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Experimental Investigation of Effects of the Nucleating Agent on Mechanical and Crystallization Behavior of Injection-Molded Isotactic Polypropylene

Mesut KANDEMIR[1a], **İdris KARAGÖZ**[1b], **Harun SEPETÇIOĞLU**[2c]

[1]Polymer Materials Engineering Department, Yalova University, 77200, Yalova, Turkey
[2]Metallurgy and Materials Engineering Department, Selçuk University, 42075, Konya, Turkey
idris.karagoz@yalova.edu.tr

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Abstract: In this study, we investigated the effect of talc (magnesium-silicate-monohydrate/${}_3MgO.{}_4SiO_2.H_2O$) nucleating agent (NA) on the thermal and mechanical properties of isotactic-polypropylene (i-PP) at loadings ranging from 0.5 to 5% by weight using the melt compounding method. The results demonstrated that NA/i-PP exhibited significantly improved mechanical performance compared to neat i-PP. In addition, the influence of the concentration of the NA on the melting and crystallization behavior of neat i-PP was also analyzed through DSC curves. The tensile and flexural properties of i-PP enhanced as NA concentration increased. Furthermore, scanning electron microscopy revealed that there is a significant difference in the fracture surface between the NA/i-PP and the i-PP matrix. Consequently, it was determined that the NA (talc) used in this study was a good NA for i-PP, improved the mechanical and thermal properties of the product, affected the crystallization properties and the microstructure, and reduced the mold cycle time. Besides these, the use of various additives between talc and i-PP matrix that can improve interface features are expected to be an alternative approach to the new i-PP design with much higher mechanical and thermal properties.

Keywords: polypropylene, crystallization, nucleating agent, mechanical properties

1. Introduction

Nucleating agents (NA) are widely used in the plastic industry to regulate the crystallite size of crystalline polymers and improve the thermal and mechanical properties of materials. A nucleating agent influences the polymer's crystalline properties that could occur in homogeneous or heterogeneous processes [1-3]. For example, the NAs accelerate the crystalline rate of polypropylene (PP), and at the same, they increase the crystalline temperature and the crystallinity [4]. In addition, the NAs can be used to control the morphology of semi-crystalline polymers such as PP, allowing the PP to produce the α-form, β-form, γ-form, δ-form, and smectic phase [5, 6]. These crystalline phases and their morphologies determine PP's basic mechanical, thermal and optic properties [4, 7].

It has been well known that different kinds of NA, such as α-NA and β-NA modify the toughness, strength, and stiffness of isotactic polypropylene (i-PP) [8-10]. In this context, talc, calcite, silica [5], nanoparticle [11], fiber [1], and organic salt [12] are preferred as NA to modify i-PP in industrial production. In addition, the NAs based on sorbitol acetals that increase the crystallization rate alter the haze and clarity level of i-PP [7,8,10].

Many studies about the clarity level on i-PP are tried to control the crystalline structure (spherulite's diameter, lamella thickness, etc.) of i-PP by adding NAs that increase clarification [13-17]. Besides, the talc, one of the silicate derivatives, can be improved clarification of i-PP by promoting

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ORCID ID: [a]0000-0003-3498-4389; [b]0000-0002-2644-8511; [c]0000-0001-5746-4234

crystallization with the nucleating effect. In addition, the viscosity, crystallization temperature and melting temperature, thermal conductivity, and thermal diffusivity of i-PP can be governed by the addition of talc [18-21].

Comprehensive works have been reported in the literature on the PP mechanical properties related to the crystallization characteristics and morphology nucleated with the various artificial nucleation agents [19, 22-24]. Kim at al. [25] investigated the crystallization behaviors of PP using nucleated NC-4 (bis(p-ethylbenzylidene) and EC-4 (p-chloro, p'- methyl). They reported that the NC-4 is more effective in nucleation activity than EC-4 at a cooling rate of 4.0 °C/min. The crystallization peak temperatures also raised the temperature by 17 °C as the concentration of the nucleating agents increased. Besides these, they highlighted that the number of nuclei decreases due to agglomeration as the weight percentage of agents increases up to their saturation concentrations. Qui et al. [26] investigated the effect of silane-grafted polypropylene on mechanical properties and crystallization temperature of talc/PP. They reported that the silane-grafted polypropylene improves the mechanical properties of talc/PP due to enhancement in the talc nucleating activity, and thus, the formation of a finer and more resistant crystalline texture of PP in the polymer blends. Dong et al. [27] studied the TMB-5 effects of PP's fast and slow crystallization on its microstructure and thermal stability. They stated that TMB-5, a kind of commercial β type-nucleating agent, has been efficient as a nucleating agent for PP. They also emphasized that the thickness of the lamellar and the crystal's stability rise with increment the concentration of the nucleating agent.

