



Determination of Mechanical Performance and Microstructural Properties of Ethylene-1-Octene Copolymer (EOC) Elastomer and Glass Beads (GB) Filled Polypropylene (PP) Composites

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Abstract

This study was carried out to determine the mechanical performance of the parts (bumper, panel, console, and accessories) where stiffness and toughness are required in the automotive industry and to examine their microstructure properties. Mechanical properties of the blend and composite materials were determined by adding ethylene-1-octene (EOC) elastomer to provide toughness, micron-sized glass beads (GB) for rigidity and a mixture of both in certain proportions to polypropylene (PP) polymer. 12wt.% EOC elastomer added PP blend, 8wt.% glass bead filled PP composite, and 12wt.% EOC elastomer and 8wt.% glass bead hybrid filled PP composites were first produced in granule form in a twin-screw compounding machine. Then, a conventional injection molding machine was used to produce test samples conforming to the standards. Tensile and impact tests were carried out for mechanical tests. In order to give an idea about the fluidity, the melt flow index test was carried out.

A scanning electron microscope (SEM) was used for microstructure investigations of the polymer blend and composites. At the same time, EDS analysis was performed for characterization. As a result of the experiments, the glass bead increased the stiffness of the composite, while EOC elastomer increased the toughness of the blend and composite. It has been determined that the interfacial bonding between the glass beads and the polymer main matrix is not good, therefore the mechanical properties are reduced.

Keywords: Polypropylene, elastomer, glass beads, mechanical properties, microstructure

1. Introduction

These days, efforts to reduce weight in vehicles and to produce better vehicles in terms of both economy and efficiency continue without slowing down. Especially globally, petroleum-derived fuels become more expensive without slowing down, forcing manufacturers to produce more economical vehicles [1]. The trend of using polymer materials for use in light vehicles is increasing [2, 3]. In the automotive sector, it has become important to examine the mechanical properties of the parts (bumper, panel,

console and accessories) where stiffness and toughness are required and to determine the microstructure changes.

The addition of Ethylene-1-Octene Copolymer (EOC) to blends of linear low density [4] or high density polyethylene [5] improves the mechanical properties. Also, the addition of Ethylene-1-Octene Copolymer (EOC) to blends of polyethylene, the modulus of elasticity in tensile also decreases [6] When ethylene-1-octene elastomer is added to the

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polypropylene main matrix, the toughness of the polymer blend increases [7, 8].

Adding a low rate of glass beads to the pure polypropylene homopolymer polymer causes a decrease in the tensile strength of the produced composite [7, 9–11]. The addition of glass beads to PA6 polymer increases the elastic modulus of the composite [12]. In composites with linear low density polyethylene (LDPE) matrix and glass bead panels, consoles, and accessories and to examine the microstructure properties of polymer parts used in the vehicle industry, especially in the automotive industry, where stiffness and toughness are required. Mechanical properties of the mixture and composite materials were determined by adding ethylene-1-octene (EOC) elastomer to provide toughness, micron-sized glass bead (GB) for rigidity and a mixture of both in certain proportions to polypropylene (PP) polymer. PP composite with EOC elastomer additives, PP composite and EOC elastomer/glass bead additives in certain amounts were produced in a conventional injection molding machine. Tensile and

additive, an increase in the ratio of glass beads and an increase in tensile strength is observed [13]. When glass bead is added to the pure PP polymer main matrix, the stiffness of the composite and the modulus of elasticity in tensile also increase [14]. This situation is explained by the porcelain theory [15].

In this study, it was carried out to determine the mechanical performance of parts such as bumpers, impact tests were carried out for mechanical tests. Information about the fluidity was obtained by the melt flow index test. A scanning electron microscope (SEM) was used for microstructure investigations of mixtures and composites. At the same time, EDS analysis was performed for characterization. As a result of the examinations, the glass bead increased the rigidity of the composite, while EOC elastomer increased the toughness of the mixture and composite. It has been determined that the interfacial bond between the glass bead and the polymer main matrix is not good, therefore the mechanical properties are reduced.

2. Experimental Studies

2.1. Material and Method

In this study, polypropylene homopolymer (PP-H) polymer was used as the main matrix material. The PP-H used in the experiments has a homopolymer structure and was obtained in granule form. PP-H is suitable for injection molding and has a density of 0.90g/cm^3 . Polypropylene homopolymer is naturally colored and was purchased from Borealis-Austria company with the trade code BUPLEN 6531. Ethylene-1-octene copolymer polyolefin elastomer with an estimated molecular weight of over 10,000 g/mol, and a density of 0.88g/cm^3 is compatible with

polypropylene polymer. The elastomer used to provide high impact resistance was obtained from Dow Chemical as Engage brand and 8000 series. It was developed to increase the impact strength of commonly used polymers such as both polypropylene and polyethylene. This elastomer is used in bumper, panel, console, and accessory materials in the automotive industry. Technical properties such as physical, mechanical and thermal properties of ethylene-1-octene copolymer polyolefin elastomer are given in Table 1.

