

Original Research Paper

Fabrication and Mechanical Properties of Glass Fiber/Talc/CaCO₃ Filled Recycled PP Composites

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Abstract: Until now, recycling studies brought to the agenda after the rapid increasing of plastic materials in every area and causing those plastics to environmental pollution after discarding them following the utilization. The purpose of recycling plastic waste is to minimize environmental pollution and to create of new resources. To perform the present study, Recycled Polypropylene Granules (RPP) belonging to Polypropylene (PP) will be used in most particularly in the automotive and in the packaging industry finding application area behind the Low Density Polyethylene (LDPE). To develop the several properties (physical and thermal) of RPP polymer and to get close to or greater value to the original PP features, different rates of micron-sized glass fibers, talc and CaCO₃ are added into the polymer. By recycling waste, demand for natural resources (such as oil, which is plastic's raw materials) will decrease and rapid consumption of energy sources will be prevented while providing the protection of natural resources. Raw materials imports would be reduced due to the usage of recycled products. In this study, Recycled Polypropylene Granules (RPP) were obtained from companies and glass fiber/talc/CaCO₃ additive RPP granules in different ratios were produced with compounding extrusion process. These produced new composite pellets were produced in injection molding machine by means of designed mold to perform tensile and impact tests. In the thermal analysis, the melting temperature and crystallization rate were determined by DSC analysis; the thermal decomposition temperature was determined by TGA analysis. Micro structural examination was done using a Scanning Electron Microscope (SEM).

Keywords: Recycling, Polypropylene, Fillers, Mechanical Properties

Introduction

Currently, environmental alarming has led to a renovated concern in usual materials and problems like recyclability and ecological care are happening gradually significant for the initiation of novel materials and artifacts (Arbelaiz *et al.*, 2005). Trash is sensed as a main issue, particularly for vast expenditure polymers such as polypropylene (Brachet *et al.*, 2008). Plastic recycling has been effectively applied with main treated materials causing in economic profits (Da Costa *et al.*, 2007).

In the current study, recycled PP was used as a matrix for devising the composites. Recycled PP is obtainable as a trash material and generates many ecological harms.

To decrease the ecological harms, PP can be reprocessed to fabricate new value added artifact with little fabrication price (AlMaadeed *et al.*, 2012). Due to its decent mach inability, excessive recyclability and little price, PP has been noticed a wide range of claims in the textile, packaging, car and furniture productions. To enlarge the variety of claims, growing the strength and modulus of PP has revived significant concern. Discharging PP with hard inert specks is an operative, inexpensive and suitable method to increase the strength and stiffness (Zheng *et al.*, 2009).

Generally, the mechanical properties of recycled mixed polymers are evidently developed by summing fibres have ample greater strength and rigidity than those

of the matrices (Homkhiew *et al.*, 2013). In this study we used glass fibre, talc and talc as reinforcement for making the composites. Several authors have investigated the recycling of polypropylene (Beg and Pickering, 2008). Dintcheva *et al.* (2001) studied the influence of three dissimilar fillers specifically glass fibres, calcium carbonate and wood fibres with the polyolefin reprocessed plastic and reported that glass fibres overtakes others by growing its tensile modulus from 460 to 935 MPa with 20% glass fibre. Nevertheless, the drop of elongation at break was detected. A comparable investigation was directed by Putra *et al.* (2009) on the influence of glass fibre, talc, wollastonite and gypsum on the mechanical properties of recycled-plastic composites. Both fillers and reinforcement displays growth in the toughness of the materials. Their investigation proved that glass fibres demonstrate the finest outcomes by refining its tensile and flexural strength by 30% and the important growth in modulus by 250% with the adding of 30% glass fibre. The addition of glass fibre from 10-30% by weight results in considerable growth in elastic modulus, along with a rise in strength with abridged ductility (Xanthos *et al.*, 1995). According to the Bank's article (Lawrence, 2006), it can be understood that with the adding of 30% glass fibres, the elongation of break can decrease from 25 to 2%.