Talc has been using as a NA in industrial production to improve clarifier properties. However, the literature has not reported the mechanical properties and crystalline morphology of i-PP containing a talc. Furthermore, it has not been studied whether the nucleating effect on i-PP depends on NA concentration. Therefore, it is needed to investigate how this NA and its concentration modifies i-PP crystallinity, thermal, and mechanical properties. Hence, this study has focused on the effect of talc additive on i-PP crystallization, thermal and mechanical properties.

2. Experimental Methods

2.1. Materials

A commercial isotactic polypropylene (i-PP) was used under the trade name ISPLEN® PP 070 G2M (Repsol-YPF, Spain) with a Young's Modulus of 1550 MPa, notched Charpy impact resistance of 4 kJ/m^2, melt flow index of 12.0 g/10 min, and a density of 0.905 g/cm^3, according to producer data. A nucleating agent that is a masterbatch (Product Code PP/F 109311) was supplied by Aksoy Plastic, Turkey.

2.2. Preparation of Sample

A masterbatch of 10 wt.% NA (talc) and 90 wt% i-PP was melt blended with i-PP to obtain the samples containing wt.% 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 4.0, and 5.0 of NA. The melt blending of blends was carried out on a twin-screw extruder (Gulnar, Turkey) with a 24 L/D ratio, Ø16 mm screw diameter, a screw speed of 250 rpm, and nozzle temperatures of 210 °C. After producing granules, they were injection-molded, and the standard samples were molded using a universal injection-molding machine (Engel, Turkey) with melt temperatures of 185–220 °C and a mould temperature of 30 °C for characterization tests. Then, the i-PP/NA blends compositions containing 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 4.0, and 5.0 wt.% of the nucleating agent were designated as PP/NA0.5, PP/NA1.0, PP/NA1.5, PP/NA2.0, PP/NA2.5, PP/NA3.0, PP/NA4.0 and PP/NA5.0 respectively.

2.3. Characterization

A masterbatch of 10 wt.% NA (talc) and 90 wt% i-PP was melt blended with i-PP to obtain the samples containing wt.% 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 4.0 and 5.0 of NA. The melt blending of blends was carried out on a twin-screw extruder (Gulnar, Turkey) with a 24 L/D ratio, Ø16 mm screw diameter, a screw speed of 250 rpm, and nozzle temperatures of 210 °C. After producing granules, they were injection-molded, and the standard samples were molded using a universal injection-molding machine (Engel, Turkey) with melt temperatures of 185–220 °C and a mould temperature of 30 °C for characterization tests. Then, the i-PP/NA blends compositions containing 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 4.0 and 5.0 wt.% of the nucleating agent were designated as PP/NA0.5, PP/NA1.0, PP/NA1.5, PP/NA2.0, PP/NA2.5, PP/NA3.0, PP/NA4.0 and PP/NA5.0 respectively.

The uniaxial tensile measurements of samples were performed on a Zwick/Roell-Z020 model tensile machine following ISO 527-1 at the cross-head speed of 50 mm/min and ambient temperature of 25 °C. Standard deviation values for Young's modulus, ultimate tensile strength, and strain at break were calculated for each blend.

The samples used in the flexural test were prepared following the ISO 527-2 standard. Tests were carried out with Zwick/Roell-Z020 model test device and carried out with a span-to-depth ratio of 16:1 at a cross-head speed of 2.0 mm/min and room temperature. Four samples were tested for each sample, and the flexural results were given as the mean and standard deviation.

The notched Charpy impact test was performed using an impact tester (Instron-120D, USA) with a 5.0 J pendulum hammer according to ISO179-1. In addition, a V-notched, which is 0.1 radii following ISO 294-1 were created on the samples. The mean and standard deviation values were obtained from at least five samples for each sample. All the impact tests were implemented at room temperature.

The Shore-D hardness measurements of samples were carried out following the ISO 868 standard using an analog hand-held Zwick/Roell hardness device.

The VST tests following ISO 306-B120 were performed at silicone oil bath through an HDT Vicat Testing device (CEAST HV3, USA), with a temperature ramp of 2 °C/min. The results were presented by calculating the mean and standard deviation values.

