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Research Article

The effect of various mineral fillers on thermal, mechanical, and rheological properties of polypropylene

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Abstract

Polypropylene is one of the commodity thermoplastics that is widely used in many different applications such as automotive, building, electric-electronics, textile, and packaging industries due to its properties and favorable cost-benefit ratio. The incorporation of mineral fillers such as mica, talc, wollastonite, and calcium carbonate into thermoplastics is a common practice in the plastics industry in order to reduce the production costs of molded products and enhance processibility, mechanical, and thermal properties. Mineral filled polypropylene composites provides high mechanical stiffness, thermal stability, and good dimensional stability over a wide temperature range. The filler weight ratio, type, size, and dimension have significant effect on mechanical, thermal, physical, and rheological properties of composites. Four different mineral fillers (talc, mica, calcite, and feldspar) at weight fractions of 40% were compounded with polypropylene by using a twin-screw extruder. Test specimens were obtained by injection molding process. The effect of various filler types on rheological and mechanical properties of polypropylene was investigated in this study. It was found that talc and mica loadings at 40 wt. % into polypropylene increased the flexural strength of polypropylene. The rheological properties of samples were more affected by the talc than calcium carbonate.

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1. Introduction

Polypropylene (PP) is one of the commodity thermoplastics that is used in various industrial applications including, but not limited to, automotive, electronics, construction, household appliances, films, fabrics, sheets, etc. Being lightest polymer with a very low density, polypropylene shows excellent mechanical properties and can be reprocessed several times without significant loss of its mechanical, physical and thermal properties [1-9]. Polypropylene based composite materials provide low-cost solutions to the needs of the many engineering problems. The biggest advantage of using PP as a matrix material is its compatibility with many fillers that will change its structure to have properties set for specific applications. That is to say, the incorporation of proper fillers into PP has been an accepted route to cost saving and enhancing material properties [1-3]. Among many filler types, metal powders, ceramics, carbons, and minerals have been investigated as promising fillers for thermoplastics-based composites. The studies about functional thermoplastics have shown that stiffness, durability, and dimensional stability could be improved by combination of these types of fillers [10].

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Talc is one of the preferred mineral fillers, with platelet structure and low hardness, which improves processing properties and reduces costs [11]. Addition of talc in thermoplastics generally results in improvement in mechanical properties, but it decreases impact strength [5, 12]. Furthermore, talc, which reduces and homogenizes the molding shrinkage, also facilitates the shaping of polypropylene. The percentage of talc as a filler in thermoplastics is usually between 20 and 40 wt. % [13]. Mica also has a layered crystal structure, but the bonds between the layers are stronger than the talc. It is particularly used in large proportions in thermoplastic polymers to provide mechanical reinforcement along a plane with excellent electrical characteristics [14]. Calcium carbonate, referred as calcite, which can be produced from chalk, limestone and marble, is one of the most widely used mineral in the end-use industries (plastic, paper, adhesive, sealants, coatings, and paints etc.) as a filler. Calcite is preferred as a mineral filler in large number of thermoplastics due to its low cost, good stability, white color, easy processing and many other advantages [14]. Feldspars, forming about 51% of the rocks on Earth's crust, are the most abundant group of tectosilicate minerals. The use of feldspar in thermoplastics has attracted great attention because of its cost efficiency and abundance. Feldspars have a cubic shape, high energy adsorption capacity and are capable of exchanging ions on silicate layers with reactive groups on the surface. Also, due to their optical and thermal properties, feldspars are preferably used in light and thermal management applications[15].

The aim of this article is to investigate the effects of four different types of mineral fillers on the tensile, flexural, impact, thermal and rheological properties of PP based composites. For this reason, talc, calcite, mica and feldspar at 40 % weight fraction were used in polypropylene during compounding with a twin-screw extruder. Mineral filled polypropylene composites test specimens were obtained by injection molding of composite granules. The effects of the filler loading on the physical, mechanical, thermal and rheological properties of mineral filled polypropylene composites were investigated by several characterization methods.