Another study conducted by Gupta *et al.* (1989) focused on the influence of high temperature on the tensile properties of unreinforced and reinforced PP. They observed brittle behavior at inferior temperatures whereas it displays ductile behavior at upper temperature. The results of the study further designates that there is a fall of in excess of 50% tensile strength for the temperature of 20 to 55°C for together reinforced and unreinforced specimens. Conversely, the decrease in together tensile strength and modulus at raised temperature is under that of unreinforced specimens comparing to the glass fiber reinforcements. The researchers further reported that with the increment of test temperature, the interfacial shear strength decreased for all composite specimens which could ascend either from a flagging of chemical bond or from the reduction of thermal stresses and the decreased of bond strength at the fibre-matrix interface.

The mechanical properties of recycled mixed plastic leftover with the glass fiber displays better development as detected by Xanthos and Narh (1998; AlMaadeed *et al.*, 2012; Hugo *et al.*, 2011; Putra *et al.*, 2009; Dintcheva *et al.*, 2001) have stated that the melt flow index decreases with the increment of the fiber content owing to the great weight and the increment of the viscosity of the composite. A comparable state was described by Hugo *et al.* (2011) who stated that because of high price and treating restrictions for the recycled composite, the extreme quantity of glass fiber which could be combined in the artifact is 30% by

weight. In this study, recycled polypropylene granules (G-PP) were obtained from companies and glass fiber/talc/CaCO₃ additive G-PP granules in different ratios were produced with compounding extrusion process. These produced new composite pellets were produced in injection molding machine by means of designed mold to perform tensile and impact tests.

Materials and Methods

Materials

A Recycled Polypropylene Granules (RPP) obtained from the Polipro Company was used as the polymer matrix material having a density of 0.92 g/cm³. Talc and CaCO₃ additive materials have been obtained from the Omya Company. Commercially available glass fiber with 4.55 µm-1 mm average length and 2.9 g/cm³ average density was used as reinforcement for making the composites.

Composite Preparation

Talc, CaCO₃ and glass fibers filled recycling PP polymer composites are produced using Brabender twin screw extruder. The extruder temperature was used in the range of 190-230 °C for filled RPP. RPP samples were prepared for 30% of talc, CaCO₃ and glass fiber. The polymers mixtures were extruded, cooled and then granulated. Test samples were prepared by Arburg injection molding machine.

Morphological Analysis

Morphological analysis was carried out with the utilization of a Scanning Electron Microscope (Nano SEM 650). The cross-sections of the samples were examined after tensile tests to investigated the fracture and bonding between fillers and matrix.

Mechanical Testing

Tensile Properties

The tensile tests were accomplished consistent with the ASTM D 638-02 standard, by utilizing a Universal Testing Machine at a crosshead velocity of 10 mm/min. The standard dimensions are 20 mm length, 12.5 mm width and 4 mm thickness. The Young's modulus was determined routinely by software, Lloyd-LC Instruments by choosing tangent technique. The tensile test direction was uniaxial and five samples were taken for the tensile test. In each case, five samples were tested and the mean value was declared.

Impact Properties

Notched Charpy impact tests were carried out with a impact tester with pendulum energy of 15 J, according to ASTM D 256. All the reported values for the impact tests were the average values of 5 samples.

Thermal Testing

Differential Scanning Calorimetry (DSC)

The RPP and RPP composites were analyzed by DSC using a thermal analyzer (Setaram-Labsys evo). The measurements were taken by keeping fixed heating ratio of 10°C/min under nitrogen atmosphere. The crystallinity fraction (X_c %) of the RPP and RPP composites was determined by means of the subsequent equation:

$$X_c (\%) = (\Delta H_m / \Delta H_m^0 \times W_{polymer}) \times 100 \quad (2.1)$$

$W_{polymer}$ = The weight ratio of polymer matrix

ΔH_m = The heat of fusion

ΔH_m^0 = The heat of fusion of 100% crystalline PP (207.1 J/g) (Velasco *et al.*, 2002; Wal *et al.*, 1998)

Thermo Gravimetric Analysis (TGA)

Thermo gravimetric analysis (TGA) measurements were performed in nitrogen atmosphere using a Setaram-Labsys evo instrument at a heating rate of 10°C/min.

