillers, as well as the interfacial compatibility between the metal particles and the polymer matrix, customize the physical and mechanical properties of the composite materials.[16–18]

The preparation of nylon 6/metal composites can be performed by solvent processing, melt-compounding, or in situ polymerization.[19–24] The last method was applied to preparation of the nylon 6/zinc composite materials; it involves the dispersion of metallic particles in monomer followed by the polymerization process.

In this study, nylon-6 and nylon-6/zinc composite materials were prepared in situ via anionic ring-opening polymerization of ϵ-caprolactam (CL) in the presence of zinc (Zn) filler at various levels of loading (6.0–25.0 wt.%) by rotational molding technique. Their physical properties were investigated in terms of thermal and mechanical characteristics. The thermal behavior of the nylon 6 and nylon 6 composites was investigated by differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). Crystal structures of the pure nylon 6 and nylon 6/zinc composites were examined by means of wide-angle X-ray diffraction (WAXD) as a function of Zn content. The mechanical properties of the anionic nylon 6 and its composite materials evaluated in this study are notched Izod impact strengths and flexural properties as well as flexural strength and flexural modulus.

The experimental results were correlated with the initial content of metal fillers and literature data and are reported herein.

EXPERIMENTAL SECTION

Materials

ε-Caprolactam (CL) monomer (technical grade, Sigma-Aldrich Chem. Gmbh), purified according to the literature,[8,24–27] and the metallic filler, zinc (Sigma-Aldrich Chem. Gmbh), in the form of fine powder, with average particle size of $15 \pm 10\,\mu m$, a density of about $7.14\,g\,cm^{-3}$, and melting point of 419°C were used as starting materials.

The Zn powder was thoroughly dried before use at 80°C in vacuum for 48 h to eliminate possibly absorbed water on the surface of the particles and then was kept in a dessicator before use. The ε-caprolactam magnesium bromide (1.6 mol/kg concentration in ε-caprolactam) and *N,N′*-isophthaloyl-bis-ε-caprolactam obtained in our laboratories according to the procedure described in the literature[8,27] and[26,28,29] were used to initiate and activate, respectively, the polymerization process. All other reagents were of reagent grade and used without further purification.

Synthesis of Anionic Nylon 6 and Nylon 6/Zinc Composite Materials

Anionic nylon 6 and nylon 6/Zn composite materials have been successfully obtained by means of reactive rotational molding process via anionic ring-opening polymerization of CL in the presence of various weight contents of zinc particles (6.0, 8.0, 12.0, 15.0, 20.0, and 25.0 Zn wt.%).

The process was performed in a laboratory-scale rotational molding installation. With respect to CL monomer, the contents of *N,N′*-isophthaloyl-bis-ε-caprolactam as activator and ε-caprolactam magnesium bromide as initiator were kept constant at 0.4 mol.% and 0.6 mol.%, respectively. The initial polymerization temperature was kept constant at 160 ± 2°C for all tests. The details of the synthesis of anionic nylon 6 and nylon 6/Zn composite materials were described in previously published articles.[24,27]

CHARACTERIZATION

Differential Scanning Calorimetry (DSC)

Thermal analyses of nylon 6 and nylon 6/zinc composites were carried out using DSC and TGA. The nonisothermal crystallization and melting behavior of nylon 6 and nylon 6/zinc composite materials (coming directly from the synthesis) was examined using DSC (Metler DSC 12E) under nitrogen atmosphere in order to protect the nylons from degradation. The weight of samples was in the range of 7–10 mg. The measurements were performed for two heating cycles. All samples were first heated from 25° to 250°C, and then cooled to 25°C and finally heated a second time from 25° to 250°C. The heating and cooling rates for all runs were 10°C/min. The characteristics of the nylon 6 matrix in nylon 6/zinc composite materials for melting temperature (T_m), crystallization temperature (T_c). and degree of crystallinity (α_{DSC}) were evaluated from the DSC measurement. Degree of crystallinity (α_{DSC}) (first and second heating scan) of both nylon 6 and nylon 6 matrix was calculated from the following relationship:

$$\alpha_{\mathrm{DSC}} = \frac{\Delta H_m}{(1 - W_f)\Delta H_f^0} \times 100 \tag{1}$$

where $\Delta H_{\mathrm{m}}^{\mathrm{o}}$ is the enthalpy of fusion of the pure crystalline nylon 6. $\Delta H_{\mathrm{m}}^{\mathrm{o}}$ was assumed to be 191 $\mathrm{J\,g^{-1}}$,[8,25,26,29] $\Delta \mathrm{H_m}$ ($\mathrm{J\,g^{-1}}$) is the enthalpy of fusion of the nylon 6 matrix, and $\mathrm{W_f}$ is the weight fraction of the Zn filler in the nylon composites.

Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) was carried out with a F. Paulik Derivatograph Q-1500D thermogravimetric analyzer in an air atmosphere at a flow rate of 30 mL/min and a temperature range of 50°–700°C at scanning rate of 10°C/min. The samples used for this type of analysis were previously subjected to extraction with methanol.

Wide-Angle X-Ray Diffraction

Crystal structure of the pure nylon 6 and nylon 6/zinc composite samples was evaluated by means of wide-angle X-ray diffraction (WAXD) as a function of Zn content, on a PW 1830 Philips diffractometer equipped with nickel-filtered $\mathrm{CuK_\alpha}$ radiation of wavelength 0.1542 nm, in the range of diffraction angles (2θ) from 5 to 45° at a speed of $2^\circ\,\mathrm{min^{-1}}$. The voltage and tube current were 40 kV and 30 mA, respectively.

Mechanical Testing

The mechanical properties of nylon 6 and its composite materials evaluated here are notched Izod impact strengths and flexural properties as well as flexural strength and flexural modulus. The notched Izod impact strength and flexural properties were determined according to ASTM D 256 and ASTM D790-2003, respectively, as previously reported.[25,27] The mechanical measurements on materials under study were performed just after the drying of samples coming from synthesis to avoid water absorption prior to testing. The values reported reflect an average from five measurements.

RESULTS AND DISCUSSION

DSC Analysis

Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) were used to study the thermal properties of nylon 6 and its composites. The melting temperature $\mathrm{T_m}$, degree of crystallinity α_{DSC} (the first and the second heating scan), and crystallization temperature $\mathrm{T_c}$ of the pure nylon 6 and

Table I. Nonisothermal DSC results for the measured nylon 6 and its composite materials

		First heating scan			Cooling scan		Second heating scan			
Sample code	Zinc (wt.%)	T_{m1} (°C)	ΔH_f (Jg^{-1})	α_{DSC} (%)	T_c (°C)	ΔH_c (Jg^{-1})	T_{m2} (°C)	T_{m3} (°C)	ΔH_f (Jg^{-1})	α_{DSC} (%)
PA6/0%Zn	0.0	225.0	95.18	51.6	182	78.38	219.0	212	78.14	36.3
PA6/6%Zn	6.0	225.0	96.14	41.7	179	76.34	220.0	212	75.69	31.5
PA6/8%Zn	8.0	225.0	97.34	40.3	178	77.69	220.5	212	76.92	28.5
PA6/12%Zn	12.0	224.0	94.63	35.1	184	79.67	219.5	213	78.65	26.3
PA6/15%Zn	15.0	222.0	95.21	32.5	186	82.12	219.0	214	82.31	23.5
PA6/20%Zn	20.0	222.0	94.65	21.9	186	83.21	218.0	214	79.65	20.8
PA6/25%Zn	25.0	221.0	98.16	20.5	186	84.36	217.5	214	78.88	18.9

of the composite materials evaluated by DSC at the heating and cooling rate of 10°C/min are shown in Table I and Figures 1–3. The results revealed the effect of the addition of metallic particles on the melting (the first and the second heating scan) and crystallization behavior of the samples under study.

In order to obtain more information about the effect of Zn content on melting and crystallization behavior of nylon 6 and its composites, nonisothermal crystallization analyses were applied to all samples. In comparison with the isothermal crystallization, the conditions used in this technique are much closer to the real processing conditions.