2. Materials and Methods

2.1. Materials

Homo-polymer-grade polypropylene (PP) (IMS Polymers, Turkey) resin with melt flow index of 25 g/10 min used in this study. Four types of minerals that were incorporated into PP were talc, calcite, mica and feldspar. Talc and calcite with a density of 2.78 g/cm³ and 2.71 g/cm³, respectively were purchased from Omya Mining, Turkey. Mica and feldspar with a density of 2.81 g/cm³ and 2.62 g/cm³, were obtained from Mikron'S, Turkey and Şişecam, Turkey, respectively.

Bulk densities and chemical compositions of the fillers provided by the suppliers are given in Table 1. Talc is a natural fine powder with its 42% SiO₂ and 29% MgO content. Feldspar is naturally occurring aluminum silicate as it is understood from its chemical composition. Calcium carbonate powder obtained from high purity white marble and its CaCO₃ content is around 98.5%. Feldspar has mostly 70% SiO₂, 18.5% Al₂O₃ and 10.5% Na₂O content. Mica is produced from deposits with high quality SiO₂ and Al₂O₃.

Table 1. Bulk density and chemical composition of the fillers.

Filler	Bulk Density (g/cm ³)	Chemical Composition (%)								
		SiO ₂	MgO	CaO	CaCO ₃	MgCO ₃	Al ₂ O ₃	Na ₂ O	Fe ₂ O ₃	K ₂ O
Talc	0.55	42	29	9	-	-	-	-	-	-
Calcite	1.3	-	-	-	98.5	1.5	-	-	-	-
Feldspar	0.61	70	0.1	0.7	-	-	18.5	10.5	0.02	-
Mica	0.31	53.0	-	-	-	-	29.5	-	1.13	9.1

2.2. Manufacturing of Compound

PP and mineral fillers at 40 wt. % were compounded with twin screw extruder at different barrel temperatures (195-215 °C) and screw speeds (200-300 rpm). The amount of each mineral added to the extruder was controlled by the help of gravimetric feeders (Figure 1a). The extrusion run condition for the all the composite formulations were kept the same. Polymer strands with a diameter of 3 mm were obtained after mineral filled polymer melt passed through the extruder die. Then, following the water bath, composite granules were produced by pelletizer. The water bath used during compounding process was kept at 16 °C. Figure 1b shows a picture of the water bath and the pelletizer. In order to produce test specimens injection molding machine was used (Figure 1c). The injection molding conditions for each test sample were kept constant as long as the samples could be molded.

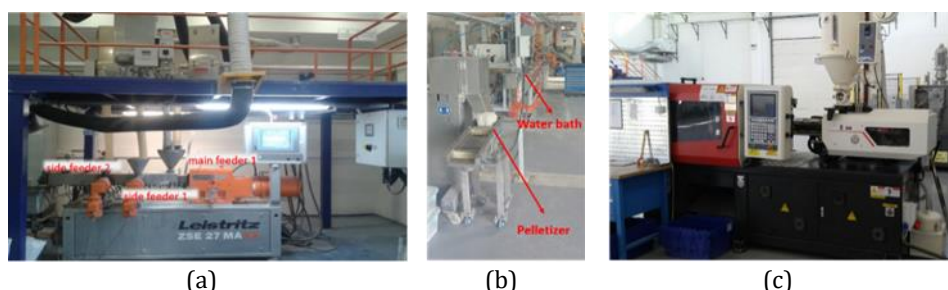


Fig. 1. a) twin screw extruder and b) water bath and pelletizer c) Injection molding

