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Effect of waste glass reinforcements particle size on some properties of HDPE-based composites

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Abstract

Composites were produced by mixing waste glass powder (WGP) of different particle sizes (from 841 µm to smaller than 177 µm) with high-density polyethylene (HDPE) to determine the effects of WGP particle size on some properties of HDPE. WGP was added into HDPE at a rate of 25 wt. % and then mixed with an extruder. As WGP particle sizes decreased, the densities of the composites increased. Adding WGP to HDPE increased the modulus and hardness values of the composites. The lowest flexural modulus was 1079 N/mm^2 in the composite with WGP between 400 and 841 µm, and the highest flexural modulus was determined to be 1671 N/mm^2 with WGP smaller than 177 µm. In addition, WGP decreased the flexural and tensile strengths of the composites. The flexural strength of neat HDPE was determined as 37.2 N/mm^2, the lowest flexural strength was determined as 31.2 N/mm^2 (for WGP size 400 – 841 µm) and the highest flexural strength was determined as 33.2 N/mm^2 (for WGP smaller than 177 µm). According to the thermal analysis results, WGP slightly increased the melting temperature of the HDPE. However, the particle size difference of WGP did not affect the melting temperatures of the polymer much. The melting temperature of neat HDPE was 127.7 °C and the highest melting temperature was determined as 131.3 °C for the composite with WGP filler size between 177 and 250 µm.

1. Introduction

The usage areas of polymer-based composite materials are increasing day by day. Different fillers are used for various purposes in the production of these materials. One of these purposes is to reduce production costs by reducing the use of neat polymers by adding waste materials to polymers as fillers. In addition, in recent years, important steps have been taken to eliminate the environmental impacts of solid waste. For this purpose, new composite products are produced using waste [1]. Many studies have been conducted in the past on polymer-based composite materials produced using different fillers. In some of these studies, polymer-based composite materials were produced using fibrous wood flour and a polymer material, and some selected properties were examined. Stark and Matuana [2] investigated the mechanical properties, FTIR analysis and photodegradation of the sheets they produced using pine wood flour and high-density polyethylene (HDPE) after the ageing test. In the study conducted by Ndiaye et al. [3], it was stated that the bending strength of composite materials produced using pine wood flour and polypropylene (PP) first increased and then decreased with the percentage of wood flour, the elasticity modulus increased and the tensile strength decreased. Mengeloğlu and Karakuş [4] produced composite sheets using eucalyptus wood flour and recycled high-density polyethylene. They reported that as the amount of wood flour in the boards increases, the tensile and bending modulus of elasticity increase and the tensile strength,

elongation at break, bending strength and impact strength decrease. Çavuş [5] stated that in the composites produced using mahogany wood flour and PP, as the percentage of wood flour increases, the flexural strength and flexural modulus increase, the tensile strength decreases, but the tensile modulus increases and the percentage of elongation at break decreases. Narlıoğlu et al. [6] reported that as the wood flour ratio of composite boards produced using pine wood flour and PP increases, the tensile strength and impact resistance decrease and the flexural strength and flexural modulus increase. Bal [7] produced composite boards by mixing Tetra Pak® boxes and pine wood flour with recycled polyethylene and then investigated some selected properties of the boards. In another study, Bal [8] produced HDPE composites using wood flour and walnut shell flour as fillers and investigated some mechanical properties of the produced composites.