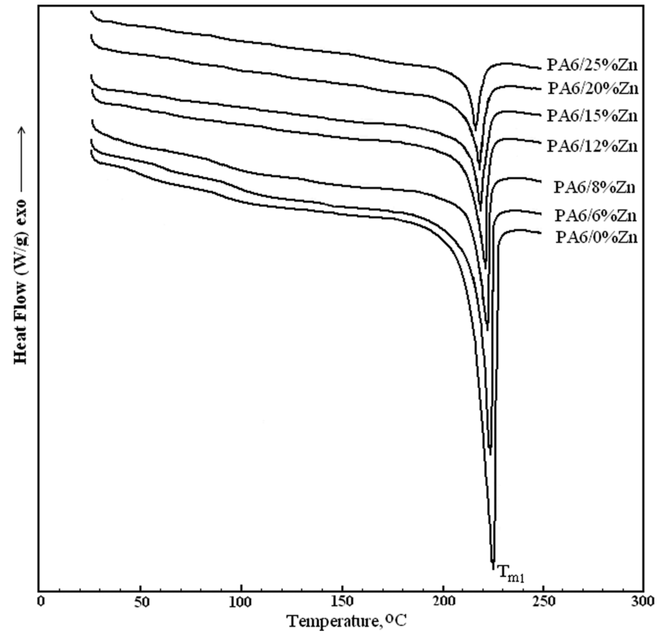

Figure 1. DSC thermograms obtained during the first heating scan for pure nylon 6 and nylon 6/Zn composite materials prepared by rotational molding via anionic ring-opening polymerization of CL in the presence of Zn particles (composition indicated on curves).

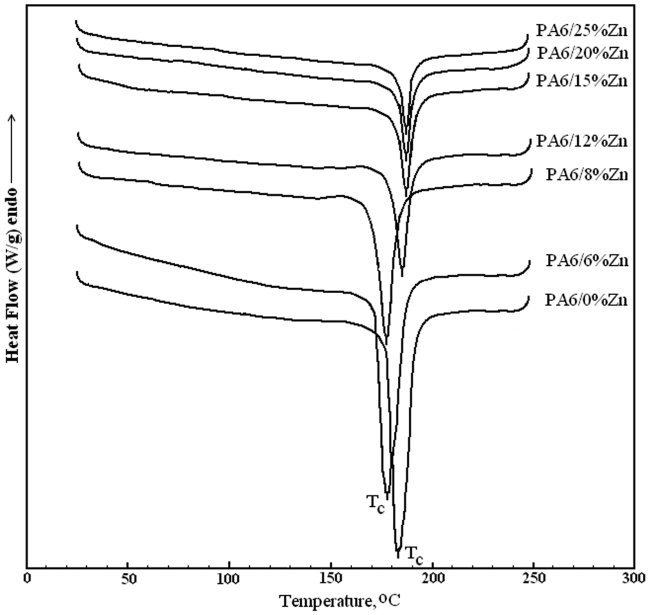

Figure 2. DSC thermograms obtained during the cooling scan for pure nylon 6 and nylon 6/zinc composite materials prepared by rotational molding via anionic ring-opening polymerization of CL in the presence of Zn particles (composition indicated on curves).