2.3. Characterization Methods

In this study, the effects of various minerals on density, tensile properties, flexural properties, impact properties, melt flow index, rheological properties, vicat softening temperature, melting and crystallization and decomposition temperatures, and thermal expansion coefficient of mineral filled PP composites were examined. Density measurement of the mineral filled PP composite specimens was done using Densimeter MD-200S by following the ISO1183 standard [16]. Hegewald & Peschke Inspect 20 tensile-compression testing machine was used to investigate the tensile and flexural properties. Tensile tests were done according to the ISO 527 standard with 50 mm/min and non-contact video extensometer [17]. Three-point bending tests were done with a 1 mm/min deformation rate according to the ISO 178 standard for flexural properties [18]. Izod notched and un-notched impact strengths of the samples were determined according to ISO 180 standard by using a pendulum-type tester [19]. Göttfert Melt indexer mi2.2 was used to measure melt flow index (MFI) of the samples. The used melt temperature and piston load were 230 °C and 2.16 kg, respectively during the MFI tests according to ASTM D-1238 standard [20]. Capillary rheometer (Göttferd, Rheograph 20) was used to determine the viscosity of PP and mineral filled polypropylene composites. The vicat softening temperature was measured according to the ISO 75-1 standard with an applied

load of 10 N in the testing apparatus [21]. Differential scanning calorimeter (DSC-Q20, TA Instruments, Inc.) and TG Analyzer (TA Instruments, Inc., TGA-Q50) were used in order to investigate thermal properties of polypropylene and its composites. Both analyses were done under nitrogen environment with a heating rate of 10 °C/min. Thermo-mechanical analyzer (TA Instruments, Inc., TMA 400) was used to determine the thermal expansion coefficient of the samples.

3. Results and Discussion

3.1. Density

Density values of PP and mineral filled polypropylene composites are given in Figure 2. The density of the talc, calcium carbonate, mica and feldspar is relatively higher than that of PP (0.900 g/cm³); thus, it can be said that the incorporation of minerals into PP increases the density of the neat PP. Since feldspar is found to be 6% lighter compared to talc, it shows promise to be used in various applications where being lightweight is significantly important.

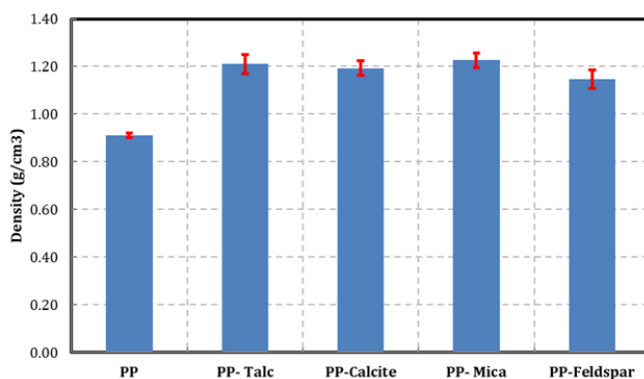


Fig. 2. The effects of mineral types on density of mineral filled PP composites

3.2. Mechanical Testing

Table 2 shows the effect of mineral types on mechanical properties of PP and mineral filled PP composites. The tensile and flexural strength of the PP were about 31 and 39 MPa, respectively. Tensile strength values of PP-Talc, PP-Calcite, PP-Mica and PP-Feldspar were obtained to be 30, 21, 29, and 21 MPa, respectively. It can be seen from Table 2 that talc and mica filled PP composites have not led to considerable variation in tensile strength value of PP. While talc and mica loadings into PP increased the flexural strength of PP, calcium carbonate and feldspar loadings decreased the flexural strength of PP. One can say that the polymer matrix could wet the fillers in a better way since talc and mica have a high aspect ratio and layered silicate structures[22]. This has been stated before that this phenomenon creates fewer micro-voids between the fillers and matrix [23, 24] resulting enhanced wettability. Calcium carbonate and feldspar loading into PP decreased the tensile and flexural strengths of PP remarkably due to relatively low aspect ratio of calcium carbonate and feldspar compared to talc and mica. PP-Talc and PP-Mica composites showed increased tensile strength values as compared to PP-Calcite and PP-Feldspar composites. This improvement shows better surface interactions between filler and matrix, which results in better stress transfer from matrix to the filler under high loadings.