Table 1. Physical, mechanical, and thermal properties of ethylene-1-octene copolymer elastomer

Material properties	Ethylene-1-octene copolymer elastomer		
	Amount	Unit	Test standard
Density	0.88	g/cm^3	ASTM D792
Melt flow index	18	g/10min	ASTM D1238
Tensile strength	10.6	MPa	ASTM D638
Modulus of elasticity in tensile	3.30	MPa	ASTM D638
% Elongation at break	>1000	%	ASTM D638
Modulus of elasti-	19.5	MPa	ASTM D790

city in bending			
Tear strength	47.5	kN/m	ASTM D624
Stiffness	76	Shore A	ASTM D785
Melting temperature	76	°C	Dow method
Glassy transition temperature	-50	°C	Dow method
Vicat softening temperature	45	°C	ASTM D1525

ASTM: American Society for Testing and Materials

The glass bead was purchased from Potters Industries with the code CP-3 with an additive surface increase the rigidity of the composite material planned to be produced and to obtain a beautiful appearance at the same time. In order to increase the thermal resistance during the compound production, the heat stabilizer at certain rates and lubricants and/or additives that facilitate processing are added

modification having an average particle size of 10 μ m. Glass bead additive material is used to prevent polymer adhesion to the screw and sleeve. The compositions of PP- based hybrid composites with ethylene-1-octene copolymer elastomer and glass bead added used in the experiments are given in Table 2.

Table 2. Compositions of ethylene-1-octene copolymer elastomer and glass bead added PP homopolymer hybrid composites

Composite	EOC additive % ratio (by weight)	Glass bead additive % ratio (by weight)	PP homo-polymer % ratio (by weight)
PP-H	-	-	100
PP-H/12EOCR	12	-	92
PP-H/8CB	-	8	92
PP-H/12EOCR/8CB	12	8	84

In addition, the extrusion and injection molding process conditions of the test samples are given in Table 3.

Table 3. Process conditions for the production of test specimens

Process	Extrusion	Injection
Temperature, °C	170-235	180-230
Pressure, bar	~ 40	80
Screw speed, rev/minute	30	30
Mold temperature, °C	-	~ 40
Mold waiting time, sec	-	30

Since the polypropylene homopolymer matrix material and ethylene-1-octene copolymer elastomer in granule form used in the experiments were in the form of granules, they were first mixed mechanically and then fed to a twin-screw compounding machine from the same feeding unit. Glass bead additive was added to the machine from a second feeding zone. The compounding machine is cylindrical and double screwed in order to provide a homogeneous mixture.

While producing a compound by extrusion, extruder heater temperatures were set between 175-230 °C. The long spaghetti-shaped bars from the die head were immediately directed to the cooling pool when exiting the mold and cooled in the pool. At the exit of the cooling pool, the air was blown with the help of air-blowing fans and the moisture on the polymer in the form of spaghetti pasta was removed. Then, cutting was made with the granulator in the system,

PP polymer mixture in accordance with the recipe given in Table 2, and PP-based composite semi-finished granules were produced. In Table 3, the process conditions of the compound extruder used in the production of granules are given. A conventional

type injection molding machine was used for the shaping of the tensile and impact test specimens, and some specific injection process conditions are given in Table 3.

2.2. Experimental setup and process

A mold prepared in accordance with ISO standards was used for the tensile and impact test samples used in this experimental study. The samples were produced by an injection molding technique. Melting flow index measurement tests were carried out in accordance with the ISO 1133 standard, on the Zwick Brand MFI test device. In the experiment, the Tensile test samples were produced in accordance with ISO 527 standard. Tensile tests were carried out on a Zwick brand Z020 tensile device with a capacity of 2 tons. The experiments were carried out in a conditioned room at 50% humidity and 23 °C room temperature at a tensile velocity of 10 mm/min. Izod impact tests with 45° angle V notch of the samples prepared in the dimensions of 80x10x4 mm³ prepared according to the ISO 180 standard were carried out in a Zwick brand impact tester. In the experiments, mechanical properties such as tensile strength, % elongation at break, modulus of elasticity at tensile and impact strength were determined. The