In addition to studies using organic materials such as wood flour, there are also studies using some minerals in particle structure as fillers. Essabir et al. [9] produced wood plastic composite using HDPE, talc and apricot shell flour and investigated some mechanical and thermal properties of the composites they produced. Awad et al. [10] investigated the thermal and mechanical properties of marble and granite powder-filled HDPE composites. They reported that as the amount of fillers increases, some mechanical and thermal properties of the composite material improve and as the particle size increases, some mechanical properties are negatively affected. Mustapha et al. [11] investigated the mechanical properties of PP-based composite material filled with calcite and eggshell and reported that these fillers improved the mechanical properties. Sun et al. [12] investigated the effect of graphite particle size in graphite-filled HDPE and ethylene vinyl acetate-based composites on the thermal stability, flammability and mechanical properties of the composites. It has been reported that the tensile strength increases as the graphite particle size decreases. In another study, Zhang et al. [13] investigated the effects of particle size and content on the thermal conductivity and mechanical properties of aluminium oxide-filled HDPE-based composites and stated that the thermal conductivity and tensile strength of the composites increased as the particle size decreased. Additionally, some properties of waste glass powder (WGP) filled polymer-based composites were also investigated in previous studies. Sadik et al. [14] reported that as the amount of WGP in the WGP-filled HDPE-based composites they produced increased, the mechanical properties of the composites decreased. Bhaskar et al. [15] investigated the effect of the amount of glass powder filling on some mechanical properties of polyester-based composites. As a result of the research, it was reported that the glass powder filling initially increased the mechanical properties and decreased as the glass powder filling increased. Heriyanto et al. [16] investigated some properties of PP-based composite material filled with wood powder and glass powder. It was stated that some mechanical properties of the samples using glass powder as filler were higher than those using wood powder.

This study was designed with the idea that adding glass bottle waste to polymers would positively contribute to solid waste recycling. When previous studies were investigated, it was seen that there were a limited number of studies on glass powder-filled HDPE-based composite materials, and the effect of glass powder particle size on the properties of composites was not sufficiently explained. This study aims to investigate some mechanical and thermal properties of HDPE-based composites filled with WGP of different particle sizes.

In addition to polymer-based composites reinforced with organic and inorganic materials, WGP-filled HDPE-based composites can be used instead of neat polymer materials in various areas where low and medium strength is required. For example, formwork, wall and floor coverings in construction and interior and exterior trims of vehicles in the automotive industry.

2. Materials and methods

2.1 Materials

In this study, high-density polyethylene (HDPE) obtained from Petkim (Turkey) was used as the polymer matrix. The density of HDPE is 0.95 g/cm^3, the melting point is 131 °C and the melt flow index is 190 °C/2.16 kg. In addition, glass powder obtained from grinding waste glass bottles (Fig. 1a) resulting from domestic use was used for filling material in HDPE. After cleaning the paper or plastic labels on the glass bottles, they were ground in a high-speed grinder (Demsan Brader 1500). After the glass powder was sieved using

different mesh sieves, they were classified as glass powder groups in five different particle sizes: the particles remaining on the 20 (841 µm), 40 (400 µm), 60 (250 µm) and 80 (177 µm) mesh sieves and the particles passing through the 80 mesh sieve (Fig. 1b). Glass powder that remained above 40, 60 and 80 mesh and passed through an 80 mesh sieve were used in the study.

Figure 1. Materials: (a) waste glass bottles and (b) sieved waste glass powders

2.2 Preparation of composites

The amount of 25 wt. % WGP was used as a filler in the production of composites. In addition, no additives or fillers were used in the control group. The mixing ratios of composite powders are given in Table 1.

Table 1. Mixture ratios of composite powders

Group	1	2	3	4	5
Mesh size passed	–	40	60	80	pan
WGP, wt. %	0	25			
HDPE, wt. %	100	75			

Firstly, WGP and HDPE were mixed in a single-screw extruder for the production of composites (Fig. 2a). The extruder barrel temperature was set to 160, 175 and 190 °C degrees from the feeding section to the exit die section, respectively. Also, the main screw speed was adjusted to 16 rpm. The WGP and HDPE mixture fed into the extruder was not fed at once, but divided into 5 equal parts of 100 g each and fed into the extruder. The extruded materials were obtained as filaments that came out from a 3 mm diameter die. These filaments were handled from the extruder mouth in pieces of about 50 cm in length and left to cool at room conditions on a table (Figs. 2b and 2c). The filaments were broken and turned into pellets after cooling (Fig. 2d). Then, these pellets were extruded again and homogeneous composite filaments were obtained. The filaments were cooled under room conditions and turned into pellets again. These pellets were first transferred into the metal mould. Then, the pellets were placed between electrically heated metal plates without applying pressure and melted at 190 °C. Then, the molten material was transported to the cold press with the metal mould and cooled by pressing under 16 MPa pressure. Then, sheets were obtained with dimensions of 3.5 × 175 × 175 mm. For each sample group, four sheets were produced and test samples were obtained from these plates.