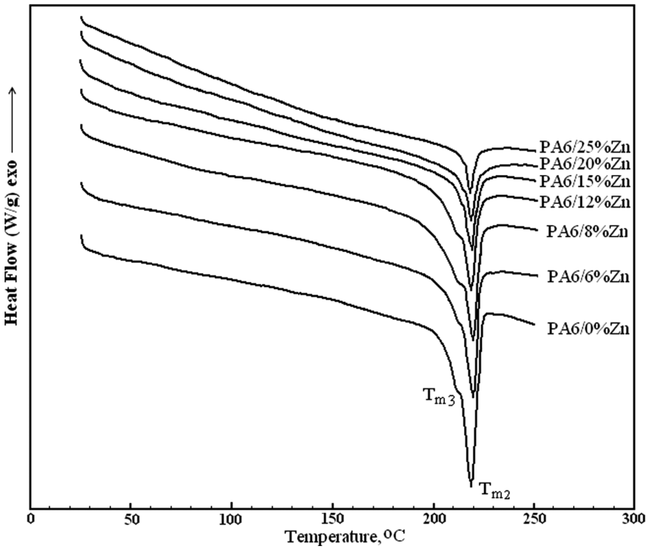

Figure 3. DSC thermograms obtained during the second heating scan for pure nylon 6 and nylon 6/zinc composite materials prepared by rotational molding via anionic ring-opening polymerization of CL in the presence of Zn particles (content indicated on curves).

Figure 1 shows DSC scanning curves (the first heating scan) of nylon 6 and its composite materials. Table I gives some DSC characteristic parameters of nylon 6 and its composite materials.

Melting temperatures (the first and second heating scan, noted as T_{m1}, T_{m2}, and T_{m3}, respectively) of the neat nylon 6 and its composites were taken in correspondence to the enthalpy peak maxima.

As given in Table I and Figure 1, only one endothermic peak occurs in nylon 6 and its composites (the first heating scan), associated to the melting of its α-crystalline phase.[30] The results show that melting temperature (T_{m1}) value for pure nylon 6 is $T_{m1} = 225°C$, corresponding to the melting of its α-crystalline phase.[30]

It can also be seen that melting temperature (the first heating scan) (T_{m1}) of the nylon 6 composite materials remains unchanged to above 225°C with increasing zinc powder content $\leq$8.0 wt.%, after which it begins to decrease very slightly with increasing percentage over 8.0 wt.% powder. The γ-crystalline form was absent in all samples. The shifting of melting temperature (T_{m1}) to lower temperatures upon addition of zinc powder (>8.0 wt.%) can probably be attributed to a decrease in crystallite size in the presence of metallic filler.[31,32]

The DSC cooling scan of nylon 6 and nylon 6/zinc composite materials is shown in Figure 2.

The crystallization temperature (T_c) of nylon 6 and its composites was measured from the maximum of the exothermic peak. The results presented in Table I and Figure 2 show that the crystallization temperature value for pure nylon 6 is $T_c = 182°C$, while the crystallization temperature of nylon 6 composite materials changes to lower values when the Zn particle content is $\leq$8.0 wt.% and begins to increase with increasing percentage of over 8 wt.% metallic powder in the initial reaction mixture. These data demonstrate that metallic particle filler (when the filling content was greater than 8.0 wt.%) has some influence on the nucleation of nylon 6 crystallites by increasing the crystallization temperature. The results are in agreement with the literature data.[27,33,34]

Melting behavior (second heating scan, after cooling) of nylon 6 and its composite materials with various weight contents of Zn particles is presented in Table I and Figure 3.

Pure nylon 6 gives double melting peaks, T_{m2} at 219°C and T_{m3} at 212°C, attributed to the α-form and γ-form crystals, respectively. It can seen that all composite materials prepared by rotational molding via anionic ring-opening polymerization of ϵ-caprolactam in the presence of zinc powder particles also exhibit two melting endothermic peaks at T_{m2} and T_{m3} corresponding to the melting of the α-crystalline form and γ-crystalline form respectively.[27,35]

The values of enthalpy of fusion of higher melting endotherm, T_{m2}, slightly increase when the Zn particle content is $\leq$8.0 wt.% and begins to slightly decrease with increase of the percentage over 8 wt.% of metallic powder in the initial reaction mixture. In the same time, the values of lower melting endotherm, T_{m3}, remain constant (at 212.0°C) when the percentage of Zn particles is between 0.0% and 12.0%, then they start to rise slightly with increase of the percentage of metallic powder over 12 wt.%.