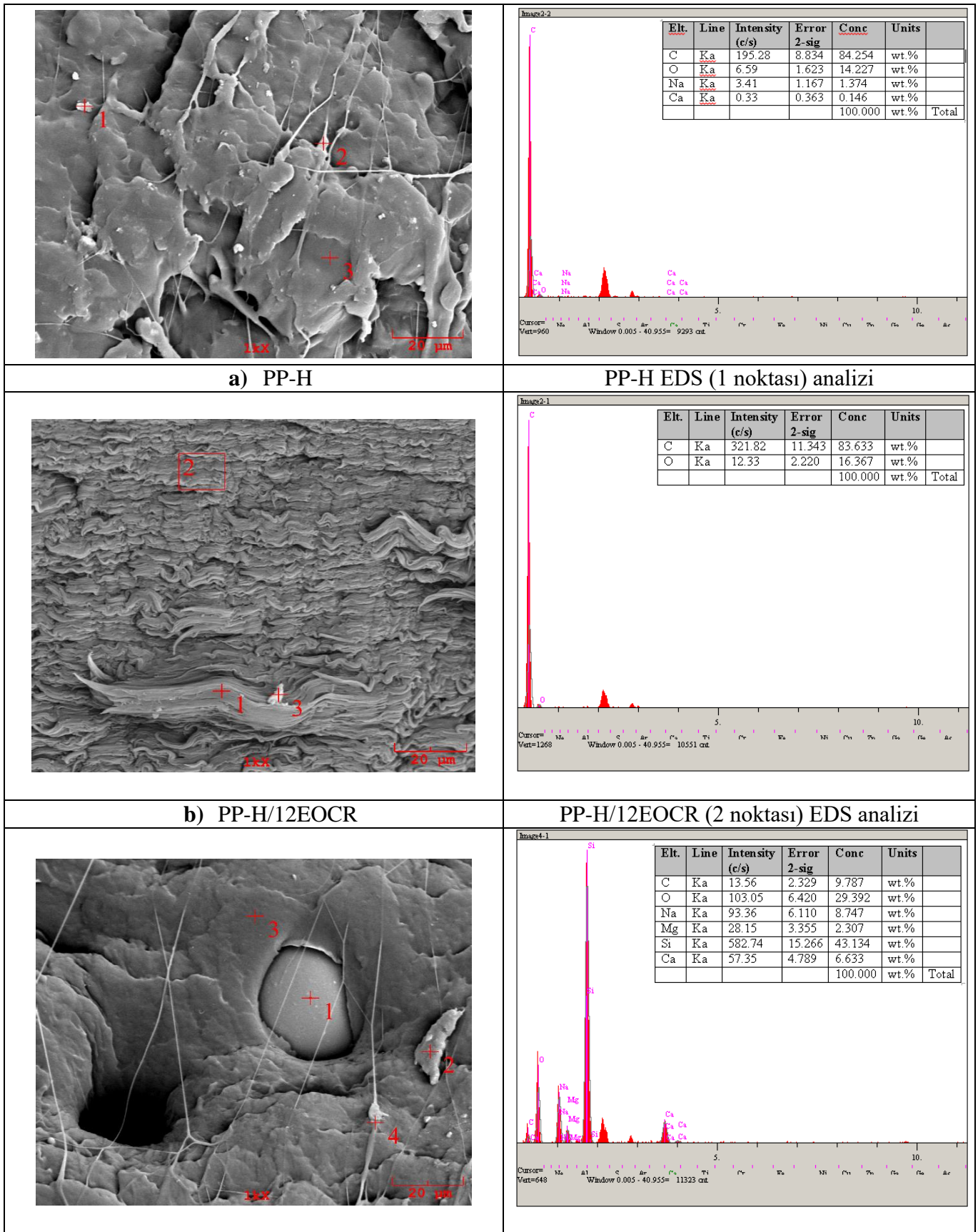
amount of polymer material flowing in one minute was taken and weighed, and multiplied by 10 to determine the melt flow index values of the materials used in the experiments in g/10 minutes. This experiment was carried out to get an idea about the fluidity of the materials used in the experiments.

mechanical values obtained in the study are given as Relative. The relative values are actually an indicator of the comparison of the values of the other tested materials with respect to the PP-H polymer. That is, the relative melt flow index, relative tensile strength, relative modulus of elasticity, relative elongation at break and relative impact strength values are given by dividing the values of pure PP-H polymer. All experiments were repeated at least three times and the average of the arithmetic values was used. A JEOL JSM-6060LV brand electron microscope was used to examine the fracture surface microstructure of the tensile test samples.

3. Results and Discussion

The materials used in the experiments (pure polypropylene homopolymer, 12% ethylene-1-octene copolymer elastomer added PP composite, 8% glass bead added PP composite, 8% glass bead and 12% ethylene-1-octene elastomer added PP composite))

fracture surface microstructure images obtained from the tensile test were taken using scanning electron microscopy (SEM). SEM images and EDS analyzes of the samples used in the experiments are given in Figure 1 a-d.