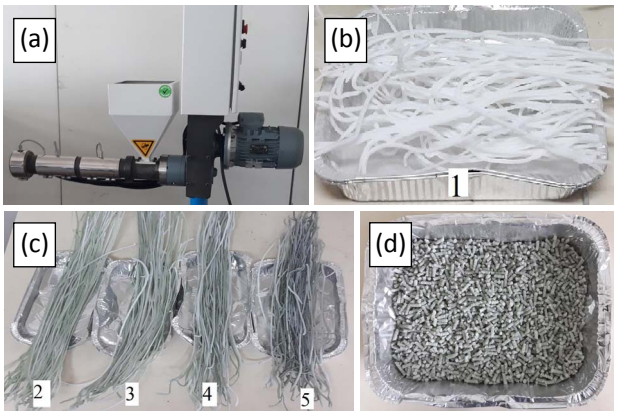

Figure 2. Preparation of composites: (a) single screw extruder, (b) neat HDPE filaments, (c) WGP-filled HDPE filaments and (d) pellets

2.3 Determination of mechanical properties and statistical analysis

Density, flexural and tensile strength and Shore D tests of the samples were carried out according to ASTM D792 [17], ASTM D790 [18], ASTM D638 [19] and ASTM D2240 [20], respectively. Density values were measured by dividing the weight of test samples measuring 3.5 × 20 × 20 mm in air by their volume in water. Flexural and tensile strength tests were performed using the electromechanical universal testing machine (Natek). Flexural strength test samples were prepared with dimensions of 3.5 × 20 × 80 mm. Measurements were made on 12 test samples for each group to determine the flexural tests. The preload was set as 2 N, the support opening as 56 mm, the test speed as 2 mm/min and the end of the test as 80 % of the

maximum measured force in the flexural strength tests. In the flexural tests, the maximum deformation amount at the end of the test is given. The maximum percent of elongation at break was also calculated at the end of the tensile test. A total of 16 test samples were prepared for each group in the tensile tests. Tensile test samples were prepared with dimensions of 3.5 × 20 × 165 mm. A CNC milling machine was used to prepare "dog-bone" shape specimens for tensile tests. The preload was set to 5 N and the test speed was set to 2 mm/min during the tensile test. The elastic modulus values measured during the flexural and tensile tests are also calculated. Hardness tests were carried out on the Shore D testing machine (Loyka LXD-D) using 16 samples for each composite group.

One-way ANOVA and Duncan tests [21] were performed on the data obtained at the end of the tests using a statistical program (SPSS 13.0). In the Duncan multiple comparison test at $\alpha = 0.05$, arithmetic means indicated by the same letter are not significantly different from each other.

2.4 Determination of thermal properties

The thermogravimetric analysis (TGA) and derivative thermogravimetric analysis (DrTGA) were performed using a thermogravimetric analyzer (Shimadzu TA-50). The TGA measurement was carried out between 25 and 600 °C at a heating rate of 10 °C/min in a nitrogen atmosphere with a 50 ml/min flow rate. The differential scanning calorimetry (DSC) analysis was performed using a differential scanning calorimeter (Shimadzu DSC-60). The DSC measurement was performed between 25 and 200 °C. Approximately 10 mg of sample was used for thermal measurements.

