

# Processing and Property Relationship of Polypropylene Monofilaments Containing Nanoclay

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**ABSTRACT:** This study focuses on combining traditional polymeric materials with nanoclays to engineer new composite monofilaments for nontraditional industries using extrusion process. Polypropylene/nanoclay composite monofilaments were produced using single screw extruder, and the structure and properties of new monofilaments were characterized using scanning electron microscopy (SEM), X-Ray diffraction (XRD), Fourier transform infrared spectrometry (FT-IR), thermal gravimetric analyzer (TGA), differential scanning calorimeter, and Instron universal testing machine. Composite monofilaments were prepared at different weight percentage of nanoclay (1%, 2%, and 3%) and compatibilizer (10%, 15%, and 20%) contents. The results showed that the amount of the nanoclay and compatibilizer affected the formation and final properties of the new monofilaments. It was observed that tensile stress of composite monofilament decreased compared to pure polypropylene monofilament. Melting temperature and degree of crystallinity were not affected much; however, there was significant increase at decomposition temperature of composite polypropylene monofilaments.

**KEY WORDS:** high performance fibers, processing, fiber, yarn, fabric formation, nanoclay, polypropylene, monofilament.

## INTRODUCTION

**P**OLYMERIC COMPOSITE MATERIALS are reinforced with nanoparticles to obtain certain properties. Basically, the nanoparticles are dispersed in

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the polymer matrix at low amounts, most of the time less than 6% by weight [1].

Nano particle reinforced composites have been studied in recent years because of their superior strength, modulus, reduced gas permeability, improved solvent resistance, and heat distortion temperature [2–4]. Polymeric properties can be improved even at low loading without negative effect on density, transparency, and process ability [1]. The conditions for successful reinforcement and good properties are the homogeneous distribution and dispersion of the reinforcing components, high aspect ratio of the nanolevel fillers, and good adhesion between polymer matrix and fillers [5].

One of the most important nanofillers is layered silicates (nanoclays). Montmorillonite clay has two silicate tetrahedral layers sandwiching an inner octahedral layer which consist of either alumina or magnesia [6,7]. The duty of the  $\text{Na}^+$  is to counterbalance the Al or Mg for generation of the negative charge in platelet. Montmorillonite has many platelets (layers), and there are Van der Waals forces between these layers [8]. Montmorillonite is in powder form with a mean of about 8  $\mu\text{m}$  particle size, and in each particle of powder there are more than 3000 platelets [9]. The thickness of each layer is almost 1 nm, and the length of these layers can vary from 300 Å to several microns [10]. Therefore, it has high aspect ratio.

The polymer layered silicate nanocomposites are based on separation and dispersion of the clay platelets in polymer matrix, and this dispersion range goes from simple intercalation to complete exfoliation of the silicate layers [3,11]. Clay layers are placed between the structure, and they exist as particles containing tactoids at immiscible nanostructures. Clays are more separated at intercalated systems, and penetration of the polymer chain may occur in the interlayer spacing. Clay layers are separated individually and better dispersions are achieved in exfoliated nanostructure compared to the other two systems [2,7]. However, it is very difficult to obtain full exfoliation due to poor affinity between nanofillers and polymer matrix [6,12,13].

Most of the time, polymer/clay nanocomposites have the combination of good processability characteristic of the polymers, and great physical properties of the clays such as mechanical, thermal, and electrical properties [14–16]. Polypropylene-nanoclay nanocomposites are interesting for industrial applications because PP is one of the most widely used thermoplastic polymer and nanoclays are effective as filler materials [17]. However, there are some difficulties to form these nanostructures. Nanoclays are known to be naturally hydrophilic while PP has no polar groups in its backbone and therefore, it is a hydrophobic polymer [1,18]. Most of the time, poor interfacial bonding between the PP matrix and clay surface is obtained with low level of dispersion of the clay plates [19]. Pretreatment of clays using

ammonium salts or alkyl phosphonium makes the clays organophilic; the modified clays are called organoclays [2,20]. Compatibilizers (PP-g-MA) are used to improve intercalation/exfoliation of the organoclay and maximize its interfacial contact with polymer matrix [2,4]. PP-MA has polar groups (–OH, COOH) and increases the polarity of the polypropylene [21]. They help to bond the clay and PP together due to the hydrogen bond between the polar carboxylic group of PP-MA and the oxygen or hydroxyl group in silicates [22]. The interlayer spacing of the montmorillonite increases, and interaction between the silicate layers gets weaker.