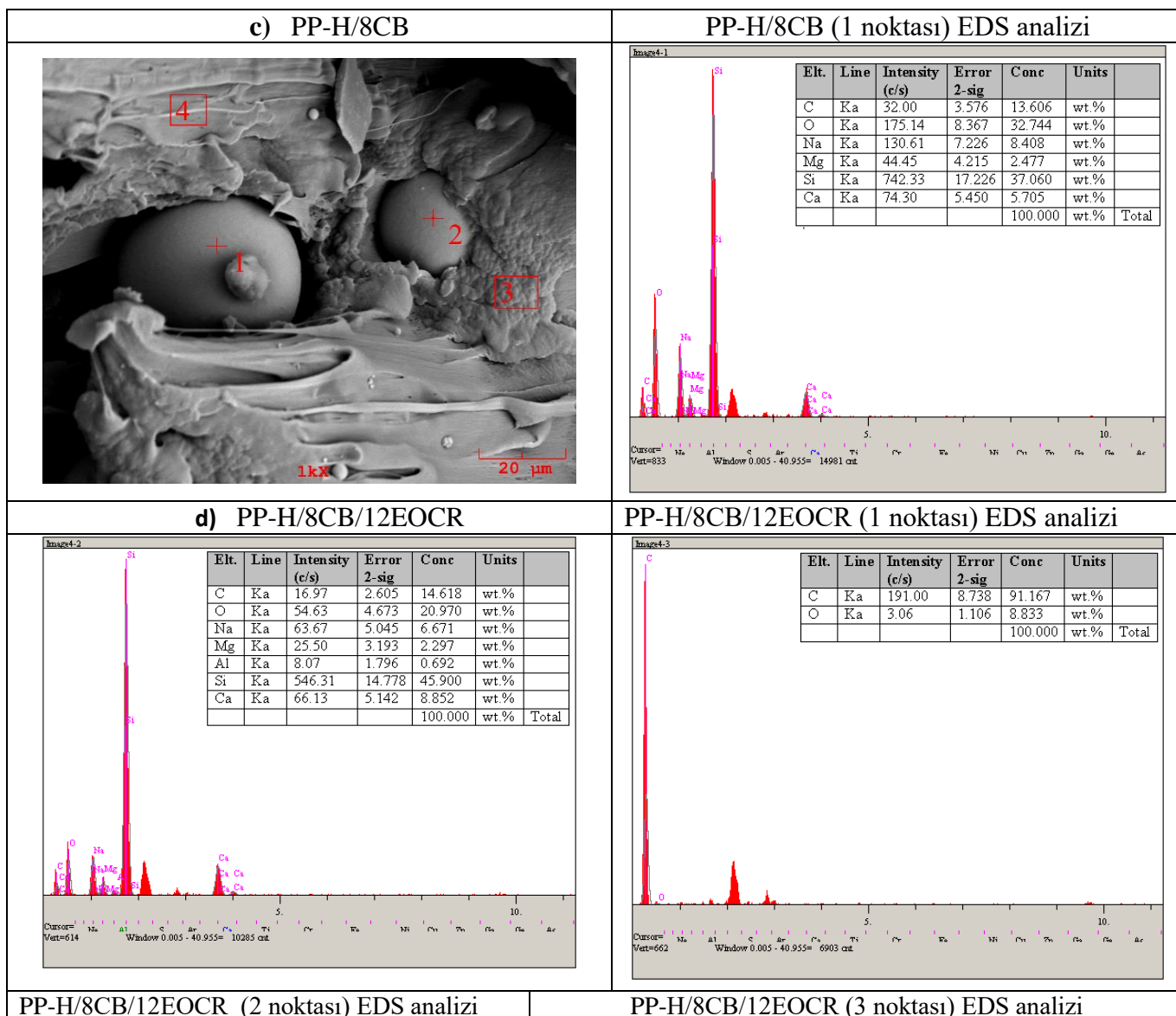


Figure 1. Fracture surface scanning electron microscope images and EDS analyses of a) PP-H, b) 12% EOCR elastomer added PP-H mixture, c) 8% glass bead added PP-H, d) 8% glass bead and 12% EOCR elastomer added PP-H obtained after tensile test

A scanning electron microscope image of the pure PP-H homopolymer is given in Figure 1a, and the EDS analysis taken over the SEM image is given on the right side of the image. EDS analysis was made from the 2 points that look different on the surface and it was determined that the carbon (C) peak was effective. In other words, it is understood from this result that the main matrix is pure PP-H polymer. In Figure 1b, a scanning electron microscope image of the 12% EOCR-doped PP-H mixture is given, and the result of the EDS analysis is given on the right side of the figure. Since the EOCR elastomer added to the polypropylene polymer is also organic, it was

determined that it was C effective in the EDS analysis. In Figure 1c, the SEM image of the glass bead added PP-H polymer composite is given, and the EDS analysis is given on the right side of the figure. In the EDS analysis taken from the point shown with number 1 on the SEM image, it was observed that the Si element was effective. It is known that this comes from the silicon in the structure of the glass bead additive. EDS analysis was performed from a different-looking point (point 1) on the surface and it was observed that there was a C peak. That is, it is understood that the matrix is pure PP-H. SEM image of PP-H/12EOCR/8CB

polymer composite is given in Figure 1d, and the result of EDS analysis is given on the right side of the figure. In the EDS analysis taken from points 2 and 5 on the image, it can be said that Silicon is active at point 2, that is, the presence of glass bead, and C is active at point 5, which is due to the presence of polymer matrix or EOCR elastomer.