3. Results and discussion

The results obtained from the density test are shown in Table 2. It was noticed that the density value increased significantly by adding WGP to HDPE. Similar results have been reported in other studies on glass powder-filled polymer composites [16,22,23]. In this study, the lowest density value was determined as 951 kg/m^3 in the unfilled samples (Group 1) and the highest density value was determined as 1118 kg/m^3 in the HDPE-based composites filled with WGP that passed through an 80 mesh sieve (Group 5). As a result of the ANOVA test, it was seen that the density differences between the test groups were statistically significant.

Table 2. Density of test groups

Group	1	2	3	4	5	p-value
X, kg/m^3	951	1100	1109	1115	1118	< 0.001
SD, kg/m^3	5.6	12.6	8.2	6.0	5.6	
Duncan test	D	C	B	A B	A	

X – arithmetic mean; *SD* – standard deviation

The density differences seen between the groups are due to the differences in the particle sizes of the glass powder added to the polymer matrix. This is related to the increasing surface area as the particle size added to the polymer matrix decreases. Also, as the size of solid particles decreases, they tend to cluster [24]. The densities of the groups are different from each other due to the relationship between the glass particles with different surface areas and the polymer matrix. The lowest density value among composite groups is determined as 1100 kg/m^3. The reason this value is lower than the densities of other composite groups is that glass powder particles between 400 and 841 µm have less surface area than other groups. In addition, large particle-filled groups exhibited lower density values than small particle-filled groups, as the glass powder tended to cluster less than others and the gaps between them were filled with polymer matrix with lower density.

Table 3 shows the flexural strength, flexural modulus and deformation at break values of the test samples. It has been noticed that adding WGP to HDPE polymer negatively affects flexural strength. The lack of improvement in flexural strength was attributed to the low interface bonding of WGP with HDPE and the lack of fibrous structure of WGP. In a study [22], it was reported that adding glass powder to the polymer matrix negatively affected the flexural strength of some polymers. In this study, the control group (Group 1) exhibited a higher flexural strength value than the WGP-filled composite groups, with a flexural strength value of 37.2 N/mm^2. Different values were obtained for the flexural strength of composite groups depending on the particle size of the glass powder. The highest flexural strength among composites was determined as 33.2 N/mm^2 in WGP-filled HDPE-based composite with particle size smaller than 177 µm. It was registered that WGP-filled HDPE-based composites with particle sizes of 400 – 841 µm and 250 – 400 µm (Groups 2 and 3) exhibited flexural strength values of 31.2 and 31.5 N/mm^2, respectively. In addition, it was

noticed that there was no statistically significant difference between these groups. Similarly, it was noticed that there was no statistically significant difference in the flexural strength values of WG-filled HDPE-based composites with particle sizes of 177 – 250 µm and smaller than 177 µm (Groups 4 and 5).

Table 3. Flexural strength, flexural modulus and deformation at bending values of test groups

Group	1	2	3	4	5	p-value
Flexural strength						
X, N/mm^2	37.2	31.2	31.5	32.4	33.2	< 0.001
SD, N/mm^2	1.9	1.2	1.3	1.3	0.9	
Duncan test	A	C	C	B	B C	
Flexural modulus						
X, N/mm^2	1079	1298	1309	1423	1671	< 0.001
SD, N/mm^2	101	81	118	124	154	
Duncan test	D	C	C	B	A	
Deformation at bending						
X, mm	19.0	17.5	17.7	17.8	17.9	< 0.001
SD, mm	0.7	0.7	0.2	0.5	0.5	
Duncan test	A	B	B	B	B	