Melt Flow Index

Melt Flow Index (MFI) is defined as the weight of the polymers in grams extruded in 10 min through capillary of specific dimensions by pressure applied through dead weight via piston. Melt flow index was conducted using a Ceast model machine. ASTM D1238-Method A was applied for determining the MFI of the recycled polymers.

Morphological Study

The morphologies of talc, calcium carbonate and glass fiber reinforced polymer composites were examined by Scanning Electron Microscopy (SEM) on the fracture surface of the tensile specimens. The samples were covered with a thin film gold and analyzed at an accelerating voltage of 1.00 kV.

Results and Discussion

TGA Analysis Results

The Polypropylene (PP) polymer and talc, calcium carbonate and glass fiber reinforced polymer composites samples are given in Fig. 1 for thermal decomposition curves (thermo gram). While the temperature at which the degradation of the PP polymer of approximately 380°C, talc, CaCO₃ and glass fiber reinforced PP polymer composites begin to

decompose at approximately 400°C. The decomposition temperature depends on the type of additives increased on average by 5.2%. Similar results for glass fiber and wood flour were obtained from recycled polypropylene reinforced hybrid composites (AlMaadeed *et al.*, 2012). In this case, the additives were added to PP polymer improve to the thermal properties of polymer and demonstrating that provides possibility for use at higher temperatures. Analysis of results losses of mass in the PP polymer composites samples were found around 70%.

DSC Analysis Results

In Table 1, the DSC analysis results are given for the samples of RPP polymer, RPP+30% talc, RPP+30% CaCO₃ and RPP+30% GF polymer composites. As seen from the table, the melting temperature (T_m) and enthalpy (ΔH_m) and crystallization rate (% X_c) of RPP polymer and doped RPP composites was obtained. There is no difference in T_m of RPP polymer composites. As seen from the table that crystallization degree of talc, CaCO₃ and glass fiber reinforced RPP polymer composites was obtained lower than that of undoped RPP polymer. While crystallization degree of RPP polymer was obtained as 36.87%, its crystallization degree was attained as 36.35, 30.83 and 29.74% with the addition of talc, CaCO₃ and glass fiber, respectively. The fillers prevent the movement of the PP macromolecular chain and the macromolecular section from getting ordered position of the crystals. The fillers hinder development of crystallinity. Consequently, the X_c of polymer composites are reduced (AlMaadeed *et al.*, 2012).

Mechanical Tests

The Recycled Polypropylene (RPP) polymer, 30% talc filled RPP, 30% calcium carbonate filled RPP and 30% glass fiber Reinforced Polymer (RPP) composite samples were given in Fig. 2 for tensile strength results. While tensile strength of RPP polymer was obtained as 30 MPa, its tensile strength decreased with the addition of talc and CaCO₃ and attained as 23 and 20.6 MPa, respectively. The tensile strength of the RPP polymer decreased to 23% and 31% for talc and CaCO₃ respectively. This behavior could be generally caused by the reduced interaction at the interface level between the fibers and the polymer matrix (Mutje *et al.*, 2006). The tensile strength was obtained as 68MPa with the increment of 126% by the addition of 30% glass fiber into RPP. Similar results were also obtained by AlMaadeed *et al.* (2012; Beg and Pickering, 2008; Zheng *et al.*, 2009; Serrano *et al.*, 2014).