The materials used in the experiments (pure polypropylene homopolymer, 12% ethylene-1-octene copolymer elastomer added PP polymer mixture, 8% glass bead added PP composite, 8% glass bead and 12% ethylene-1-octene elastomer added PP The

variation of the relative melt flow index values according to the type of material (composite) is given in Figure 2. The melt flow index of the PP-H/12EOCR polymer mixture increased by about 4% compared to the melt flow index value of the pure PP-H polymer. The reason for this increase is due to 12% EOCR rubber.

When compared to the melt flow index values of PP-H/8CB composite and PP-H/8CB/12EOCR polymer composite, a decrease of approximately 37% and 15.4% was observed in the melt flow index values.

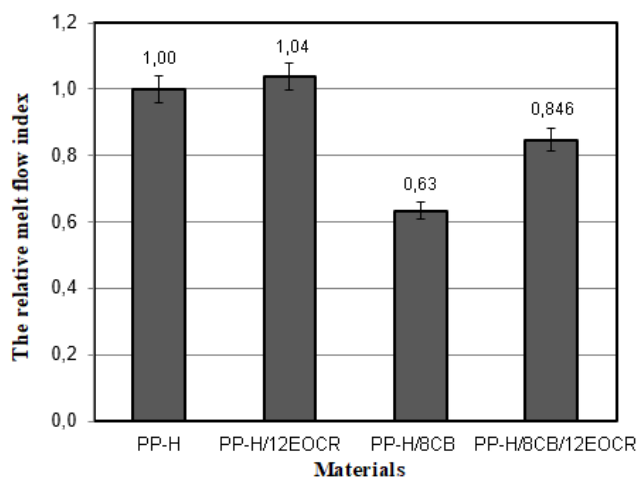


Figure 2. Melt flow index-material relationship of polymers and composites used in the experiments.

When an 8% glass bead additive was added to the pure PP polymer, a decrease was observed in the melt flow index of the composite. In other words, the decrease of 37% in MFI value is due to the glass bead additive in the composite. In other words, the glass bead added to the polymer matrix structure at the rate of 8% reduces the fluidity of the composite. Melting flow index values of PP-H composites with 8% glass beads and 12% EOCR elastomer added were decreased by approximately 15.4% compared to pure PP-H. The possible reason for the decrease in MFI value with the addition of EOCR elastomer can be explained by the contribution of EOCR rubber phase and glass bead in the composite. Since the melt flow index value of the rubber phase is higher than

the MFI value of the pure PP polymer, the MFI value is higher than the glass bead added PP composite.

The relative tensile strength changes of pure PP-H polymer and PP-H composite with 8% glass bead added, PP-H mixture with 12% EOCR elastomer added and 8% glass bead/12% EOCR elastomer added PP composites used in the experiments are given in Figure 3. As seen in the figure, when the tensile strengths of the PP-H/12EOCR polymer mixture, PP-H/8CB composite and PP-H/8CB/12EOCR polymer composite are compared according to the tensile strength of the pure PP-H polymer, the tensile strengths are approximately 13%, 16% and 12.8% respectively decrease was observed.

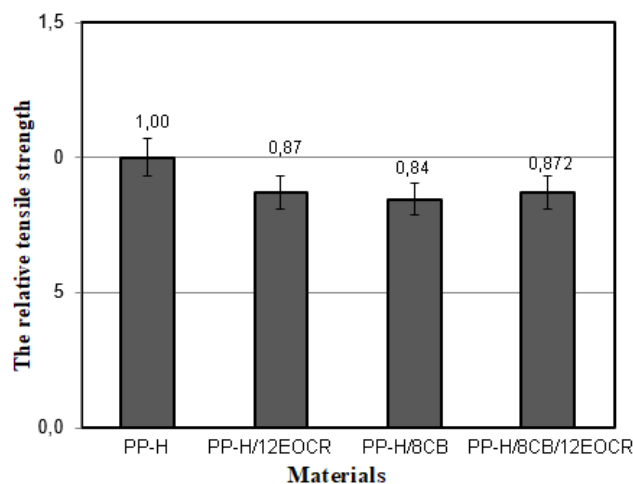


Figure 3. Relative tensile strength-material relationship of polymers and composites used in the experiments

When EOCR elastomer was added to the pure PP polymer at a rate of 12%, a 13% decrease was observed in the tensile strength compared to the pure PP-H. The possible reason for the decrease in tensile strength with the addition of EOCR elastomer can be explained by the soft rubber phase. Similar results were also found in some previous studies in the literature [6–9]. The possible reason for the decrease in tensile strength can be explained by the poor interfacial bonding of the PP-H polymer matrix and the glass bead additive, according to the microstructure investigations in Figure 1c. However, in some previous studies in the literature, it has been found that there is an increase in the tensile strength of polymer-based composites with glass bead reinforcement. An increase of approximately 12% was observed in the tensile strength of Unal et al. [12] in the 10% glass bead added polyamide-based polymer composite. In another literature study, an increase of approximately 33% in tensile strength was observed with an increase in the ratio of glass beads (0-10% by weight) in linear low-density polyethylene (LDPE) matrix and glass bead added composites by Wei et al. [13]. They attributed this to

explained by the soft rubber phase. Similarly, the addition of an 8% glass bead to the pure PP-H polymer caused a decrease of approximately 16% in the tensile strength of the produced composite.

the good interfacial bonding of the polymer matrix with the glass bead additive. The tensile strength of the composite with 12% ethylene-1-octene elastomer and 8% glass bead added to PP homopolymer was reduced by 12.8% compared to pure PP-H.