The montmorillonite intercalated with the PP-MA oligomers have good contacts with PP under strong shear [20,23]. Stacked platelets can be broken in to smaller pieces and the interlayer spacing increases during extrusion process. This space increase helps for better dispersion and distribution of the nanoclays in polymer matrix. There are different techniques to disperse the nanofillers at nanoscales in polymer matrix; *in situ* polymerization, solution blending, and melt intercalation [11,20,24–26]. Melt intercalation is the most widely used method for monofilament processing, and it is environmental friendly due to lack of solvent during mixing or processing. The features of the extruder and screw configuration are important to obtain good organoclay dispersion. Better dispersion can be achieved with longer residence times in the extruder. In some cases, having a higher melt viscosity is useful in obtaining dispersion, because of the higher stresses that can be enforced on the clay particles [27]. Other parameters such as barrel and die temperature, draw-down ratio, and heater temperature can be adjusted for better exfoliation. Correct settings of these parameters can change the reinforcing effect, agglomeration of clay, and synergistic effect of nanoparticles.

Some of the polypropylene monofilament application areas are nets and fabrics for agricultural end uses, geotextiles, any kind of sun breakers, sun blinds, shade nets, wind walls, safety nets, swimming pool covers, reinforcements in various coated products (such as plastics, and papers), filtration, carpeting, packaging, concrete reinforcement, and medical sutures.

## EXPERIMENTAL

### Materials

Polypropylene copolymer 30 Melt 2 Izod Natural with melt flow index (MFI) of 30 g/10 minutes, and density of 0.91 g/cm<sup>3</sup> was purchased from Premier Plastic Resins, Inc. (USA). Cloisite 15A, which was already modified with a quaternary ammonium salt, was purchased from Southern

Clay Products, Inc. Polybond 3200 (PP grafted with malefic anhydride; 1.0 weight % maleic anhydride level) was donated by Chemtura Corporation (USA). It has 115 g/10 minutes MFI, and 0.91 g/cm<sup>3</sup> density.

### Preparation of PP/Nanoclay Composite Monofilaments

All PP/Nanoclay monofilaments were prepared with melt intercalation method through a Wayne single screw extruder ( $L/D = 30$ ). Different amounts of nanoclay and compatabilizer were used to form composite monofilaments. Attempts to produce polypropylene/nanoclay composite monofilaments without compatabilizer were unsuccessful. Table 1 shows the master batches and sample codes.

Cloisite 15A was dried in vacuum oven at 65°C for 12 hours prior to the manufacturing. Pure polypropylene pellets, Cloisite 15A, and Polybond 3200 were mixed manually before processing of the monofilaments. Processing temperatures are given in Table 2.

The extruder screw speed was 100 RPM, and melt pump speed was set to 13 RPM. A draw-down ratio of 7 was applied to the monofilaments. After the water tank, air was blown on the monofilaments to dry them before drawing section. After the drawing, all monofilaments, except pure polypropylene, were chopped and re-extruded to obtain better dispersion and distribution of the nanoclays in polymer matrix.

## CHARACTERIZATION

The surface morphology of the pure and composite monofilaments was investigated by using Zeiss EVO 50VP scanning electron microscopy (SEM).

*Table 1. Sample codes and blends of master batches.*

| Sample code | Clay loading (%) | Compatabilizer loading (%) |
|-------------|------------------|----------------------------|
| PP          | —                | —                          |
| P1-C10      | 1                | 10                         |
| P1-C15      | 1                | 15                         |
| P1-C20      | 1                | 20                         |
| P2-C10      | 2                | 10                         |
| P2-C15      | 2                | 15                         |
| P2-C20      | 2                | 20                         |
| P3-C10      | 3                | 10                         |
| P3-C15      | 3                | 15                         |
| P3-C20      | 3                | 20                         |

Probe current was adjusted between 700–900 pA. All specimens were coated with gold before the experiment.

Basic crystalline structures of pure and composite monofilaments were analyzed using Bruker APEX single crystal X-ray diffractometer. Sample collections were made between  $2\theta$  range of  $5^\circ$ – $40^\circ$  with  $\text{CuK}\alpha 2$  radiation at 40 kv voltage, and 40 mA generator current. Scanning time was set to 750 seconds, and step size was  $0.01^\circ$ .