Table 1. Melting temperature and % crystallization rates obtained by DSC analysis results

Sample	Melting temperature (°C)		Enthalpy of melting, (J/g) ΔH_m	% Crystallinity X_c
	T_m			
RPP	165.74		76.363	36.87
RPP-30%Talc	166.25		52.697	36.35
RPP-30%CaCO ₃	169.19		44.699	30.83
RPP-30%Glass fiber	167.46		43.128	29.74

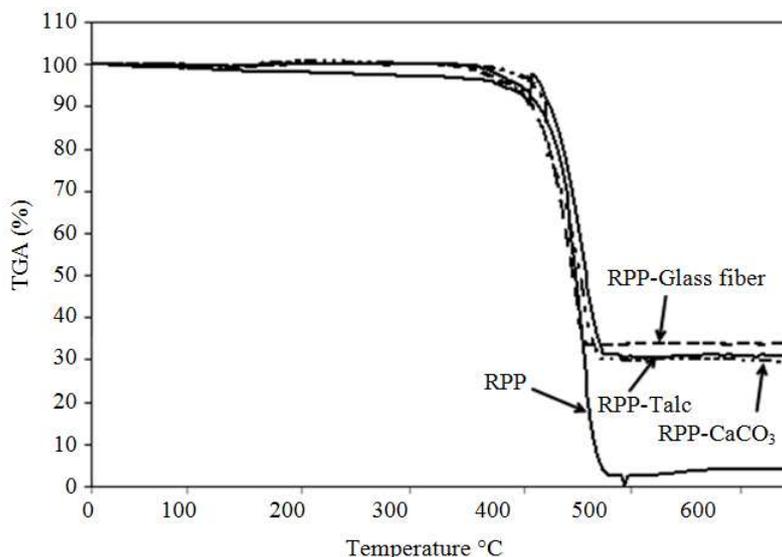


Fig. 1. TGA analysis results of RPP polymers and composites

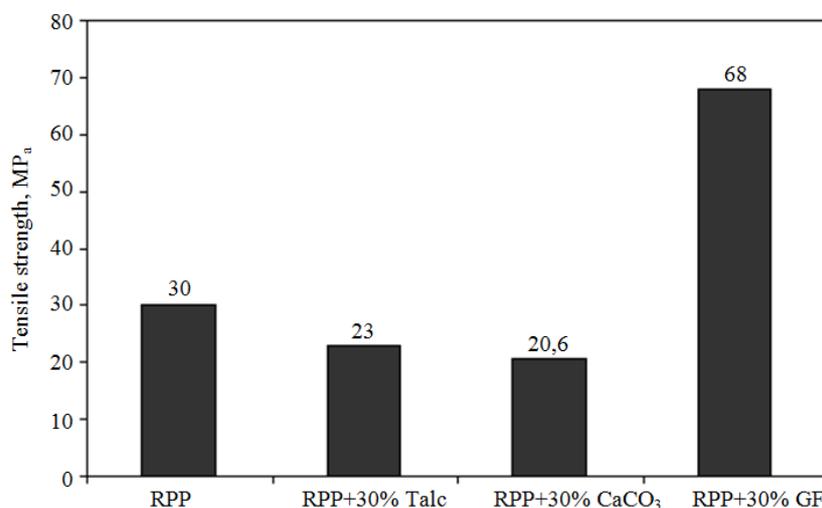


Fig. 2. Tensile strength of various polymeric samples of RPP, RPP+%30 talc, RPP+%30 CaCO₃ and RPP+%30 GF

In Fig. 3, the yield strength results were given for RPP polymer, RPP+30% talc, RPP + 30% CaCO₃ and RPP+30% GF polymer composite samples. Similar to the results of tensile strength, the yield strength of RPP polymer decreased to 15.3 and 26.9% with the addition of talc and CaCO₃, respectively. This is because; talc and CaCO₃ additives are separated easily from the matrix

due to the creation of a weak customization interface. The yield strength of GF reinforced composites is higher because it establishes a bond with the PP matrix at the same contribution rate. The yield strength of RPP+30% GF polymer composite increased to 142% compared to the RPP polymer. Similar results were also obtained by Li *et al.* (2012) in the earlier work.