The samples' crystallinity index, melting temperature, and crystallization temperature were investigated by means of the DSC (Seiko 7020, Japan). The samples were placed in aluminum sample pans. After that, the samples were heated from 50 to 250 °C at the heating rate of 10 °C/min, and the samples were cooled down to 30 °C at the cooling rate of 10 °C/min in order to erase any previous thermal history. All measurements were carried out in a nitrogen atmosphere. After the isothermal crystallization, the samples were heated immediately to 200 °C at 10 °C/min heating rate to obtain melt samples. Then, the second heating curves were obtained. The crystallinity index (Xc) was determined according to Equation (1):

$$\mathrm{X_c}\% = \frac{\Delta \mathrm{H_m}}{\Delta \mathrm{H^0}_{100\%}(1-\theta)} \mathrm{x}\,100 \tag{1}$$

where ΔH_m is the sample fusion enthalpy measured from DSC; ΔH^0 is i-PP fusion enthalpy with 100% crystallinity degree, and its value has been assumed as 209 J/g, and θ is the weight fraction of inclusions in the matrix [22, 28]. The lamellar thickness distribution was calculated using the general Thomson- Gibbs equation for samples through the DSC melting curves. This relationship was expressed in Equation (2):

$$\mathrm{T_m} = \mathrm{T_m^0}\left(1 - \frac{2\sigma_e}{\mathrm{l}\,\Delta \mathrm{H_f}}\right) \tag{2}$$

where Tm; melting temperature of the PP, Tm; the equilibrium melting temperature, l; lamella thickness in the longitudinal dimension of the crystal, ΔHf; the melting enthalpy of the perfect crystal, σe; free surface energy of the end faces at which chains fold.

TGA curve of samples was obtained using Seiko 6300 model instrument, with nitrogen at a flow rate of 100 ml/min, and the samples were heated from 35 to 610 °C at a rate of 10 °C/min.

The fracture surface's morphology was viewed employing scanning electron microscopy (SEM) (Inspect, S-50) with an accelerating voltage of 20 kV after coating with a gold layer of 10 nm. Also, the fracture surfaces between the corresponding zones were compared to almost the same region for the samples with and without magnesium silicate monohydrate.

3. Results and Discussion

3.1. DSC Characterization

While DSC melting behaviors of neat i-PP and NA-added samples are presented in Figure 1a, crystallization behaviors are presented in Figure 1b and the melting temperature (Tm), crystallization temperature (Tc), ΔH_m, Xc%, and λ values obtained from the curves are presented in Table 1. When the curves were examined, it was found that the melting temperatures of NA-added samples were similar to those of neat i-PP. The changes in Tm were independent of the amount of NA, and NA had scarcely any effect on the melting temperatures of the samples. However, the effect of NA on Tc was apparently remarkable. The Tc value of NA-added samples was measured to be higher by 14.5-16 °C compared to neat i-PP. In Figure 1b, it is clearly observed that the exothermal peak of the DSC curves changed and expanded in longer periods of time along with the increase in Tc temperature. It is known that the initiation of crystallization at higher temperatures will improve the dimension accuracy of the materials and increase the production efficiency by shortening the cycle time of the product in molding [29-31]. When Table 1 is examined, it can be observed that there is an increase in Tc with the increasing ratio of NA. When the ratio of NA in the structure was >1.5%, a decrease was observed in Tc. It is known that this behavior is affected by different parameters such as exceeding the ideal ratio of NA, high nucleation, additive agglomeration, number of nuclei, and distribution in the microstructure [30,32-35].

When a general evaluation is made according to the Tc values obtained, it can be concluded that the increase in crystallization temperatures was efficient in NA-added samples. Although the increase in Tc values among NA-added samples was low, it does not lead to the conclusion that NA was not homogeneously distributed within the structure. However, this can be attributed to the fact that the maximum performance is obtained from the ratios of NA between 1.5 – 3 wt.%. In conclusion, it can be said that NA improved the crystallization behavior of i-PP and improved its thermal stability [36].

It was observed that NA had a limited effect on the %Xc and λ values. It was observed that the structure became stable independently of the spherulite sizes with the NA ratio between 1.5 – 3 wt.% and that the Xc% values were close to each other. Despite the closeness in crystallization rates, the differences in Tc and Tm temperatures were considered to be affected by different λ and crystal forms occurring during the recrystallization of i-PP from the melt state, depending on the NA ratio [36]. The length of the i-PP molecular chains and their molecular weight distribution also affected this situation. It is known from similar studies that the nucleus formation mechanism affects the crystallization properties, as well as the size and number of spherulite that differ depending on the NA ratio [30-36]. It is predicted that the decrease in the Xc% rate of the PP/NA2.5 coded sample was due to experimental conditions.