Another important observation is that the values of melting temperature measured for all samples in the first heating scan, corresponding to the α-crystalline form, are slightly higher than those obtained from the second heating scan. This fact reflects the imperfections of the crystalline phase formed in the course of the cooling scan (crystallization of all specimens under identical conditions). These results are in agreement with those presented in the literature.[27,36] The degree of crystallinity of the pure nylon 6 and its composites was calculated from the DSC thermographs using the heat of fusion of 191 J/g for 100% crystalline nylon 6, and the values are given in Table I.

DSC measurements show that the degree of crystallinity (see Table I) in the first heating scan decreases with increasing amount of Zn powder particles, from 51.6% for pure nylon 6 to about 20.5% for PA/Zn/25%, while in the second heating scan the degree of crystallinity exhibits a decrease from 36.3% to 18.9% with increasing initial content of metallic filler in the range 0.0–25.0 wt.%. The composites demonstrate lower degree of crystallinity (the first and the second heating scan) than pure nylon 6.

Lower degree of crystallinity ascertained in the second heating scan indicates that the conditions for crystallization during cooling are much less favorable than those in the course of the polymerization-molding process. These results are comparable to those presented in the literature.[10,27]

WAXD Results

Wide-angle X-ray diffraction (WAXD) was used to investigate the changes in crystalline structure of nylon 6 and nylon 6 matrix in these composites. Nylon 6 exhibits two crystalline forms, namely the α-form and the γ-form.[19,37] The α-form has monoclinic structure, and the hydrogen bonds are formed between antiparallel chains. The γ-form also has a monoclinic structure, but the twisted chains allow hydrogen bonds to be formed between parallel chains. Nylon 6 is the most stable in the α-form, which can be transformed into the γ-form under various conditions.[19,37] The semicrystalline character of nylon 6 makes it possible to be investigated by WAXD. It is known that the crystal structure of the polymer matrix has a key role in determining the characteristics of polymer-based composite materials.[38] The changes in crystalline structure of nylon 6 when it constitutes the matrix in composite materials under study are presented in Figure 4.

Figure 4 shows the WAXD patterns for crystalline structures of anionic nylon 6/Zn composite materials with 6.0, 8.0, and 15.0 wt.% of metallic filler content in comparison with those of pure nylon 6.

For pure anionic nylon 6, two main reflections can be observed at about $2\theta = 20.5°$ and 23.0°, which are attributed to the α_1 and α_2 crystal form attributed to the reflection of 200 and 002/202 crystalline planes, respectively. The α_1 originates from the distance between hydrogen-bonded chains, whereas α_2 comes from the distance between hydrogen-bonded sheets.[27,39] Therefore, it is evident (from Figure 3) that the α-form is the most dominant crystalline phase in pure nylon 6 but with the addition of and then increases of metallic filler (>6 wt.%) into nylon 6, the intensity peaks of the α-form reflections of nylon 6 composite materials reduced substantially. Simultaneously, nylon 6/zinc composites show a new peak at $2\theta = 21.3°$, which is associated with γ-form crystal corresponding to reflection of

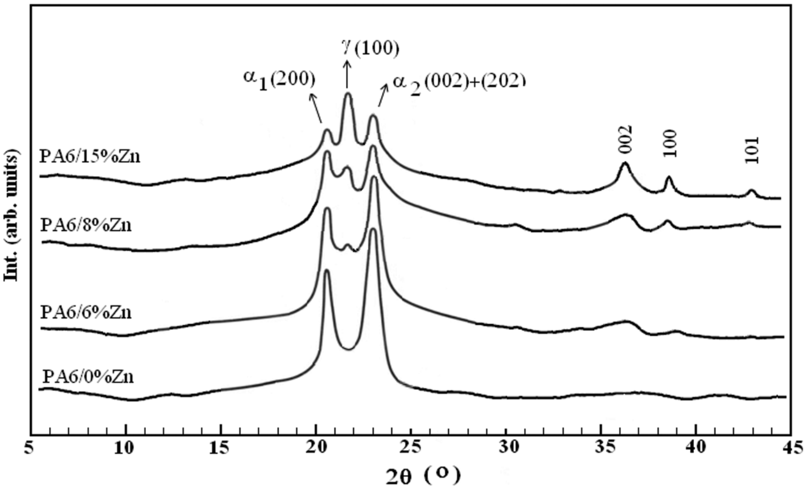

Figure 4. Wide-angle X-ray diffraction patterns of nylon 6 and nylon 6/zinc composite materials prepared by rotational molding via anionic ring-opening polymerization of CL in the presence of Zn particles (composition indicated on curves).