As shown in Table 2, flexural and tensile moduli of PP increased with mineral filler loadings. The increase in tensile and flexural properties could be because of the reduced mobility and limited deformability of the matrix as a result of filler incorporation[22]. The limitation on the mobility of polymer chains decreases the stress transfer from the whole composite which in turn causes increase in rigidity and stiffness. An increase in flexural and tensile moduli by a maximum of about 257% and 268 %, respectively, was observed in PP-Mica composites. Also, larger mica particles with rigid molecular structure compared to smaller talc, calcium carbonate and feldspar particles could have resulted in worse stress transfer in mica filled PP based composites.

As can be observed from Table 2, impact strength values decreased when mineral fillers were used in PP composites. The un-notched and notched Izod impact strength of the PP was 54.16 and 5.79 kJ/m², respectively. Un-notched Izod impact strength for the PP-Talc, PP-Calcite, PP-Mica and PP-Feldspar composites decreased by 71, 61, 69, and 13% compared to the PP, respectively. When compared to PP without any mineral fillings, 40, 36, 32, and 10%, decreases in notched Izod impact strength values were obtained for PP-Talc, PP-Calcite, PP-Mica and PP-Feldspar, respectively. It can be noted that a considerable decrease in impact strength values were obtained. The less decrease in impact strength was obtained by loading feldspar rather than other fillers. In other words, feldspar is better than talc, calcium carbonate and mica in absorbing the impact energy [25].

Table 2. The effects of mineral types on mechanical properties of mineral filled PP composites

	Tensile Properties		Flexural Properties		Izod Impact Properties	
	Tensile Strength (MPa)	Tensile Modulus (MPa)	Flexural Strength (MPa)	Flexural Modulus (MPa)	Un-notched Impact Strength (kJ/m ²)	Notched Impact Strength (kJ/m ²)
PP	31.1±2.1	1200±65	39.1±2.3	1167±57	54.2±2.7	5.8±0.4
PP- Talc	30.3±1.7	2673±72	54.3±3.2	3540±62	15.7±2.0	3.5±0.3
PP-Calcite	20.9±1.5	2879±78	38.4±2.1	3806±69	21.0±1.8	3.7±0.4
PP- Mica	29.4±1.5	4417±102	48.2±2.8	4168±75	17.0±1.1	3.9±0.4
PP- Feldspar	21.2±1.4	2768±85	40.1±1.6	3782±82	47.3±2.5	5.2±0.4

3.3. Rheological Analysis

The rheological behavior of PP is highly affected by mineral fillers. It is expected that incorporation of minerals creates higher viscosity of PP melts compared to the pure PP matrix. The melt flow index (MFI) of mineral filled PP based composites are shown in Figure 3. As shown from Figure 2, lower MFI values in mineral filled composites were obtained compared to that of neat PP. This is because of the fact that when minerals are added to the polymer, viscosity of the polymer matrix increases which restricts polymer flow and decreases the value of MFI [23]. Among all the minerals that were used in this study, calcium carbonate and feldspar filled PP composites demonstrated higher MFI values than that of the other composites. One can say that, calcium carbonate and feldspar have the ability to increase the processability of the polymer. As can be seen, mica gave a slightly lower MFI than calcium carbonate and feldspar and lowest MFI was obtained when talc was the filler in PP composites.

The literature shows that due to their platelet/lamellar structure, talc and mica particles at lower weight fractions could slide against each other during the application of shear forces which increases flowability in the composites. Nevertheless, MFI values of talc or

mica filled PP composites were obtained to be low when minerals are used at 40 wt.% in the (Figure 3). It can be contributed to that, this ability depends on the location of the filler particles in the die which has significant effect on resulting MFI of the composites [23].