Melting behavior and degree of crystallinity measurements were made by using Perkin-Elmer DSC Q2000 instrument under nitrogen atmosphere. Sample sizes were between 5–10 mg. Samples were heated from  $50^\circ\text{C}$  to  $250^\circ\text{C}$  at  $10^\circ\text{C}/\text{minute}$ , and held there 2 minutes, then cooled to room temperature. The crystallinity of the samples was calculated according to the following formula:

$$X_c = \frac{\Delta H}{\Delta H_{PP}^0} \quad (1)$$

$\Delta H$  is the enthalpy of fusion of the sample (J/g) and  $\Delta H_{PP}^0$  is the enthalpy of fusion of completely crystalline PP ( $\sim 209$  J/g) [28].

Thermal stability of monofilaments was measured by using Perkin-Elmer TGA Q500 instrument. The heating rate was  $20^\circ\text{C}/\text{minute}$  for all samples, and the temperature range was  $50$ – $600^\circ\text{C}$  under atmospheric air.

Nicolet 6700 FTIR spectrometer was used to characterize the molecular conformation of the pure and composite monofilaments. The number of each scan was made at 32, and experimental scan was set at 16.

Samples were tested according to the ASTM D-3822-07 Standard Test Method for Tensile Properties of Single Textile Fibers with Instron 5565 Testing Machine. The diameter of each sample was measured with a

Table 2. Temperature settings.

| Heating zones | Temperature ( $^\circ\text{F}$ ) |
|---------------|----------------------------------|
| Barrel zone-1 | 400                              |
| Barrel zone-2 | 400                              |
| Barrel zone-3 | 400                              |
| Barrel zone-4 | 400                              |
| Die zone-1    | 425                              |
| Die zone-2    | 425                              |
| Die zone-3    | 425                              |
| Die zone-4    | 425                              |
| Die zone-5    | 425                              |
| Die zone-6    | 425                              |

microscope. Five tests were done for each sample and the average values were used for the evaluation of the mechanical properties.

## RESULTS AND DISCUSSION

The surface morphology of pure polypropylene and some of polypropylene/nanoclay composite monofilaments are presented in Figure 1 (a)–(d). SEM images show the distribution of nanoclay particles along the monofilaments. P3-C10 sample has bigger particles compared to P1-C10