X – arithmetic mean; *SD* – standard deviation

The addition of WGP to HDPE increased the flexural modulus, which is attributed to the rigid structure of WGP added to the polymer matrix. It has been reported in many studies that the addition of rigid organic (such as lignocellulosic) or inorganic (such as glass powder) materials into polyolefins increases their flexural modulus up to certain limits [16,22,23,25]. In a similar study [16], it was reported that glass powder added to the composite mixture increased the flexural modulus. In this study, the flexural modulus of the control group was determined as 1079 N/mm^2. It has been determined that as the WGP particle size in the composites decreases, the values of the flexural modulus increase. As a result of the statistical examination of the flexural modulus values of the groups, it was seen that there was no statistical difference between the flexural modulus of WGP-filled composites with particle sizes of 400 – 841 µm and 250 – 400 µm (Groups 2 and 3). On the other hand, there is a statistical difference in flexural modulus between the control group and WGP-filled HDPE groups. The highest flexural modulus value among the groups was determined as 1671 N/mm^2 in the WGP-filled HDPE sheet with a particle size smaller than 177 µm (Group 5). In addition, the lowest flexural modulus among the composites was determined as 1298 N/mm^2 in the WGP-filled group with a particle size of 400 – 841 µm (Group 2).

The addition of WGP to HDPE polymer reduced the deformation at bending. The deformation at the bending of the control group (Group 1) was determined as 19 mm. In addition, the deformation at the bending of WGP-filled test samples (Groups 2, 3, 4 and 5) was determined as 17.5, 17.7, 18.8 and 17.9 mm, respectively. It was observed that the values of the amount of deformation at the bending were statistically different between the control group and the composite groups. In addition, there is no statistically significant difference between the deformation at bending values of WGP-filled composite groups.

Tensile strength, tensile modulus and elongation at break values of the samples are given in Table 4. Adding WGP to HDPE decreased the tensile strength by about 26 %. Some researchers have reported that adding inorganic fillers such as glass to the polymer matrix negatively affects tensile strength [22,26-28]. In a study on WGP-filled HDPE-based composites [14], it was reported that adding 20 and 30 wt. % WGP into HDPE reduced the tensile strength value by 30 and 42 %, respectively. In another study [30], it was reported that adding 2.5 wt. % glass powder into recycled polypropylene reduced the tensile strength value from 20.23 MPa to 19.91 MPa.

Table 4. Tensile strength, tensile modulus and elongation at break values of test groups

Group	1	2	3	4	5	p-value
Tensile strength						
X, N/mm^2	23.0	17.4	16.5	17.3	16.7	< 0.001
SD, N/mm^2	1.0	0.6	0.7	0.4	0.5	
Duncan test	A	B	D	B C	C D	
Tensile modulus						
X, N/mm^2	356.3	640.6	744.9	720.0	713.0	< 0.001
SD, N/mm^2	90	145	113	126	133	
Duncan test	C	B	A	A B	A B	
Elongation at break						
X, %	22.2	8.7	10.5	15.7	13.2	< 0.001
SD, mm	3.7	2.4	2.3	3.7	5.0	
Duncan test	A	C	C	B	B	

X – arithmetic mean; *SD* – standard deviation

In this study, the tensile strength value of the control group (Group 1) was determined as 23 N/mm^2. The effect of adding WGP of different particle sizes to HDPE on the tensile strength of the samples was found to be statistically significant. The lowest tensile strength (16.5 N/mm^2) was observed in the WGP-filled group with particle sizes of 250 – 400 µm. The highest tensile strength (17.4 N/mm^2) was determined in the WGP-filled HDPE group with a particle size of 400 – 841 µm. In a study that examined the effect of particle size on tensile strength [12], it was stated that the tensile strength value of small particle-filled composites was higher than that of large particle-filled composites. In the same study, it was reported that the opposite result was obtained when comparing tensile modulus. Another study [29] stated that the tensile strength properties of composites containing smaller-sized tricalcium phosphate fillers at the same participation rate were higher.