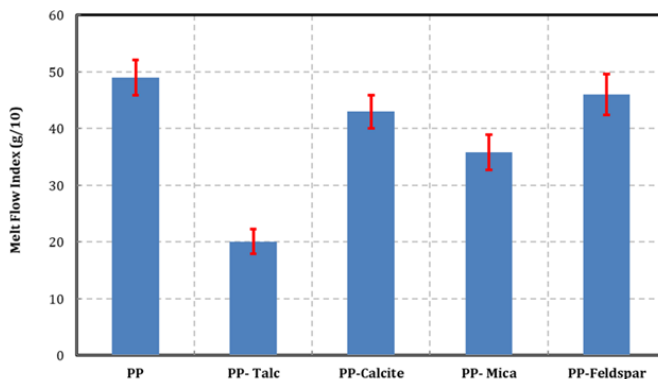


Fig. 3 MFI values of mineral filled PP composites

Change in viscosity values of PP and mineral filled PP composites as a function of shear rate is given in Figure 4. From Figure 4, decreasing trend on viscosity of the composites was seen with increasing shear rate. Especially at high shear rates, the lowest viscosity value was obtained in feldspar filled PP composites, which explains the highest MFI value in PP-Feldspar sample. It is known that at high shear rates due to broken structure of the composite, hydrodynamic interactions have a significant effect on the viscosity instead of filler particles[26].

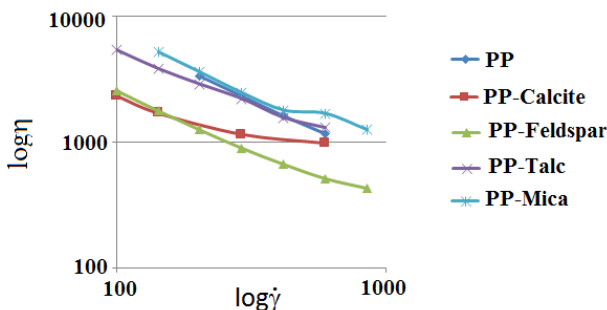


Fig. 4 log η vs. log γ̇ plots of PP and mineral filled composites

The Power Law model is used to describe the rheological behavior of the apparent viscosity decreasing as the shear rate increases (shear thinning)[27]. The Power Law model is defined by

$$\eta = k\dot{\gamma}^{(n-1)} \tag{1}$$

where k is the consistency index and n is the power-law index. When n < 1 the fluid is called pseudoplastic and when n > 1, it is called dilatant [28].

The Power Law Index (n) was derived from the slope of the graphs for the logarithmic viscosity and the logarithmic shear rate [29]. Consistency index and power law index values of samples are given in Table 3. It is seen that calcite, feldspar, talc and mica filled PP composites exhibited more temperature sensitive than PP. Due to low power law index values, melt viscosity of PP-Feldspar, PP-Talc, and PP-mica changes more with increasing or decreasing shear rate than by changing the melt temperature.

Table 3. Power Law Index (n) values for PP and its composites

Sample	k-Consistency index	n-power law index
PP	6.70E+05	0.01
PP- Calcite	1.80E+04	0.52
PP-Feldspar	1.19E+05	0.15
PP-Talc	2.10E+05	0.20
PP-Mica	2.40E+05	0.21

3.4. Vicat Softening Temperature Tests

Vicat softening temperature is the point of softening when material is used at elevated temperatures. This temperature could be used to determine the potential of the material to be used as a useful product for practical applications [30]. Figure 5 shows the characteristics of vicat softening temperature with mineral types in the mineral filled PP composites. As shown in Figure 5, vicat softening temperature increased with mineral loading. It can be said that improved heat softening characteristics could be achieved in the case of mineral filled polypropylene composites. The low thermal conductivity of mineral fillers may be the reason for the increase in the vicat softening temperature of mineral filled polypropylene composites[30].