crystalline plane 100 of nylon 6 matrix. The reflections associated with γ-phase crystal and Zn particles become prominent with increase of the zinc concentration. Hence, the zinc particle filler in composite materials induces the formation of γ-form crystals of nylon 6, probably as a result of a heterogeneous nucleation mechanism. In other words, the addition of Zn particles disturbs the perfect arrangement of hydrogen-bonded sheets of the α-form.[24,40] The γ-form crystal becomes more evident with the addition of Zn particles.

The characteristic peaks of Zn powder dispersed in the nylon 6 matrix appeared at about $2\theta = 36.2$, 38.6, and 43.0°, relating to reflection of crystalline planes (002), (100), and (101), respectively.

Thermogravimetric Analysis

Thermogravimetric analysis (TGA), one of the most widely used thermal analysis techniques, can determine the thermal stability of polymers and polymer composite materials, an important property for their processing and applications. It is generally accepted that the thermal degradation reaction of semicrystalline polymers proceeds predominantly in amorphous regions due to the higher permeability to oxygen. In addition, it has been reported,[8,27,41] related to the thermal degradation of aliphatic polyamides, that the –NH–CO– bond is primarily split in the neighborhood of the carbonyl group. The TGA data corresponding to the temperatures of the maximum value of the rate of reaction (T_{max}) and the weight loss (%) at different decomposition temperatures for pure anionic nylon 6 and its composite materials are summarized in Table II.

Thermograms of pure nylon 6 and its composite materials with different contents of zinc particles, obtained under an air atmosphere at a flow rate of 30 mL/min

Table II. Thermogravimetric analysis (TGA) results for the measured nylon 6 and its composite materials

				Weight loss at different temperatures (%)			
Sample code	Zinc (wt.%)	T_{dec} (°C)	Residue (%)	350°C	400°C	450°C	500°C
PA6/0%Zn	0.0	428	7.0	4	22	81	90
PA6/6%Zn	6.0	433	13.2	5	16	75	84
PA6/8%Zn	8.0	437	15.6	4	14	73	82
PA6/12%Zn	12.0	432	19.1	5	11	69	79
PA6/15%Zn	15.0	428	22.6	4	10	66	75
PA6/20%Zn	20.0	426	27.8	4	10	62	71
PA6/25%Zn	25.0	424	33.3	4	8.0	56	69

and a temperature range of 30°–700°C at a scanning rate of 10°C/min, are shown in Figure 5.

Improved thermal stability of the nylon 6/Zn composite materials with respect to the pure nylon 6 matrix can be noticed. For example, for the thermal decomposition of the nylon 6/Zn composite materials with initial metallic filler content <12 wt.% Zn, an increase in the thermal decomposition temperature (T_{dec}) values of the nylon 6/Zn composites falls in the range of 428°–437°C (see Table II). A possible reason for the increased thermal stability of the nylon 6/Zn composites is the partially altered molecular mobility of nylon 6 chains due to their adsorption on the surface of the Zn particles. However, further incorporation of filler in the reaction mixture induces a slight decrease in the thermal stability of the nylon 6/Zn composites. Thermal decomposition temperatures of the nylon 6/Zn composites in this study were found in the range of 450°–500°C and should be associated to the break of amide groups.[8,42]