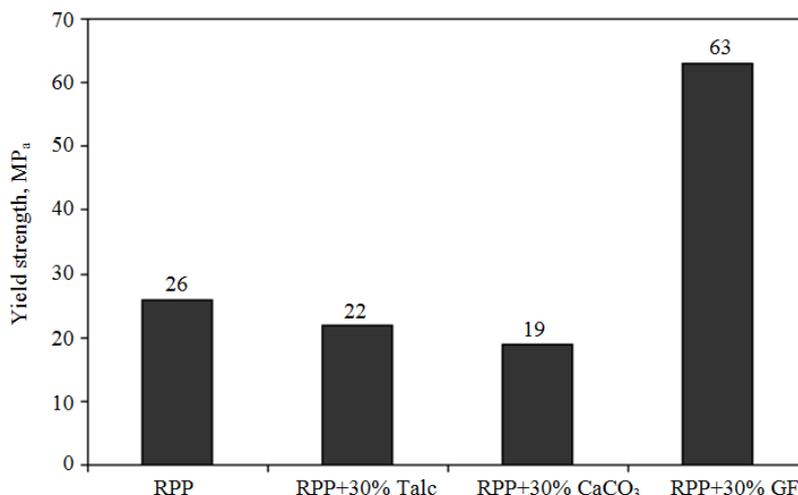


Fig. 3. The yield strength for the polymeric samples of RPP, RPP+%30 talc, RPP+%30 CaCO₃ and RPP+%30 GF

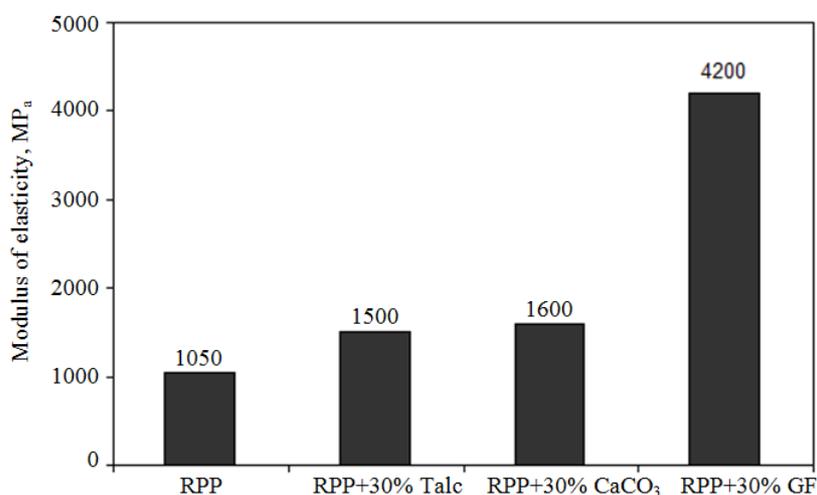


Fig. 4. The elastic modulus for the polymeric samples of RPP, RPP+%30 talc, RPP+%30 CaCO₃ and RPP+%30 GF

In Fig. 4, the results for modulus of elasticity are given for the samples of GP polymer, RPP+30% talc, RPP+30% CaCO₃ and RPP+30 GF polymer composites. The modulus of elasticity increased with talc, CaCO₃ and glass fiber into the RPP polymers. The 1 GPa elastic modulus of RPP polymer increased in 42 and 52% and reaching to the values of 1.5GPa and 1.6 GPa with the addition of talc and CaCO₃, respectively. By the accumulation of GF additives, the elastic modulus of RPP polymer increased to 300% and reaching to 4.2 GPa. The present results are consistent with the previous studies reported by (AlMaadeed *et al.*, 2012; Beg and Pickering, 2008; Zheng *et al.*, 2009 Serrano *et al.*, 2014; Li *et al.*, 2012).

In Fig. 5, the Izod impact strength results are given for the samples of RPP polymer, RPP+30% talc, RPP+30% CaCO₃ and RPP+30% GF polymer

composites. The impact strength of GPP polymer decreased with the addition of talc and CaCO₃; however the impact strength of GF increased. The impact strength (had 4.1 kJ/m²) for RPP polymer was obtained as 3.2 and 3.5 kJ/m², respectively in case of using talc and CaCO₃ additives, but these values were obtained as 8.8 kJ/m² with the addition of GF additives. Similar results were attained by the previous worker Beg and Pickering (2008).