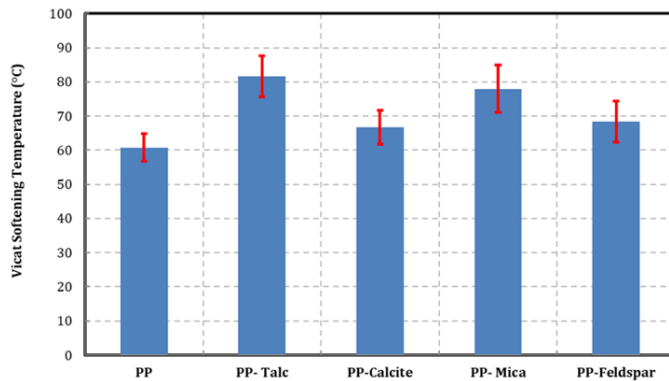


Fig. 5. Vicat softening temperature of PP and mineral filled composites

3.5. DSC Analysis

DSC analysis was performed to determine the melting and the crystallization behaviors of PP and mineral filled PP composites. With the DSC analysis, a graph of the heat flow for each sample is drawn depending on the temperature and is given in Figure 6. The melting and crystallization temperatures, the melting and crystallization enthalpies and degree of crystallinity obtained from the graph are given in Table 4. Degree of crystallization (X_c) was calculated from Equation given below

$$X_c(\%) = \frac{\Delta H_m}{(1-w)\Delta H_{0m}} \times 100 \quad (2)$$

where w is the weight fraction of mineral fillers and ΔH_{0m} is the melting enthalpy value of 100% crystalline form of PP (209 J/g) [31]

It could be seen from Table 4 that the melting temperatures (T_m) decreased, and the crystallization temperatures (T_c) increased together with the addition of mineral fillers to PP composites. However, this increase and decrease were not very pronounced with mica, calcite and feldspar fillers, although the melting temperature remained constant with the addition of talc, there was an increase of 9 °C in the crystallization temperature. So it could be said that talc acts as a nucleating agent in PP composites thus the crystallization temperature of talc filled PP composites increased [32]. When melting and crystallization enthalpies were examined, it could be seen that there was a decrease in energies in both cases compared to that of polypropylene. The reason for the decrease in melting enthalpy could be the mineral fillers absorb more heat energy during the melting of composites so the melting enthalpy than the neat PP [33]. The biggest difference was seen with the addition of 40 wt. % by weight of mica. When it was focused on the degree of crystallization (X_c) of neat PP and its composites it was observed that the X_c value of the PP is 29.57 %. The crystallinity of polypropylene changed irregularly with the addition of mineral filler. Mica and feldspar decreased the X_c while calcite and talc increased. This situation can indicate that X_c is generally affected by dispersion, loading level or surface chemistry of fillers. This was consistent with the studies in literature [32, 34, 35].

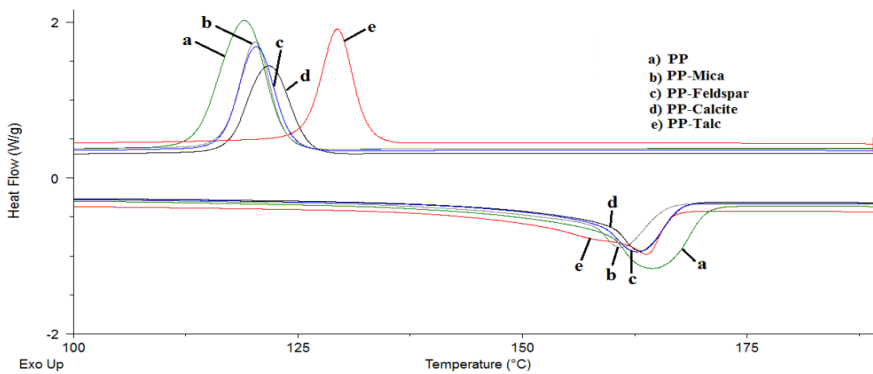


Fig. 6. DSC curves of PP and mineral filled PP composites

Table 4. DSC results for PP and mineral filled PP composites.