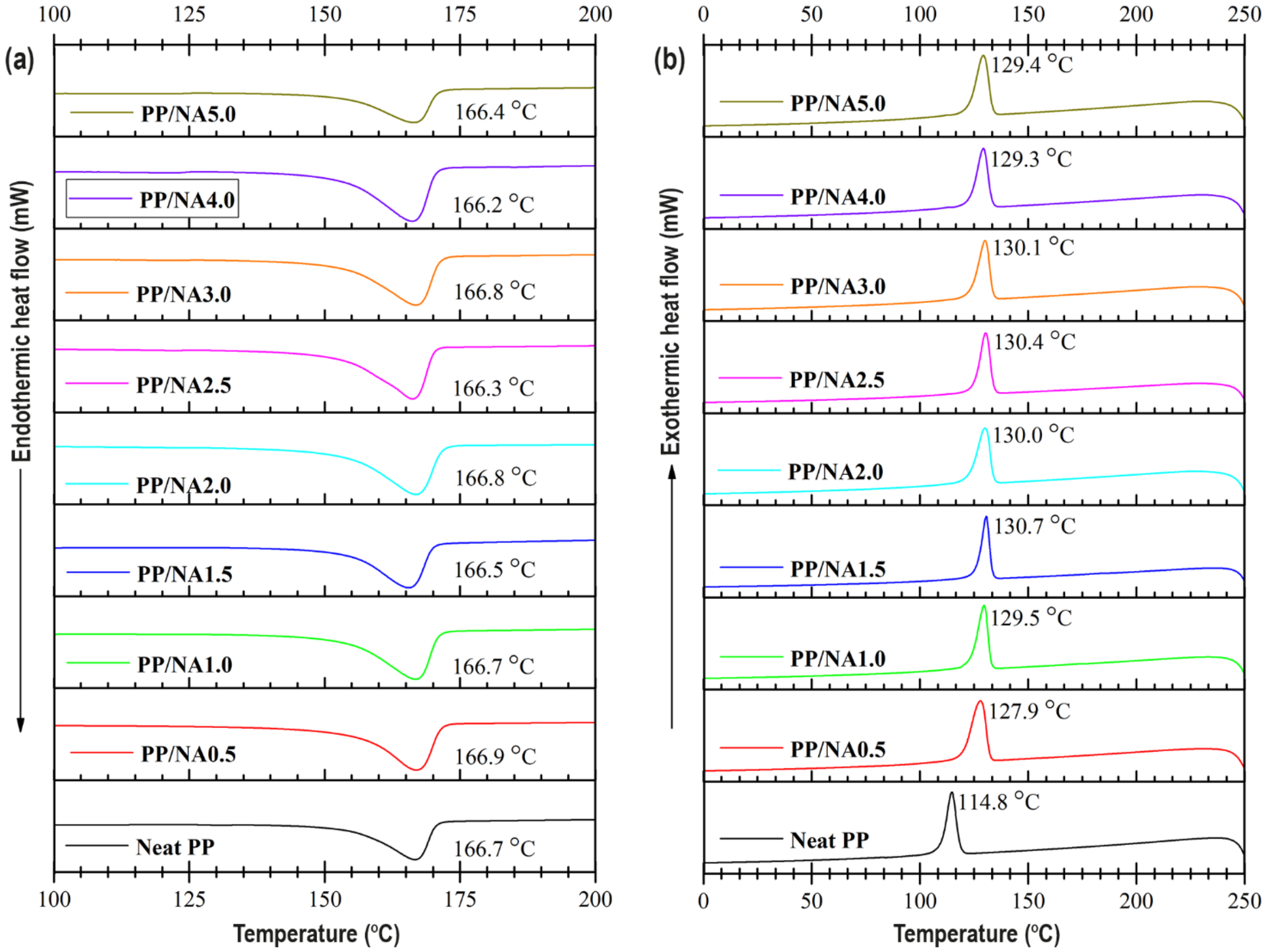

Figure 1. DSC curves a) melting curves b) crystallization curves

3.2. HDT and VST Characterization

The values of DSC, HDT and VST are presented in Table 1. According to the ratio of NA additive, HDT temperatures and VST temperatures were found to be higher between 13-32% and between 2-7%, respectively. It was clearly observed that the NA additive increased the thermal stability of the samples, as in DSC results. HDT measurements can also be expected to provide information about phase topologies as a function of NA [38], which can be observed more clearly in SEM examinations. It is known from the literature that the crystal formation mechanism, crystal size and crystal lamella thickness positively affect the mechanical properties of the materials and also increase HDT/VST temperatures [32,38-44]. In this study, the ideal ratio of the NA additive for HDT and VST values was determined as 2 wt.%. The operating temperatures can be increased with NA additives, especially for the products to be used in applications where the temperature is important by considering the material thickness and the desired mechanical properties.