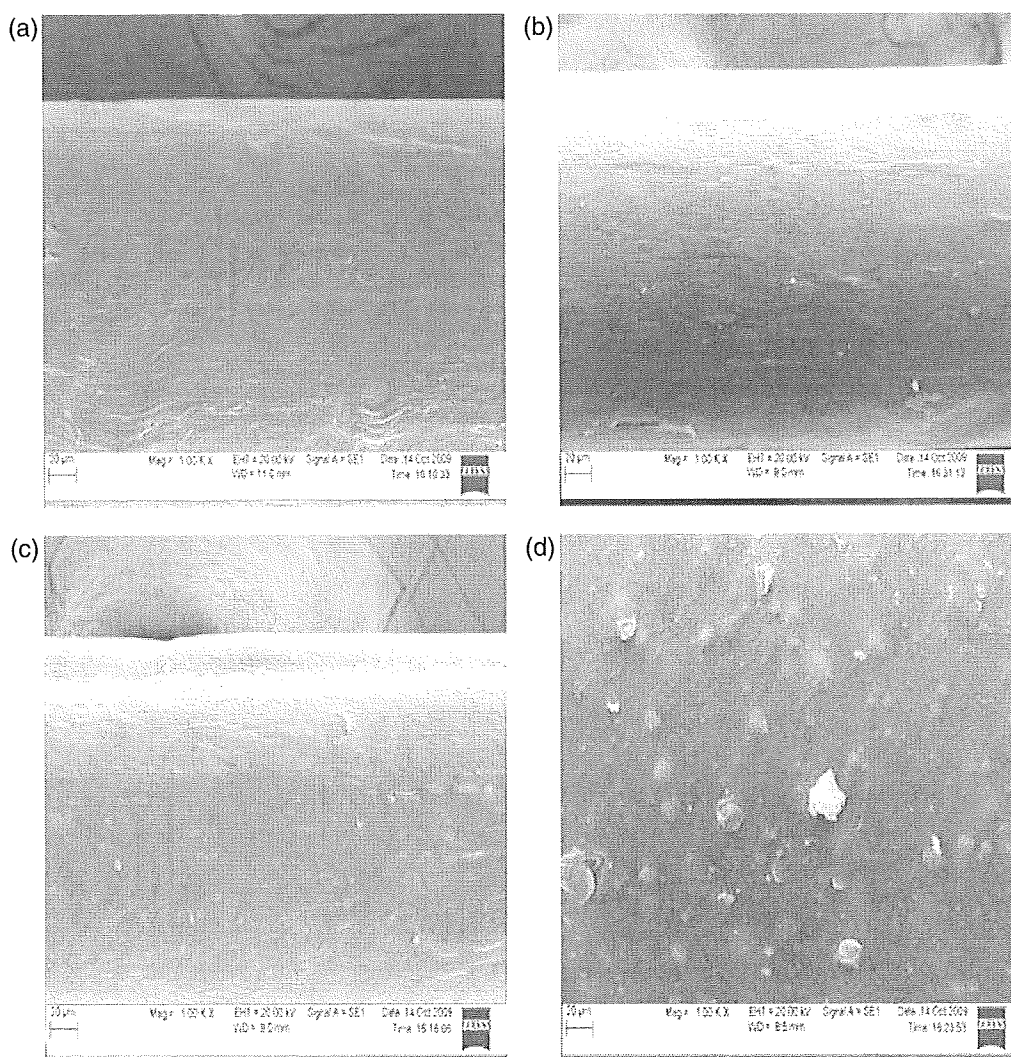


**FIGURE 1.** SEM images of surface views: (a) pure PP Filament, (b) P1-C10, (c) P2-C10, (d) P3-C10.

and P2-C10. Also agglomeration can be seen for P3-C10 sample due to low dispersion and distribution of nanoclay particles in polymer matrix. Low shear effect of single screw extruder can also cause this agglomeration. P1-C10 and P2-C10 samples have almost the same level of nanoclay dispersion. The range of the particles size is between 700 nm to 4 microns for P1-C10, and 3 to 10 microns for P2-C10. However, there are also smaller nanoscale particles which may help for reinforcement of the monofilaments. On the other hand, drawing of the monofilaments also helps with the distribution of the nanoclay along the monofilament. In addition, sample P3-C10 could not be drawn well during processing compared to other samples due to agglomeration of the nanoclays.

The FTIR spectrum of pure polypropylene and composite monofilaments is given in Figure 2. Si-O stretching ( $1005\text{ cm}^{-1}$ ) and Si-O bending ( $480\text{ cm}^{-1}$ ) can be observed for Cloisite 15A. There are vibration of peaks between  $900$  and  $1000\text{ cm}^{-1}$ , and silicates and carboxyl groups might form bond in this range. However, all the peaks of composite monofilaments show similar characteristics compared to pure polypropylene monofilaments even if the clay loading is increased. This shows that there are weak bonds between nanoclays and polymer matrix due to agglomeration or lower dispersion of nanoclays.

Figure 3 shows the X-ray diffraction (XRD) of pure polypropylene monofilaments, P1-C10, P2-C10, and P3-C10 drawn monofilaments. The result can give a general view about the nanocomposite structure.

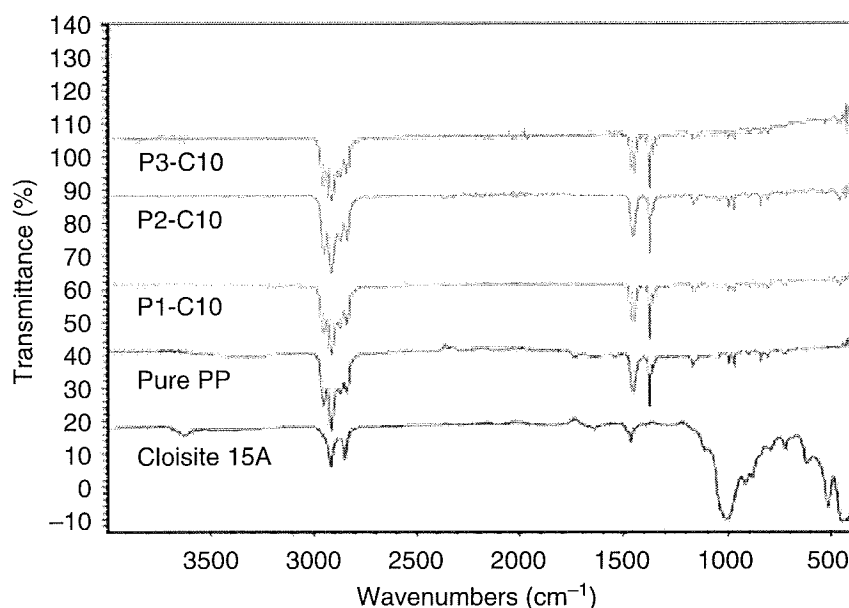


FIGURE 2. FTIR spectra of pure and composite monofilaments.