Sample	$T_m (^{\circ}C)$	$T_c (^{\circ}C)$	$\Delta H_m (J/g)$	$\Delta H_c (J/g)$	$X_c (%)$
PP	164	119	61.2	64.3	29.57
PP-Mica	161	120	34.3	38.3	27.62
PP-Calcite	163	122	37.8	40.1	30.43
PP-Feldspar	163	120	35.9	40.7	28.90
PP-Talc	164	129	40.0	42.9	32.21

3.6. TG Analysis

TG analysis was performed on the samples to determine the maximum degradation temperature, temperature of 5% of mass loss and the weight loss due to temperature change in PP and its composites. TG and DTG curves of the samples are given in Figure 7.

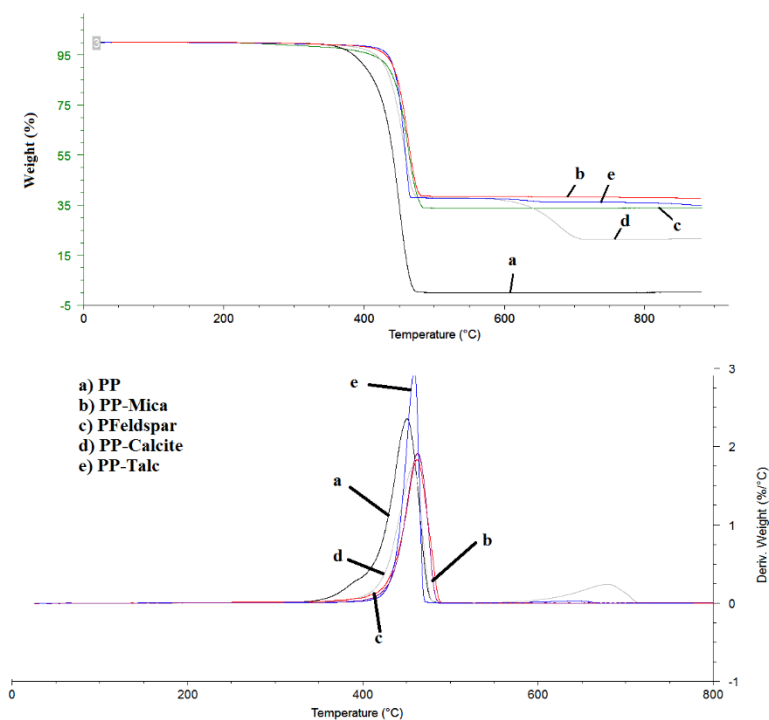


Fig. 7. TG/DTG curves for PP and its composites

Table 5 shows the maximum decomposition temperature obtained with the help of graph and the remaining weight without degradation after the temperature of 800 °C. From Table 5, it was seen that the decomposition temperature of PP increased when mineral fillers were used in PP composites. The largest increase was 22 °C with the addition of mica as a filler to PP, while the least increase was with the addition of calcite. It could be said that the mineral fillers can be improved the thermal stability of PP composites. The increased thermal stability can be attributed to the impeded diffusion of volatile degradation products in composites. Qin et al. have similar studies in the literature [36]. According to these results it was seen that most stable composite was mica-filled PP, while the least stable was calcite-filled PP. On the other hand, while PP lost 5% of its mass at 384 °C, this

temperature increased with the addition of minerals. Although the highest decomposition temperature was obtained with the addition of mica, the temperature at which talc-filled PP composites lose 5% by mass was higher. Looking at the percentage mass that remains un-degraded at the end of 800 °C, it was seen that the most temperature resistant composite was PP with mica filling.

Table 5. TGA results for PP and mineral filled PP composites

Sample	T_{max} (°C)	Temperature of 5% mass loss (°C)	Residual weight at 800 °C, %
PP	451	384	-
PP-Mica	463	431	38
PP-Calcite	457	416	22
PP-Feldspar	462	411	34
PP-Talc	459	433	36

T_{max} : maximum degradation temperature

3.7. TM Analysis

In order to determine the coefficient of thermal expansion (CTE) values thermomechanical analysis was done to the samples of PP and its composites. Table 6 and Figure 8 gives CTE values and TMA curves of PP and its composites, respectively.