Table 1. Thermal test results of the samples

Sample Codes	HDT (°C)	VST (°C)	ΔH_m (J/g)	T_c (°C)	T_m (°C)	X_c (%)	λ (nm)
Neat PP	53.40 ± 0.60	103 ± 1.02	91.3	114.8	166.7	43.68	15.14
PP/NA0.5	60.67 ± 0.16	106 ± 2.62	82.4	127.9	166.9	39.62	17.25
PP/NA1.0	62.87 ± 1.38	108 ± 0.49	92.4	129.5	166.7	44.66	14.96
PP/NA1.5	65.80 ± 1.60	109 ± 1.36	86.1	130.7	166.5	41.82	13.79
PP/NA2.0	70.60 ± 0.80	110 ± 0.09	85.3	130.0	166.8	41.65	16.43
PP/NA2.5	68.53 ± 1.62	109 ± 0.91	69.3	130.4	166.3	34.00	18.91
PP/NA3.0	68.77 ± 1.24	107 ± 0.71	84.7	130.1	166.8	41.78	16.55
PP/NA4.0	68.97 ± 1.49	107 ± 0.36	82.6	129.3	166.2	41.17	15.66
PP/NA5.0	69.20 ± 2.07	105 ± 1.13	76.9	129.4	166.4	38.73	17.27

3.4. Mechanical Properties

The experimental results of the relevant mechanical tests for different NA ratios are collectively presented in Table 2, the curves of the tensile tests are presented in Figure 2a and the curves of the bending test are presented in Figure 2b. According to the tensile strength results, the tensile strength of NA-added materials was higher compared to neat i-PP. When the tensile modulus of elasticity was compared, it was obtained to be higher in NA-added samples compared to the material without the addition of NA. The tensile modulus of elasticity in NA-added samples was similar. The elongation at break of neat i-PP was 30.7%. The elongation at break was 22.2%, 17.2%, 21%, 15.9%, 15%, 14.3%, 9.3% and 8.3%, respectively, in the samples added with NA by 0.5%, 1%, 1.5%, 2%, 2.5%, 3%, 4% and 5%. The toughness values of the materials also decreased with the elongation at break, and the change in toughness (Work up to break) supported the change in the elongation at break values. The flexural strength of the NA-added samples was obtained higher compared to neat i-PP. When a general evaluation was made, with the addition of magnesium silicate monohydrate as NA to i-PP, the tensile modulus, tensile strength, flexural modulus, and flexural strength and hardness increased and the work up to break and notched Charpy impact strength values decreased.

Table 2. Mechanical test results of the samples

Sample Codes	Tensile Strength (MPa)	Tensile Modulus (MPa)	Flexural Strength (MPa)	Flexural Modulus (MPa)	Toughness (Nmm)	Impact Strength (kJ/m^2)	Hardness (Sh. D)
Neat PP	31.8 ± 0.21	1272 ± 29	43.6 ± 0.48	1270 ± 47	26243	2.42 ± 0.03	72.5
PP/NA0.5	35.2 ± 0.06	1408 ± 41	47.8 ± 0.89	1540 ± 47	22884	2.34 ± 0.01	74.6
PP/NA1.0	35.0 ± 0.32	1400 ± 28	53.2 ± 0.77	1620 ± 39	17852	1.80 ± 0.03	75.7
PP/NA1.5	35.1 ± 0.21	1404 ± 30	54.3 ± 0.60	1780 ± 40	22820	1.78 ± 0.02	74.8
PP/NA2.0	35.4 ± 0.06	1416 ± 28	53.8 ± 0.56	1740 ± 12	15398	1.56 ± 0.01	75.4
PP/NA2.5	35.2 ± 0.07	1408 ± 92	53.2 ± 0.52	1720 ± 55	16739	1.52 ± 0.02	75.9
PP/NA3.0	35.2 ± 0.11	1408 ± 92	53.7 ± 0.17	1720 ± 40	14981	1.54 ± 0.01	75.6
PP/NA4.0	35.1 ± 0.23	1404 ± 66	55.1 ± 0.13	1800 ± 25	8911	1.55 ± 0.01	74.8
PP/NA5.0	34.5 ± 0.06	1380 ± 41	54.6 ± 0.36	1800 ± 36	7141	1.57 ± 0.02	74.0