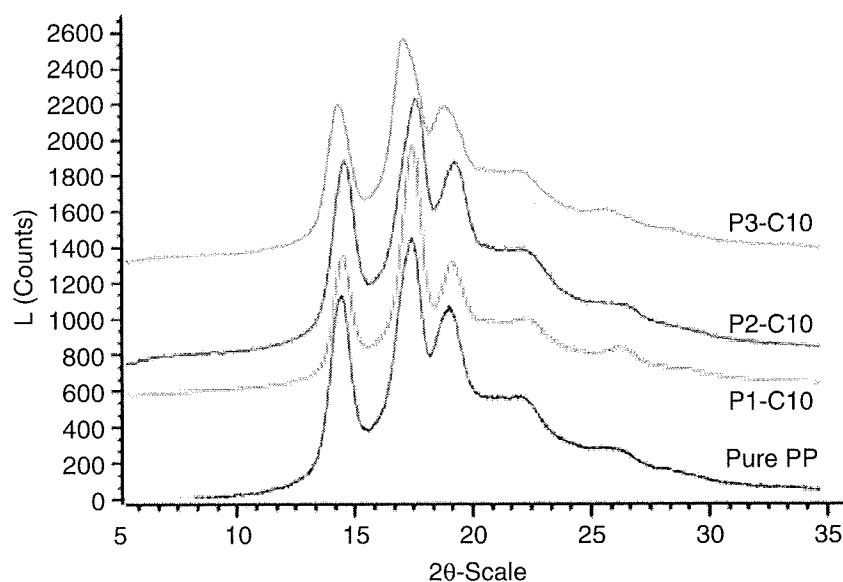


FIGURE 3. XRD curves of pure polypropylene and composite monofilaments.

Table 3. Diffraction peaks of the samples at  $2\theta$  angle range.

| Pure PP | P1-C10 | P2-C10 | P3-C10 |
|---------|--------|--------|--------|
| 14.11   | 14.04  | 14.07  | 13.97  |
| 16.9    | 16.73  | 16.88  | 16.71  |
| 18.52   | 18.44  | 18.48  | 18.35  |
| 21.69   | 21.62  | 21.63  | 21.44  |

The different peaks may indicate the distribution of clay layers and d-spacing which is the distance between the clay layers. Polymer nanocomposite peaks often show shifting to lower  $2\theta$  angle or larger d-spacing. Decrease or disappearance of characteristic peaks indicates the formation of intercalation or exfoliation nanostructures.

Table 3 shows the diffraction peaks of samples at  $2\theta$  angles. The results show that there is no significant change at peak characteristic of the samples. There is only little shifting at P3-C10 sample, and agglomerated clay particles may cause this difference. It can be concluded that addition of nanoclay did not affect basic crystal structure of the samples.

Crystallization and melting behavior of the monofilaments with different nanoclay and compatibilizer loading are shown in Table 4. Polypropylene is a semicrystalline polymer, and most of the properties depend on crystallinity, spherulites size, and the size of lamellar crystals. On the other hand,

Table 4. Thermal and tensile results of composite monofilaments.

| Samples | Decomposition temperature (°C) | Tensile strength (MPa) | Elongation at break (%) | $T_m$ (°C) | $T_c$ (°C) | $X_c$ (%) |
|---------|--------------------------------|------------------------|-------------------------|------------|------------|-----------|
| Pure PP | 358.86                         | 308.8                  | 63.6                    | 166.79     | 131.17     | 41.04     |
| P1-C10  | 368.32                         | 289.1                  | 52.0                    | 166.60     | 130.73     | 42.48     |
| P2-C10  | 376.35                         | 286                    | 53.0                    | 166.33     | 130.66     | 41.21     |
| P3-C10  | 393.95                         | 276.1                  | 54.7                    | 165.78     | 130.80     | 41.95     |
| P1-C15  | 367.23                         | 292.9                  | 55.1                    | 165.91     | 129.67     | 42.81     |
| P2-C15  | 375.77                         | 282.6                  | 52.5                    | 165.39     | 129.61     | 43.81     |
| P3-C15  | 389.75                         | 281.4                  | 53.6                    | 165.33     | 128.97     | 42.29     |
| P1-C20  | 367.61                         | 294.7                  | 63.0                    | 166.12     | 128.35     | 43.56     |
| P2-C20  | 377.65                         | 279.7                  | 64.4                    | 165.53     | 129.20     | 42.77     |
| P3-C20  | 389.23                         | 273.6                  | 64.2                    | 165.65     | 127.73     | 42.69     |
| PP-MA   | 359.15                         | —                      | —                       | 162.36     | 112.61     | 42.40     |

nanoclays are known as nucleating agents and accelerate the crystallization process [29]. It was observed that melting point of pure polypropylene decreased slightly with the addition of nanoclay as shown in Figure 4. Melting point decrease may suggest a reduction of lamellar size; probably the clay can prevent the growth of individual lamellae, and more lamellae are formed with smaller sizes [30].