Melt Flow Index (MFI) results are shown in Fig. 6. The MFI outcomes decreased with talc, CaCO₃ and glass fiber materials. The viscosity of RPP composites increased as a result of the high content of the additives. Same results were observed by several researchers Beg and Pickering (2008; Lu *et al.*, 2006). Tasdemir *et al.* (2009) studied the LDPE and PP-wood fiber composites and stated that MFI abridged with the growth of wood flour substance. Son *et al.* (2004) testified that MFI condensed with the growing of paper sludge substance.

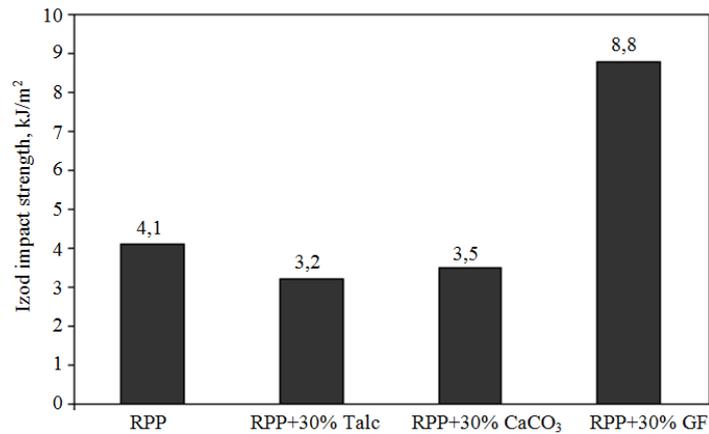


Fig. 5. The impact strength for the polymeric specimens of RPP, RPP+%30 talc, RPP+%30 CaCO₃ and RPP+%30 GF

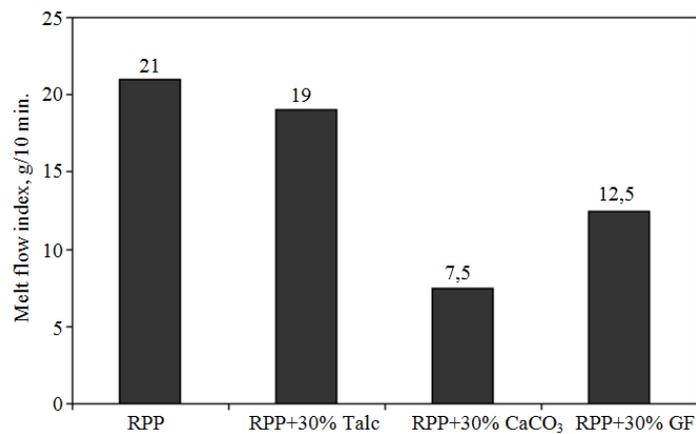


Fig. 6. The melt flow index values for the polymeric samples of RPP, RPP+%30 talc, RPP+%30 CaCO₃ and RPP+%30 GF