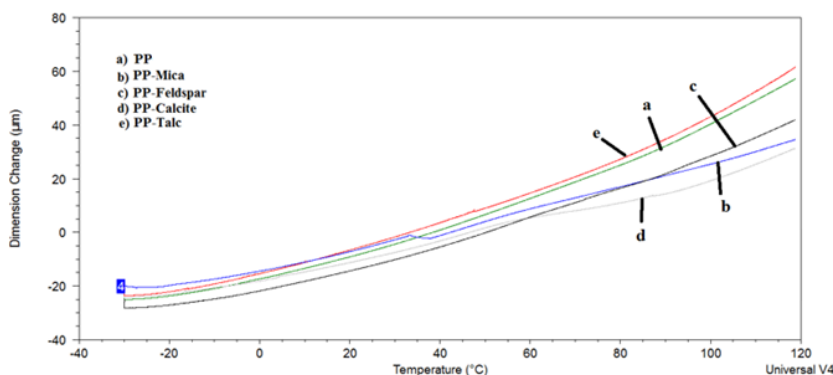


Fig. 8. Dimension change versus temperature curves for PP and mineral filled PP composites

The thermal expansion coefficient is one of the most important physical properties used to describe the changes in the size of materials with temperature. The thermal expansion coefficient is of great importance in determining the processing and application conditions of polymeric composites[37]. When compared to neat PP, it could be said that all mineral loadings, except talc, reduce the coefficient of thermal expansion. This may be because mineral fillers reduce the thermal expansion as a result of their interaction with the polymeric matrix and consequently the movement of the polymer chains is restricted [38] While the largest decrease was in mica loading, the least decrease was in feldspar filled PP composites.

Table 6. CTE values of PP and mineral filled PP composites

Sample	CTE(-10 to 50 °C) ($\mu\text{m}/\text{m}$)°C
PP	118
PP-Mica	87
PP-Calcite	92
PP-Feldspar	103
PP-Talc	120

4. Conclusion

In this study the effect of mineral types on the physical, mechanical, thermal and rheological properties of mineral filled polypropylene composites were investigated. According to this research; the higher density values were obtained with mineral filler loading. The lowest density of $1.15 \text{ g}/\text{cm}^3$ was obtained when feldspar was used as a filler. 40 wt. % mineral loading into PP decreased the tensile strength of PP. Tensile strength values of 31.1, 30.3, 20.9, 29.4 and 21.2 MPa was obtained for PP, PP- Talc, PP- Calcite, PP-Mica, and PP-Feldspar, respectively. It was demonstrated that the decrease in tensile strength in PP-Talc and PP-Mica composites were lower compared to the other mineral fillers. Mineral loadings into PP by 40 wt. % increased the tensile and flexural moduli of PP. Decrease in impact strength of PP was observed with the addition of minerals. The less decrease in impact strength was obtained by loading feldspar rather than talc, calcium carbonate and mica. MFI of PP decreased with addition of all minerals. Mineral addition increased the vicat softening temperature of PP. Vicat softening temperatures of PP, PP-Talc, PP-Calcite, PP-Mica, and PP-Feldspar were obtained to be 61, 82, 67, 78 and 68 °C, respectively. Lower viscosity rates were observed in the composites of PP-Calcite and PP-Feldspar. 40 wt. % mineral filled PP composites have higher degradation temperature than PP. Coefficient of thermal expansion decreased with all mineral loadings except talc in PP composites. Calcite, mica or feldspar loaded PP composites could be an alternative for use in automotive industry compared to the talc loaded PP composites due to their better impact properties.

Conflicts of Interest

No conflict of interest was declared by the authors.

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