Crystallization temperature ( $T_c$ ) decreased slightly with the presence of nanoclay in the system. This also indicates the nucleating affect of the nanoclays in crystallization of polypropylene. Also, addition of compatibilizer may cause this reduction because of lower crystallization temperature (112.61°C). The degree of crystallinity ( $X_c$ ) was calculated according to Equation (1), and a slight increase (41.04% to 43.81%) was observed with addition of nanoclay. It was also observed that  $X_c$  increased by increasing compatibilizer amount in composite monofilaments.

Nanoclays are known as good heat insulators due to their good thermal resistance. Nanoclays can prevent the penetration of oxygen, and provide slower decomposition. Thermal gravimetric analyzer (TGA) results are given in Table 4. The results show that addition of nanoclay increased decomposition temperature of the composite monofilaments compared to pure polypropylene monofilaments.

Figure 5 shows the comparison of pure and composite monofilaments during decomposition. Their onset temperatures are similar; but after 300°C, they start to lose their weight under temperature. Pure polypropylene shows rapid decrease of weight compared to composite monofilaments, and it decomposes at 358.86°C. The decomposition temperatures in Table 4

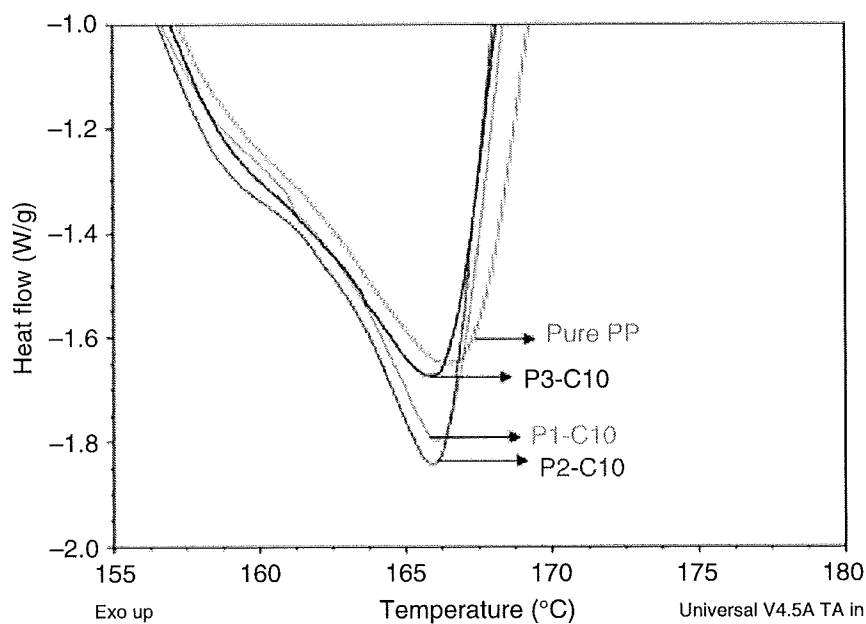


FIGURE 4. Melting behavior of pure and composite polypropylene monofilaments.