Since hard particles have much higher hardness than the polymer matrix, elasticity properties can be improved by adding micro- and nanoparticles to a polymer [27]. In this study, the addition of WGP to HDPE increased the tensile modulus. Similarly, in the study conducted by Kristiawan et al. [30], it was reported that glass powder added to recycled polypropylene increased the tensile modulus. It was noticed that the tensile modulus and elongation at break values of the samples first increased and then decreased as the particle size of the WGP filler added to HDPE decreased. The tensile modulus of the control group (Group 1) was determined as 356.3 N/mm^2. When the tensile modulus values of WGP-filled composites were examined statistically, it was seen that the composite groups were statistically different from each other. The highest tensile modulus value among the composites was determined as 744.9 N/mm^2 in the WGP-filled HDPE group with a particle size of 250 – 400 µm (Group 3). In addition, the lowest tensile modulus of 640.6 N/mm^2 was determined in the WGP-filled HDPE group with a particle size of 400 – 841 µm (Group 2).

In this study, it was noticed that the addition of WGP to HDPE negatively affected the elongation at break. This is similar to previous studies [22,28]. In the study conducted by Sadik et al. [14], the elongation at break value of neat HDPE was determined as 707 %, while the elongation at break values of composites with added 20 and 30 % WGP were determined as 21 and 13 %, respectively. In this study, the elongation at break of the control group was measured as 22.2 %. Adding WGP with a particle size of 400 – 841 µm to HDPE reduced this value to 8.7 %. In addition, it was noticed that the elongation at break of composites exhibited different values depending on the particle size of the WGP filler added to HDPE. The highest elongation at break value was determined as 15.7 % in a WGP-filled HDPE group with particle sizes of 177 – 250 µm. This value decreased to 13.2 % with the decrease in the particle size of the WGP filler. It is thought that the increase and then decrease of the tensile test results with the decrease of WGP size are caused by the tendency of the WGP filler to cluster in the polymer matrix after a certain particle size threshold. In expanded graphite-filled high-density polyethylene/ethylene vinyl acetate-based composites with two different particle sizes (45 and 150 µm), it has been reported that the elongation at break of composites with 45 µm particle size is higher [12].

Information about the strength of materials can be obtained by performing different Shore hardness tests according to material types (rubber, plastic, etc.). The hardness of the samples was determined using the Shore D hardness determination method, which mostly determines the hardness of polyolefin and its composites (Table 5). In this study, adding WGP to HDPE increased Shore D hardness. In previous studies, it was stated that fillers added to the composite mixture increased the hardness of the materials [10,31]. In the study examining the effect of marble and granite powder mixtures on the mechanical properties of HDPE [10], it was reported that the Shore D hardness of the materials decreased as a result of the decrease in the size of the filler added to the polymer. In this study, the Shore D hardness of the control group (Group 1) was determined to be 69.35 and this value increased to approximately 72 due to the addition of WGP. The Shore D hardness of the control group and the hardness of the WGP-filled groups were statistically different. In addition, it was noticed that the Shore D hardness of WGP-filled sheets with different particle sizes was statistically similar.

Table 5. Shore D hardness values of test groups

Group	1	2	3	4	5	p-value
X	69.35	71.65	72.03	71.52	71.59	< 0.001
SD	0.56	0.49	0.46	0.37	0.53	
Duncan test	B	A	A	A	A	

X – arithmetic mean; *SD* – standard deviation

The graph of the TGA results is given in Figure 3. Accordingly, when the degradation values were examined up to 600 °C, it was noticed that 98.6 % of the control group, which was neat HDPE, was degraded. The decomposition amounts of other samples at 600 °C are similar. In addition, it was concluded that the decomposition of the sample taken from Group 5 was the highest, with a remaining mass amount of 24.75 %. As the particle size decreased, the amount of remaining mass at 600 °C increased. The reason for this is that the smaller particle size provides a more homogeneous mixture. It has been noticed that low particle size partially increases the maximum decomposition temperature. It was concluded that especially Group 5 decomposed later than other samples as shown in the thermogram graph. The decomposition temperatures of the groups generally started between 410 and 420 °C and were similar. The particle size of the WGP added into the composite delayed the degradation by approximately 5 °C.