The results obtained in the mechanical properties were compatible with the crystallization properties. It is known that the mechanical properties of i-PP in crystalline structure depend on the degree of crystallinity, the size of spherulites, and the uniformity of their distribution [31, 32, 36]. The addition of NA to the structure increases the number of nuclei, reduces the spherulite size, accelerates crystallization, and ensures that the lamella size and thickness are similar, which explains the phenomenon in obtaining better mechanical properties. Contrary to expectations, it was considered that the decrease in impact strength and toughness was due to the decrease in boundary forces between spherulites and the incompatibility of interface properties [36].

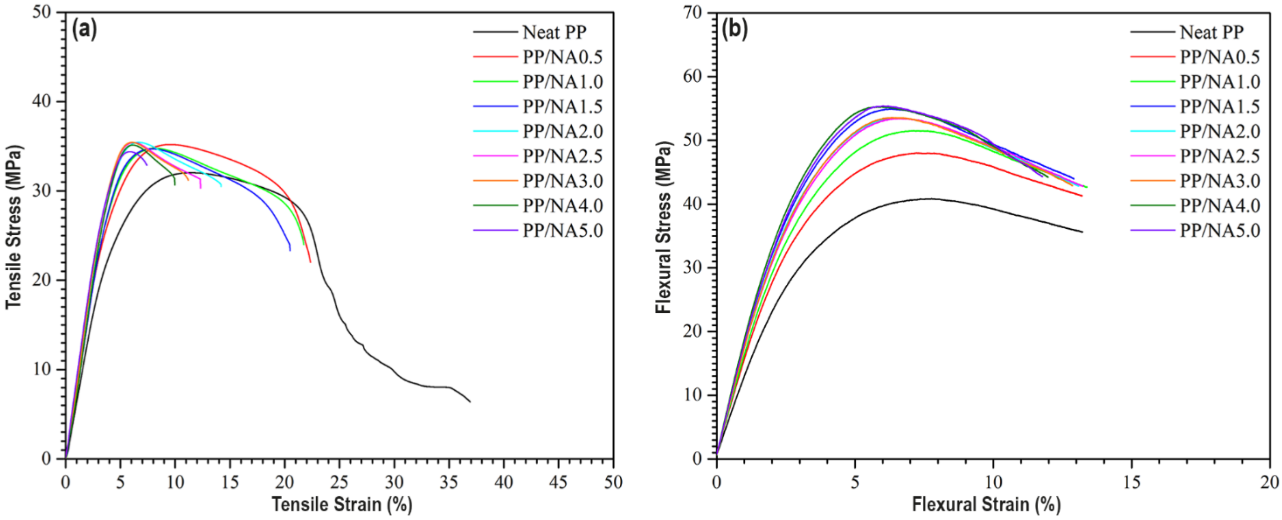

Figure 2. Tensile (a) and flexural (b) stress-strain curves

3.3. TGA Characterization

TGA curves of neat i-PP and NA added samples are given in Figure 3. The TGA results in Figure 3 also confirm the stabilization ability of magnesium silicate monohydrate toward i-PP. Increasing the magnesium silicate monohydrate content between 2-3 wt.% led to a remarkable improvement in the initial decomposition temperature (T_i). Compared with the neat i-PP, the T_i improves by about 5-6 °C at best for the PP/NA2.0, PP/NA2.5, and PP/NA3.0 samples.