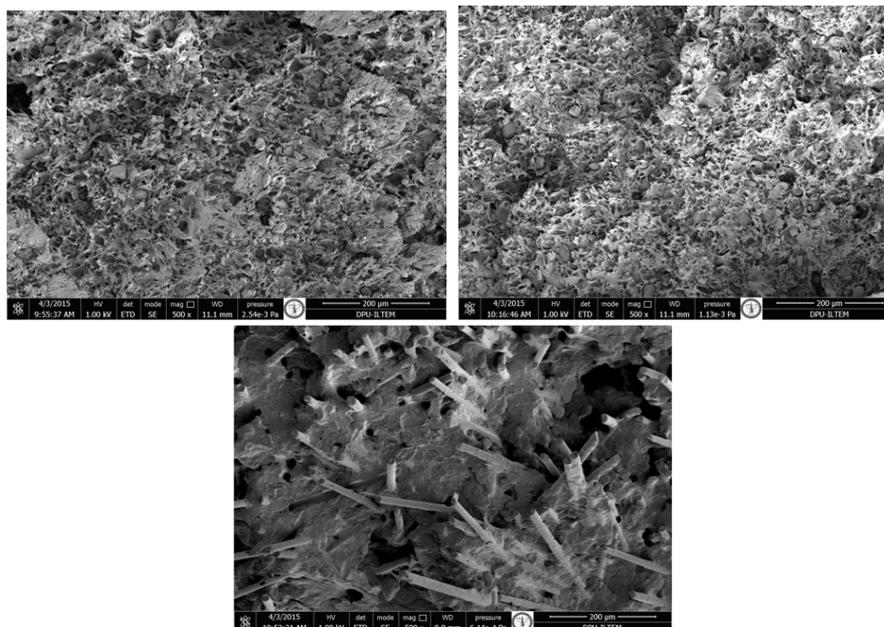


Fig. 7. SEM micrographs for the samples of (a) RPP+30% talc, (b) RPP+30% CaCO₃ and (c) RPP+30% GF

Microstructural Characterization

In Fig. 7, the SEM micrographs for RPP+30% talc, RPP+30% CaCO₃ and RPP+30% GF were obtained from the fracture surfaces of the tensile specimens. As a result of the quite weak bonding between RPP and additives, RPP recycled polymers were pulled out from the talc and CaCO₃ additives due to the high amount of talc and CaCO₃ additives. However, the mechanical properties were developed for RPP+30% GF polymers due to the both high amount of glass fibers and the better interface bonding between glass fiber and RPP matrix. It is seen from the SEM micrographs that the less glass fibers are pulled out and are broken. This situation shows strong bond interface evidence between glass fiber and RPP matrix.

Conclusion

In the present research, the following outcomes were obtained:

- The tensile strength of the RPP polymer reduced by 23 and 31% for talc and CaCO₃, respectively. Also, the tensile strength was obtained 68 MPa with the increment of 126% by the addition of 30% glass fiber into RPP
- The yield strength of RPP polymer decreased to 15.3 and 26.9% with the addition of talc and CaCO₃, respectively. In contrast, the yield strength of GF reinforced composites was higher because it established a bond with the PP matrix at the same contribution rate. The yield strength of RPP + 30% GF polymeric composite increased to 142% compared to the RPP polymer
- The modulus of elasticity was increased with talc, CaCO₃ and glass fiber into the RPP polymers. The 1GPa elastic modulus of RPP polymer increased in 42 and 52 and reaching to the values of 1.5 and 1.6 GPa with the addition of talc and CaCO₃ respectively. By the accumulation of GF additives, the elastic modulus of RPP polymer increased in 300% and reaching to 4.2 GPa
- The impact strength of RPP polymer decreased with the addition of talc and CaCO₃; however the impact strength of GF increased. The impact strength (had 4.1kJ/m²) for RPP polymer was obtained as 3.2 and 3.5 kJ/m², respectively in case of using talc and CaCO₃ additives, but these values were obtained as 8.8 kJ/m² with the addition of GF additives
- The MFI outcomes decreased with talc, CaCO₃ and glass fiber materials. Viscosity of RPP composites increased as a result of the high content of the additives
- As a result of the quite weak bonding between RPP and additives, RPP recycled polymers were pulled out from the talc and CaCO₃ additives due to the high amount of talc and CaCO₃ additives.

However, the mechanical properties were developed for RPP+30% GF polymers due to the both high amount of glass fibers and the better interface bonding between glass fiber and RPP matrix. Strong bond interface evidence was observed between glass fiber and RPP matrix

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Author's Contributions

Uğur Soy: Literature search, checked tables and figures and interpreted the data.

Fehim Fındık: SEM analysis, checked the gramatical errors, writing and revising of the manuscript.

Salih Hakan Yetgin: Mechanical and thermal tests, writing and revising of the manuscript, corresponding author.

Tolga Gökkurt: Supply material, Manufacturing of composites with extrusion and injection.

Ferhat Yıldırım: Manufacturing of composites with extrusion and injection, DSC and TGA analysis.

Ethics

The present work is not published in its present form in any journal or will not be published in any journal.

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