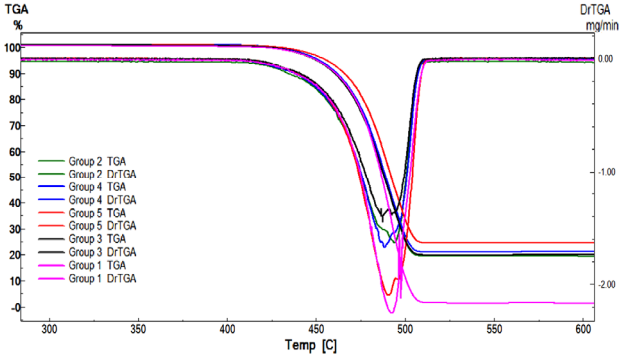

Figure 3. TGA and DrTGA curves of test groups

This study showed that the maximum decomposition temperatures of HDPE-based composites with glass powder added at the micron level are similar to those conducted by Sadik et al. [14]. In a study by Daramola et al. [32], composites were produced using lignocellulosic origin silica and titanium dioxide. As the amount of silica in the composite increased, the decomposition temperature increased. In particular, the micron-sized particle structure increased the maximum decomposition temperature to 430 °C.

The melting temperatures of the composites are similar to each other according to the examination shown in Figure 4. Group 1 consists of 100 % HDPE. Accordingly, it was noticed that the melting temperature of the polymer was 127.7 °C. In addition, the crystallisation temperature was determined to be 106.3 °C.

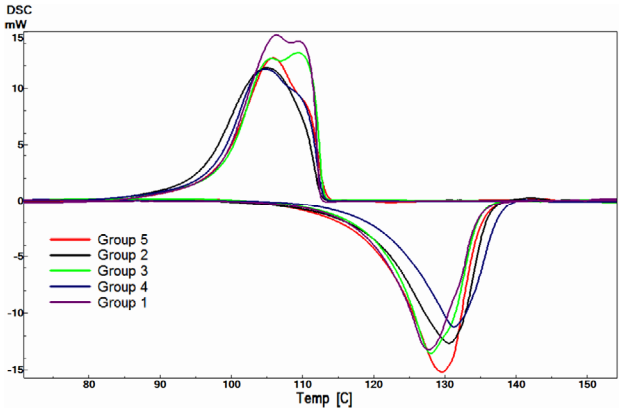

Figure 4. DSC curves of test groups

As can be noticed from Table 6, WGP added to HDPE slightly increases the melting temperature. In particular, particle size does not negatively affect the melting temperature. Melting temperatures among groups vary between 127.7 and 131.3 °C. The differences are thought to be due to differences in the amount of glass powder contained in the samples taken into the machine for testing [33].

Table 6. DSC values of test groups

Group	1	2	3	4	5
Melting temperature, °C	127.7	130.6	128.3	131.3	129.6
Melting energy, J/g	156.0	157.3	139.2	115.2	155.2
Crystallisation temperature, °C	106.3	105.0	109.4	104.8	106.0
Crystallisation energy, J/g	190.0	151.6	154.5	134.7	131.3

4. Conclusion

In this study, the effect of WPG particle size on the physical, mechanical and thermal properties of HDPE-based composites was determined. It was noticed that the effect of WPG particle size on the density of HDPE-based composites was evident. The decrease in WGP particle size increased the density of the composites.

The mechanical properties of the composites changed depending on the particle size of the WGP filler. The flexural strength of the composites increased slightly as the WGP particle size decreased, while the flexural modulus increased more than the flexural strength. In addition, the tensile strength decreased slightly as the WGP particle size decreased, whereas the tensile modulus and elongation at break increased.

According to the thermal analysis results, the decrease in WGP particle size partially increased the maximum decomposition temperature. In addition, it was noticed that WGP added to the polymer slightly increased the melting temperature and the particle size did not have a negative effect on the melting temperature.

According to the results obtained in this study, it was concluded that WGP-filled HDPE-based composites can be used in places requiring low and medium strength, such as siding, flooring and automobile interior and exterior trims. In future studies, determining the relationships between different particle sizes and different addition rates of WPG will contribute positively to this issue.

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