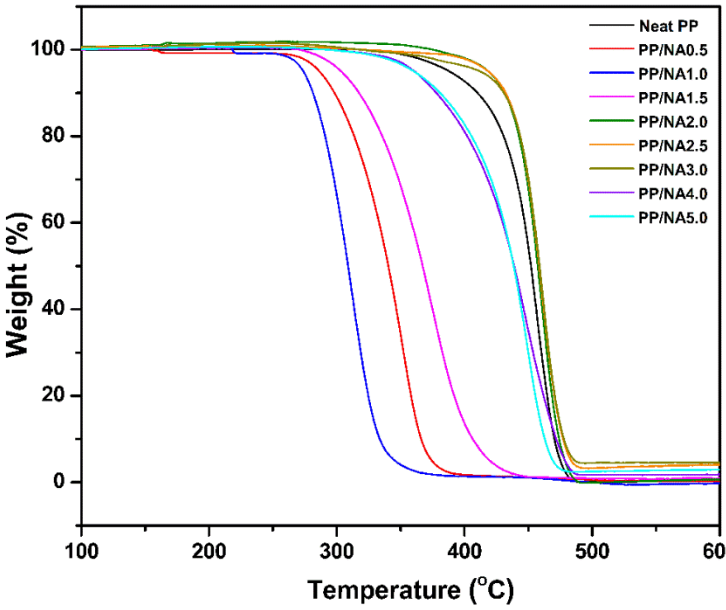

Figure 3. TGA curves

3.5. Morphological Properties

When the SEM images of the broken surface after the impact test in Figure 4 (x200) and Figure 5 (x2000) were examined, it was observed that the fracture surfaces of i-PP were relatively smooth and had a brittle appearance. While the progression of the fracture surface was quite distinct and in large steps in neat i-PP, it was in smaller steps in NA-added samples. Although the fracture was macroscopically brittle for the NA-added samples, these samples were observed to have a rough surface and grainy appearance in SEM observations. The presence of NA in i-PP, the homogeneous distribution of NA in the structure, forcing the lamellae to orientation, and its effect on the microstructure are clearly observed from the SEM images. The microstructure images and fracture progression behavior of neat i-PP and PP/NA0.5 coded sample, which had the highest impact strength among NA-added samples, were similar. The micro-voids in the structure of neat i-PP and PP/NA0.5 coded samples in Figure 5, and the rupture progression in large steps indicated the evidence of significant impact energy absorption and resistance to rupture, which explains why the impact strength of neat i-PP and PP/NA0.5 coded samples was higher compared to other NA-added samples. When a general evaluation was made, it was observed that the fracture surface images were compatible with the mechanical test results and that the presence of NA affected the microstructure, crystal formation and forced orientation. These results are consistent with those previously reported in the literature [19, 36].

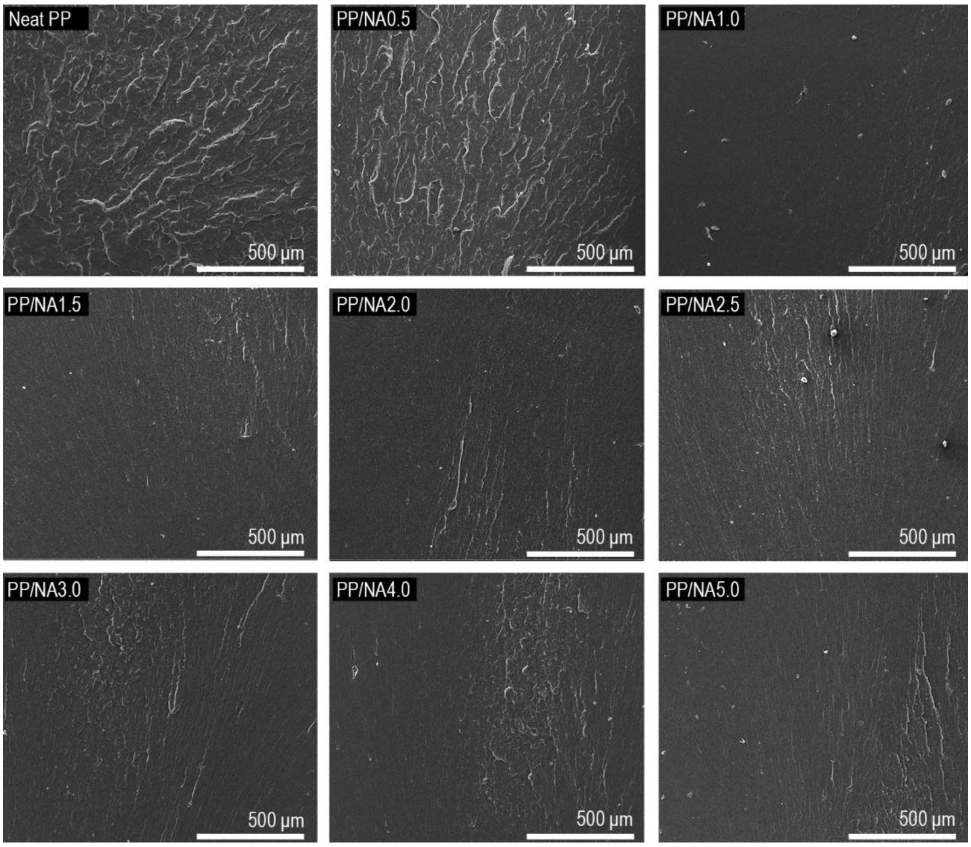

Figure 4. SEM photomicrographs of Charpy impact test fracture surfaces of neat PP and NA added PP samples (x200)