In Figure 4, the relative modulus of elasticity of pure PP-H homopolymer and 8% glass bead added PP composite, 12% EOCR elastomer added PP-H mixture and 8% glass bead/ 12% EOCR elastomer added PP composites is given. Relative modulus of elasticity of pure PP-H polymer, PP-8CB composite, PP-H/12EOCR elastomer mixture, and PP-H/8CB/12EOCR polymer composite were obtained as 1.0, 1.08, 0.907, and 1.023, respectively. When an 8% glass bead was added to the pure PP polymer main matrix, the stiffness of the composite also increased and the modulus of elasticity in tensile increased by approximately 8%.

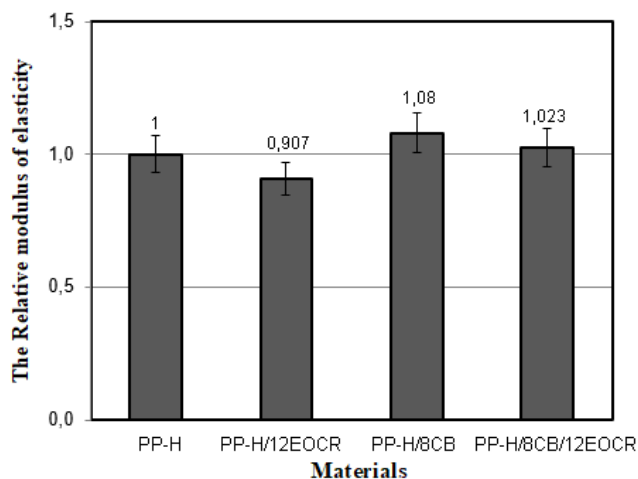


Figure 4. Relative elastic modulus-material relationship of polymers and composites used in the experiments

Similar results were obtained in some studies in the literature [12–14]. The increase in elastic modulus is explained by the Porcelation theory previously described by He and Jiang [15]. When 12% softer ethylene-1-octene elastomer was added to the pure PP homopolymer main matrix, the relative modulus of elasticity of the PP-H/12EOCR polymer mixture was determined as 0.907. The reduction rate in the modulus of elasticity was approximately 9.3%. Similar results were also seen in some previous

studies [7, 9, 11]. The relative modulus of elasticity of PP-H composite with 8% glass beads and 12% EOCR elastomer added was 1.023 compared to pure PP-H.

The change in the relative elongation at break values of pure polypropylene homopolymer, 8% glass bead added PP-H composite, 12% EOCR elastomer added PP-H polymer mixture, and 8% glass bead and 12% EOCR elastomer added PP-H composites given in Figure 5.

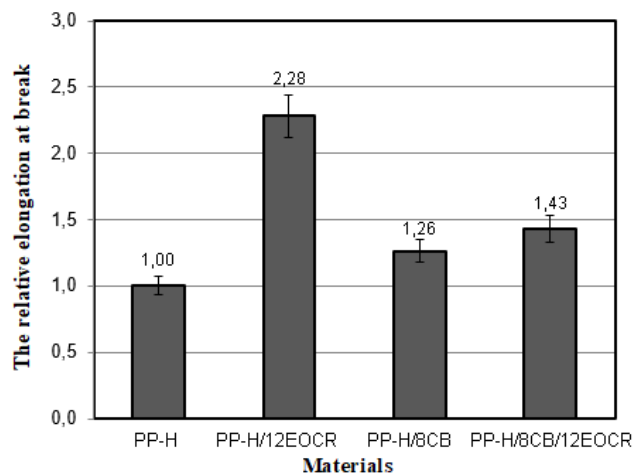


Figure 5. Relative elastic modulus-material relationship of polymers and composites used in the experiments

As seen in Figure 5, it was determined that the % elongation at break of PP-H/12EOCR polymer mixture increased by 128% compared to the % elongation at break of the pure PP-H polymer. When an 8% glass bead was added to the PP-H polymer, the relative elongation value was approximately 0.68. In other words, when the % elongation value of the

glass bead composite is compared with the pure PP value, a decrease of approximately 32% was observed. Similar results in a study by Wang et al. [11], when 10% polyolefin elastomer was added to the PP polymer, the % elongation at the break of the polymer mixture increased by 74%. The increase in % elongation at break obtained in the experiments

was also observed in a previous study on linear low-density polyethylene and glass bead composite [13] and it was stated that there was not much change in elongation. The relative elongation at break of the PP-H/8CB/12EOCR polymer composite was determined as 0.903. Compared to the elongation value of the pure PP-H polymer, it decreased by about 9.7%. However, when compared to the value

of 8% glass bead added PP composite, an increase of 32.8% was detected.