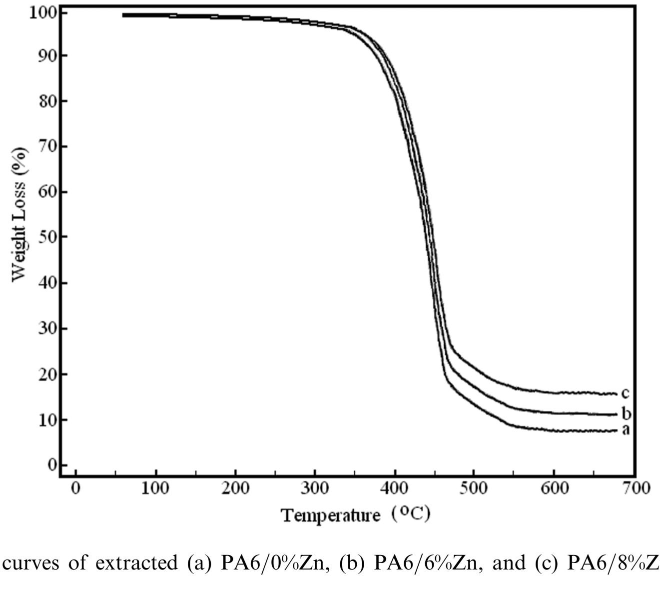

Figure 5. TGA curves of extracted (a) PA6/0%Zn, (b) PA6/6%Zn, and (c) PA6/8%Zn, as illustrative example.

Finally, one can conclude that the addition of zinc particles leads to composite materials characterized by matrix thermal stability similar or slightly improved with respect to pure nylon 6 (see Table II and Figure 5).

Mechanical Behavior

The mechanical properties of composite materials made of relatively low modulus polymeric matrix and high modulus filler, such as polyamide/metal composites, are sensitive to loading rate and temperature. All the mechanical properties of anionic nylon 6 and its composite materials evaluated in this study as a function of Zn content including notched Izod impact strengths and flexural properties as well as flexural strength and flexural modulus are presented in Figures 6 and 7. At the same time, for comparison, the properties of pure nylon 6 are also shown in these figures.

As shown in Figure 6, the introduction of Zn particles into nylon 6 resulted in a sharp decrease in notched Izod impact strength of the nylon 6 composites, such that 12 wt.% of Zn reduced notched Izod impact strength of polymer composites to about 38.55%. When the wt.% of Zn particles was increased from 15 to 25, notched Izod impact strength decreased slowly.

As can be seen in Figure 6, the impact strength decreases by 43% from 4.98 kJ m^{-2} for neat nylon 6 to 2.84 kJ m^{-2} for composite samples containing 25% metallic filler. The decrease in Izod impact strength of the nylon 6 composite materials is most likely due to the presence of Zn particles that may act as stress raiser and due to weak interfacial adhesion between the fillers and the nylon 6 matrix.[27,43,44]

It is clear from the observations in Figure 6 that the presence of metallic particles caused deterioration in impact strength in the nylon 6 composite system. Zinc particles act as the hard phase in the soft nylon 6 phase, so an addition of Zn particles would result in an increase of modulus of the composite materials. It was observed that as the amount of filler increases, the nylon zinc composites exhibit

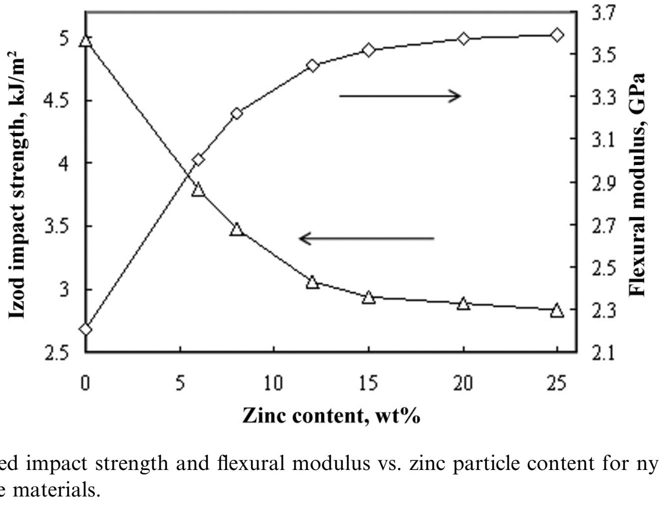

Figure 6. Notched impact strength and flexural modulus vs. zinc particle content for nylon 6 and nylon 6/zinc composite materials.