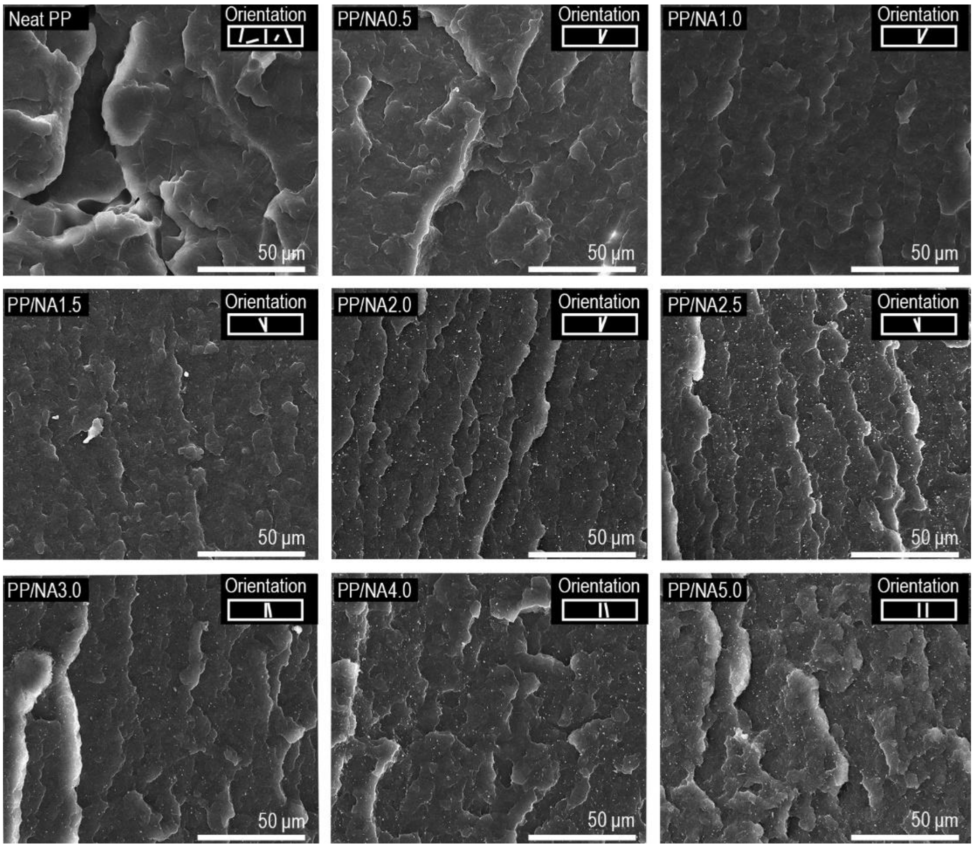

Figure 5. SEM photomicrographs of Charpy impact test fracture surfaces of neat PP and NA added PP samples (x2000)

2. Conclusions

The changes that occurred in the mechanical and thermal properties of i-PP when talk (magnesium silicate monohydrate) was added to i-PP as a nucleating agent were investigated in this study. The results showed that magnesium silicate monohydrate was an effective nucleating agent. The addition of magnesium silicate monohydrate to the polymeric structure forced the polymer to orientation and improved the crystallization rate and temperature. Delaying the softening of the polymer increased softening temperatures and thermal deformation temperatures and increased the thermal stability of the polymer. The mechanical properties of i-PP are related to its crystallization properties. NA-added i-PP had an average of 10% higher tensile strength and an average of 22% higher flexural strength compared to neat i-PP. It is considered that the addition of additional additives such as maleic anhydride to the structure, which will increase the boundary forces of spherulites and improve the interface properties between i-PP and magnesium silicate monohydrate, will ensure achieving higher toughness, impact resistance and mechanical properties.

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Authors' contributions

Conceptualization, Sample preparation, characterization progresses, methodology, writing original draft preparation contributed MK and İK; Made literature search, writing - review and editing

contributed İK and HS; Funding acquisition contributed İK and MK; Resources contributed MK, İK and HS; Supervision contributed İK.

Both authors read and approved the final manuscript.

Competing interests

The authors declare that they have no competing interests.

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