Change of Izod impact strength values of pure polypropylene polymer and 8% glass bead added PP composite, 12% ethylene-1-octene elastomer added PP-H polymer mixture, 8% glass bead and 12% EOCR elastomer added PP-H based composites is given in Figure 6.

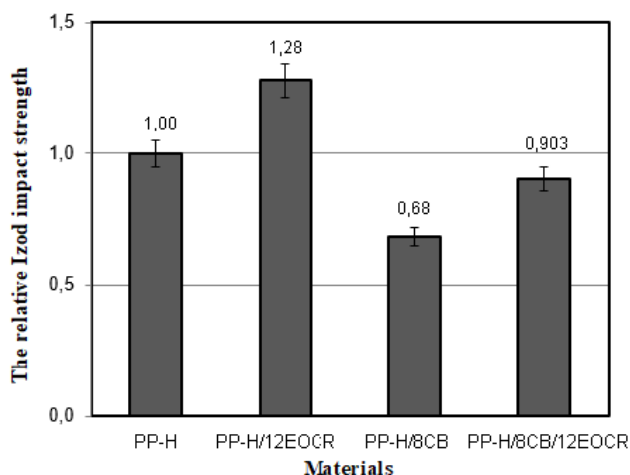


Figure 6. Relative Izod impact strength-material relationship of polymers and composites used in the experiments

As seen in Figure 6, when ethylene-1-octene elastomer was added to the polypropylene main matrix at a rate of 12%, the toughness of the polymer mixture increased by 28% compared to the Izod impact strength of the pure PP homopolymer. Similar results were also found in previous studies in the investigated the mechanical properties of polyamide 6/10% glass bead composite. In their study, they determined that there was a decrease in impact strength. They stated that the reduction in the impact strength of the composite was approximately 21%. They explained that the reason for the decrease in the impact strength is that the rigid glass bead makes the movement of the polymer chains difficult. However, in other studies [13, 14, 16] in the literature, it has been stated that the impact strength increases with increasing the ratio of glass bead additive added to polypropylene and linear low density polyethylene polymer matrices. When 8% glass beads and 12% EOCR elastomer were added to the PP polymer, the

literature [7–11]. The impact strength of 8% glass bead added PP composite decreased by 32% compared to pure PP. In other words, the addition of glass beads to polypropylene increased the brittleness of the composite. In a previous study, Unal et al. [12],

impact strength of the composite decreased by 9.7% compared to the pure PP-H polymer. The results obtained in the experiments are in agreement with previous studies [13, 14]. However, when compared to the impact strength of the glass bead added PP polymer, the impact strength of the 8% glass bead-12% EOCR elastomer added PP composite increased by 32.8%. Wang et al. [11] also stated that the impact strength of the mixture increased by 80% when 10% polyolefin elastomer was added to the PP polymer. Figure 7(a-d) shows pure PP homopolymer, 12% EOCR elastomer added PP mixture, 8% glass bead added PP-H, and 8% glass bead and 12% EOCR elastomer added PP composites in the tensile test.