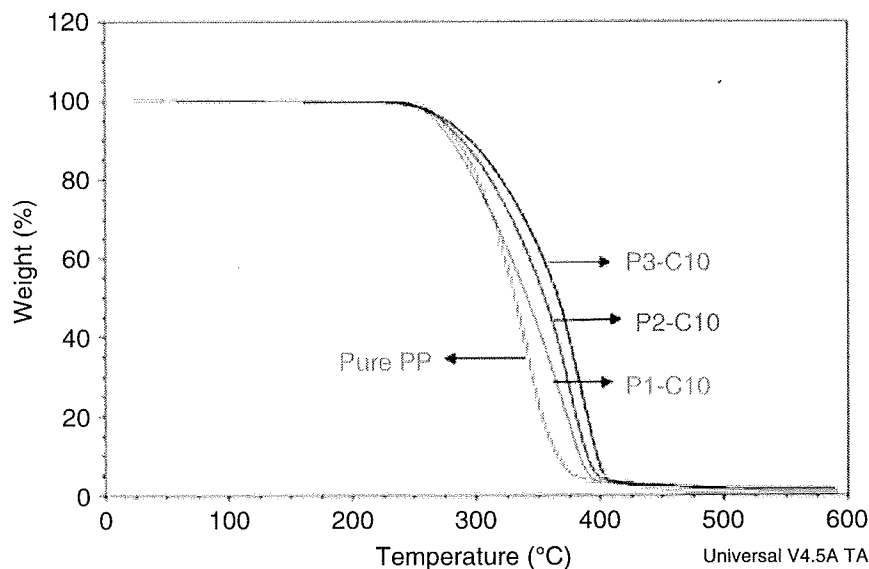


FIGURE 5. Decomposition behavior of pure and composite polypropylene monofilaments at different nanoclay loadings.

are the means of four samples' decomposition temperature for each monofilament. Figure 6 shows how to determine the decomposition temperature for one of pure polypropylene monofilaments with the TGA instrument.

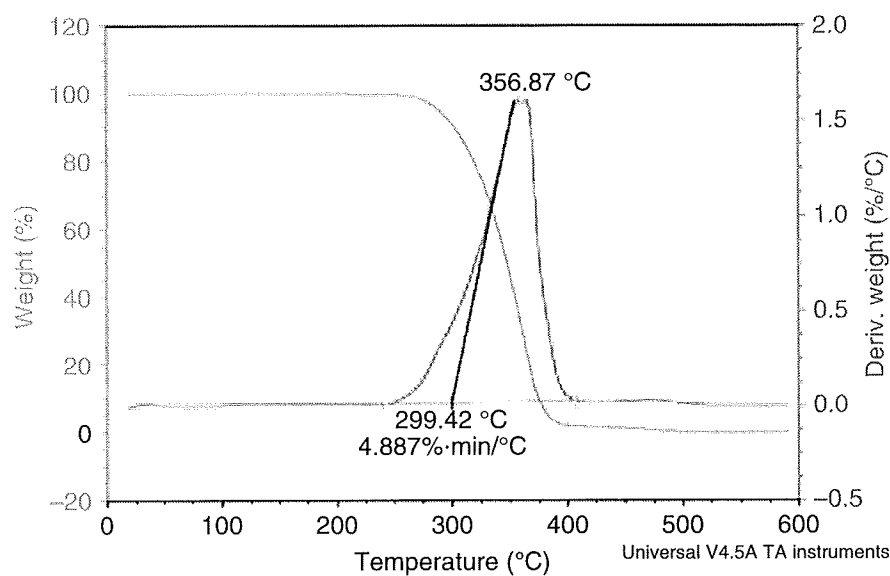


FIGURE 6. Determination of pure polypropylene monofilament's decomposition temperature.

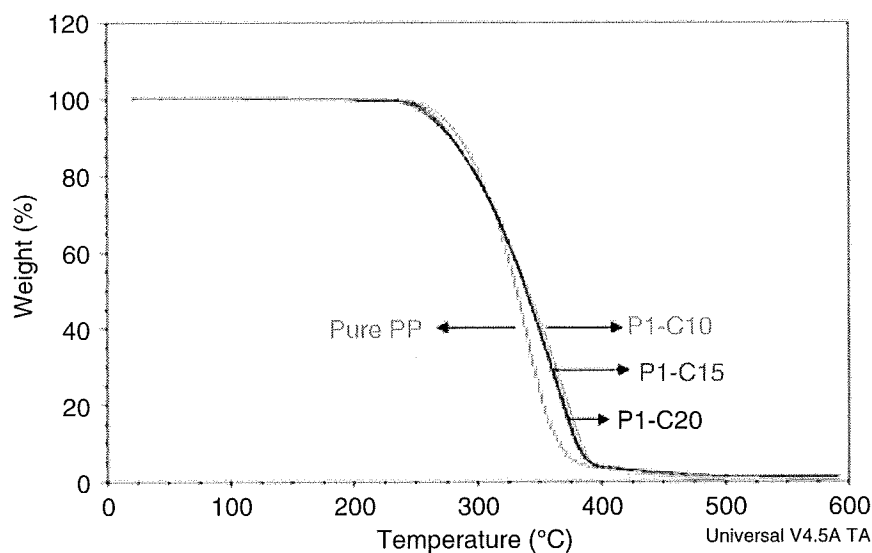


FIGURE 7. Decomposition behavior of pure and composite polypropylene monofilaments at different compatibilizer loadings.