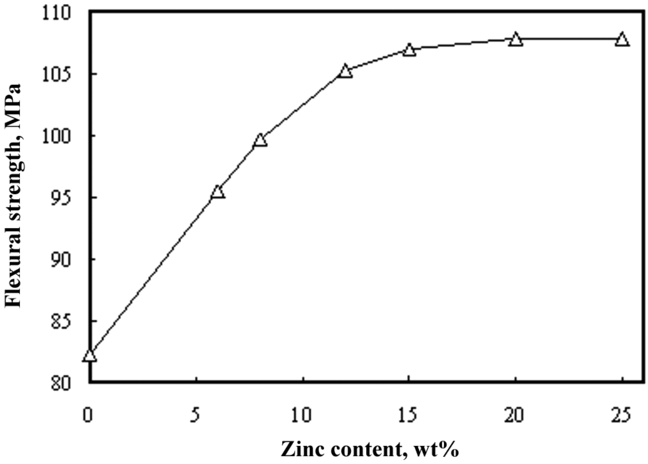

Figure 7. Flexural strength vs. zinc particle content for nylon 6 and nylon 6/zinc composite materials.

better flexural properties. It is clear from the observations in Figure 6 that as the flexural modulus increases sharply up to 12% filler concentration and then increases slowly. For pure nylon 6 sheet, the flexural modulus is 2.206 GPa; it sharply increases to 3.446 GPa for 12% metallic filler, and then slowly increases to 3.591 GPa for 25% zinc filler. A similar increasing trend with respect to level of zinc particle content is also evident in the flexural strength of nylon 6 zinc composites. Flexural strength is an important mechanical test for assessing the performance of the polymer composite materials.

Figure 7 shows the changes of flexural strength of the nylon 6 and nylon 6/zinc composite materials as a function of metal filler content.

As shown in Figure 7, the introduction of Zn particles into nylon 6 resulted in a sharp increase in flexural strength of the nylon 6 composites, such that 12 wt.% of Zn increased flexural strength of polymer composites to about 27.21%. When the wt.% of Zn particles was increased to 15% and 25%, flexural strength increased slowly. Flexural strength of the polyamide 6/zinc composite materials has direct dependency on the metallic content. Compared to pure nylon 6, the flexural modulus and flexural strength of nylon 6/Zn composite materials are improved by 62.78% and 31.0%, respectively. In contrast to the notched Izod impact strength, Zn powder particles improved the flexural properties such as flexural modulus and flexural strength of the nylon 6 composite materials.

The mechanical properties of the composite materials in this study are in general agreement with the behavior expected for polymer composites with rigid fillers.[10,27]

CONCLUSIONS

Nylon 6 and nylon 6/zinc composites with different contents of zinc particles have been prepared by rotational molding via anionic ring-opening ϵ-caprolactam polymerization in the presence of zinc particle fillers. The thermal and mechanical characteristics of the nylon 6/zinc composite materials were compared with the results for pure nylon 6.

From the above study it could be concluded that the degree of crystallinity (first and second heating scan) decreases proportionally to the amount of the filler. The crystallization temperature of the nylon 6 composites first decreases when the Zn powder particle content is <8.0 wt.%, then it begins to increase with increasing percentage of metallic over 8 wt.%. The melting temperature (the first and second heating scan) of the nylon 6 composite remained constant above 225°C and slightly increased, respectively, when the Zn powder particle content is <8.0 wt.%, after which both decrease slightly with increasing percentage of over 8.0 wt.% powder. From the result of TGA experiments, carried out in air, in the 30°–700°C temperature range nylon 6/zinc composite materials under study can be considered as a thermally stable polymers. The information obtained from DSC and X-ray analysis indicate that the samples are predominantly in the α-form. The flexural tests showed that the modulus and strength of nylon 6/zinc composites increase with increase of filler content, while the Izod impact strength decreases with increasing metal loading.

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