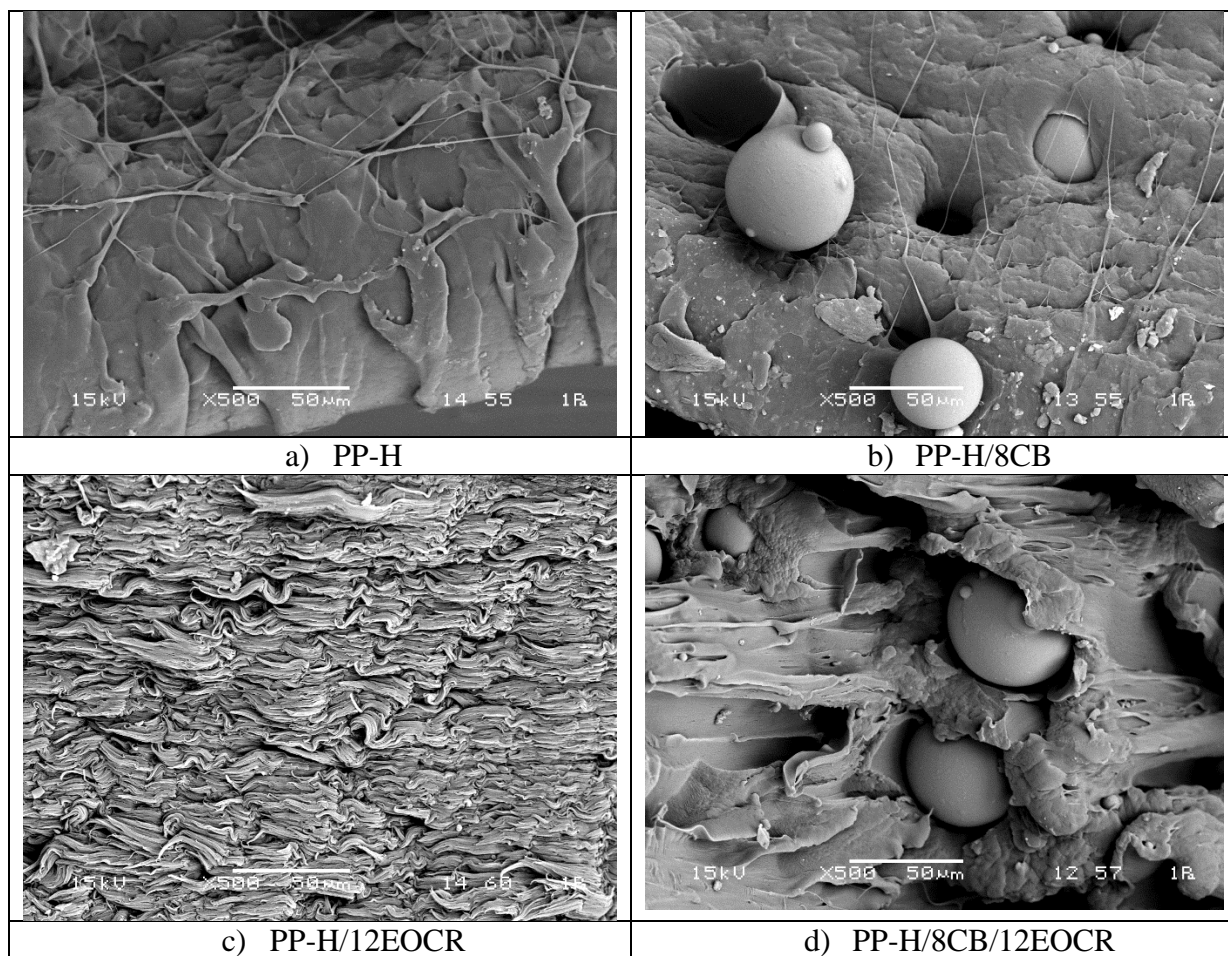


Figure 7. Scanning electron microscope (SEM) images of the fracture surface obtained after tensile test of polymers and composites of a) Pure PP-H polymer, b) PP-H/8CB composite, c) PP-H/12EOCR mixture and d) PP-H/8CB/12EOCR composites used in the experiments.

Microstructure images taken using scanning electron microscopy are given. In Figure 7a, the broken surface image of pure PP-H polymer is given, and finely deformed fibers are seen on the surface. In Figure 7b, the fracture surface image of the pure PP-H/8CB polymer composite after the tensile test is given. In the figure, it is observed that the glass bead PP polymer. The EOCR elastomer added to PP-H initiates the movement of the slip bands in the mixture as a stress concentration factor. There will be stress concentrations around the cracks that will likely occur as a result of slipping. However, EOCR elastomer increases the toughness of the mixture by playing a role in reducing stress concentrations at the crack tips. In Figure 7d, the fracture surface image of

does not bond well with the main matrix and there is a gap around the glass bead. The fracture surface image of the PP-H/12 EOCR polymer mixture is given in Figure 7c. The figure shows a lamellar and layered structure. This can be explained by the tight interfacial adhesion and good compatibility between PP-H and 12 EOCR during the deformation of the the pure PP-H/12EOCR/8CB polymer composite is given. In the figure, it was observed that the glass bead additive in the composite could not bind very well to the polymer matrix and traces were formed on the surface. It has also been determined that the EOCR elastomer softens the polymer matrix material and increases the deformation, resulting in traces.

4. Conclusion

The following results were obtained as a result of the flow, mechanical, and microstructure investigations of pure PP-H polymer and PP-H/12EOCR mixture, PP-H/8CB composite and PP-H/12EOCR/8CB composites used in the experiments.

1- The glass bead additive added to the PP-H polymer main matrix caused a decrease in the melt flow index of the composite, that is, a decrease in fluidity. In addition, the addition of EOCR (12%), which has a higher MFI value than the polymer main matrix, also caused an increase in the fluidity of the composite.

2- When the tensile strengths of PP-H/12EOCR polymer mixture, PP-H/8CB composite, and PP-H/8CB/12EOCR polymer composite are compared according to the tensile strength of the pure PP-H polymer, the tensile strengths are approximately 13%, 16%, and 12.8%, respectively decrease was observed.

3- When compared with the pure PP polymer, the relative modulus of elasticity of PP-8CB composite, PP/12EOCR elastomer mixture and PP/8CB/12EOCR polymer composite were determined as 1.08, 0.907, and 1.023, respectively.

4- The impact strength of the 12% EOCR elastomer added PP polymer blend has increased by approximately 28% compared to the pure polypropylene homopolymer. While the impact strength of the 8% glass bead added PP composite was found to decrease by 32%, the impact strength of the 8% glass bead/ 12% EOCR elastomer added PP composite was decreased by 9.7%.

5- It was observed that the interfacial bond between the PP polymer matrix material and the glass bead additive was not very strong in the microstructure examinations taken under the scanning electron microscope.

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