Figure 7 shows the effect of compatibilizer on decomposition behavior: all the samples have higher decomposition temperature than pure polypropylene monofilament. However, composite monofilaments have almost the same behavior which indicates compatibilizer may not have any

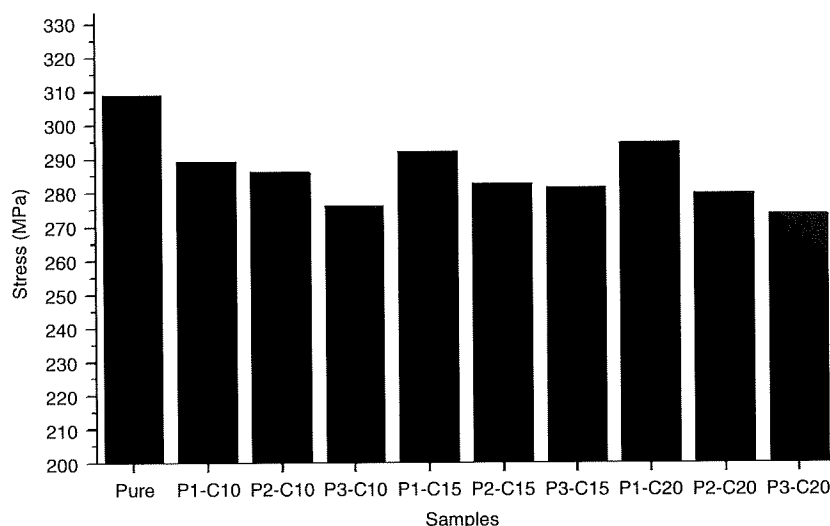


FIGURE 8. Tensile behavior of pure and composite monofilaments.

significant effect on decomposition. The decomposition temperature of the compatibilizer ( $359.15^{\circ}\text{C}$ ) is comparable to that of pure polypropylene ( $358.86^{\circ}\text{C}$ ).

Table 4 shows the tensile properties of pure and composite monofilaments. It was observed that addition of the nanoclay caused lower tensile properties. The reduction is almost 10% at 3% clay loading. Higher nanoclay containing samples showed more reduction compared to the lower ones. SEM results also indicate some agglomeration during the processing inside the monofilaments. These agglomerated particles, which are in micron sizes, can reduce interaction between nanoclay and polymer matrix, and the result will be interfacial debonding under load. Besides, these particles can cause voids and act as a crack initiator under the load. Figure 8 shows the decrease in tensile stress of the pure and composite monofilaments.

Nanoclay particles are stiff materials with no elongation properties; therefore addition of these particles can lower elongation behavior of the monofilaments. Elongation at break values decreased in the presence of nanoclays except at 20% compatibilizer content as shown in Table 4. Samples containing 20% compatibilizer showed higher elongation properties compared to 10% and 15% ones. Compatibilizer might have contributed to the elongation values after certain amount of loading at composite monofilament manufacturing.

## CONCLUSIONS

Nanoclay added PP monofilament structures were produced with single screw extruder machine. A commercial nanoclay was used in this study.

Draw-down ratio was set to 7. The formation of composite monofilament structures is affected by the amount of nanoclays, compatibilizers, and polarity of the polymer. It was more difficult to manufacture composite monofilaments while there is more wt% nanoclay content because they caused more breakage during processing. There were mechanical and thermal changes in PP properties with the addition of nanoclays. Tensile stress decreased with the addition of nanoclays due to some agglomerated particles in the system. Decomposition temperature increased compared to pure polypropylene monofilament because of good heat insulation properties of nanoclays. Proper mixing is very important to disperse the nanoclays and compatibilizer uniformly during the process. More nanoclay dispersion can be achieved by increasing the space between clay layers and having higher shearing effect during processing.

### ACKNOWLEDGEMENT

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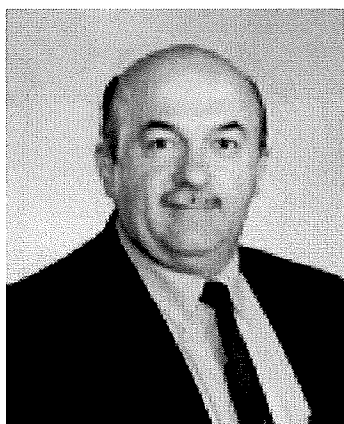
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