Effect of Content and Particle Size of Used Beverage Carton Pieces on the Properties of HDPE Composites

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This study investigated the tensile properties and thermal behavior of virgin and hot press molded HDPE composites filled with different particle size and content of used beverage cartons which were the Tetra Pak® cartons. The mechanical properties of the composites were positively influenced by particle size of the used beverage carton, such that the smallest particle size gave the highest tensile strength and tensile modulus. The tensile strength of the specimens decreased with increasing filler content (40 to 70 wt%), while the tensile modulus rose. Furthermore, the filler size and its content affected the thermal behavior of the specimens. Calorimetry analysis of composite specimens showed that melting temperature and enthalpy values of virgin HDPE and recycled-HDPE decreased with increasing Tetra Pak® content. In all composite groups produced by adding Tetra Pak®, the degree of crystallinity decreased as a function of Tetra Pak® addition compared to the pure HDPE. Increasing particle size adversely affected the crystallization degree, which decreased with increasing particle size while the HDPE maintained its crystalline form. As for the recycled-HDPE composites, the degree of crystallization was reduced by increasing the Tetra Pak® content, but this was still noticeably higher than that of the HDPE.

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INTRODUCTION

Paper and cardboard packaging is the largest proportion by weight with 33.2%, followed by flexible plastics 25.5%, rigid plastics 18.7%, metal 12.1%, glass 5.8%, 4.7 other packaging materials (Fernandez 2022). Recently, in addition to the virgin thermoplastics, the use of multilayer beverage cartons (Tetra Pak®) is composed of three materials organized in six layers: paper (75 wt%), low density polyethylene (LDPE, 20 wt%), and aluminum (5 wt%) (Ayrilmis *et al.* 2008). However, the amount of their waste has increased more and more. A thin aluminum sheet is also incorporated in the composition to improve UV resistance and oxygen barrier for aseptic cartons (Avella *et al.* 2009). Several studies were published on the utilization of used beverage carton in the production of high value-added products (Ayrilmis *et al.* 2008, 2013a; Selim *et al.* 2010;

Umezuruike 2013; Yilgor *et al.* 2014; Bekhta *et al.* 2016; Robertson 2021; Bal 2022). One of the potential applications of the used Tetra Pak® cartons is as a filler in polymer composites. Natural fibers have noticeable advantages as filler in the thermoplastics such as low cost, good tensile properties, renewability, low density, and low abrasive effect to the metal parts in the extruder or injection machine (Rohit and Dixit 2016).

The use of used beverage cartons as filler in thermoplastics was investigated by several researchers (Ayrilmis *et al.* 2008; Sanchez-Cadena *et al.* 2013; Bekhta *et al.* 2016; Rohit and Dixit 2016; Robertson 2021). The results of these studies showed that the particles of the used beverage cartons could be successfully used as filler in thermoplastic composites (Bektas *et al.* 2005; Avella *et al.* 2009; Ebadi *et al.* 2016, 2018). However, the effect of particle size of the used beverage cartons was not investigated. Previous studies reported that particle size of filler affects the mechanical properties of the polymer composites (Fu *et al.* 2008; Aldousiri *et al.* 2013; Deepak *et al.* 2019; Delviawan *et al.* 2019; Aranda-García *et al.* 2020; Hubbe and Grigsby 2020; Sinha *et al.* 2020; Kuzmin *et al.* 2022).

This study focused on the effect of used beverage carton content (Tetra Pak®) and its particle size on the water absorption and tensile properties of the HDPE composites. The differential scanning calorimetry (DSC) and morphological analysis of the specimens were also investigated. Different amounts of the Tetra Pak® filler (40 to 70 wt%) and three different particle sizes were studied in the experiments. High amount of the Tetra Pak® filler was used in the study because it was aimed to be processed by extrusion and used for decking and siding applications. Such products require high filling (40 to 70%), unlike injection molding, where 10 to 20% filling is sufficient. Moreover, high filler ratio reduces the cost of the composite, by replacing the expensive polymer matrix HDPE.

EXPERIMENTAL

As polymer matrix, virgin HDPE granules (grade: 273-83) manufactured by Kazanorgsintez PJSC company, Russia (density: 0.95 g/cm^3; melt flow rate: 0.5 g/10 min at 190 °C and a load of 50 N; yield stress at tensile: 22.6 MPa; tensile elongation: 700%) were used. In addition to the virgin polymer, recycled HDPE samples obtained from the used plastic bottles were used in the experiments (density: 0.94 g/cm^3, melt flow rate 0.9 g/10 min). The used beverage cartons (Tetra Pak™) served as filler in the HDPE composites. After washing in warm water and drying, the beverage products were chipped in a rotary knife chipper. Then, the particles were ground in a mill with three knives. The resulting particles as filler were sieved through screening machine and then classified three different size which were <630 μm, 630 to 1000 μm, and > 1000.

Compounding of the components was carried out on a laboratory rotary batch mixer Hakke Rheomix 600 OS with roller rotors. The mixing temperature, rotor speed, and the mixing time were 150 °C, 50 rpm, and 10 min, respectively. The resulting mixture was compressed into a thin panel on a Gibitre™ hydraulic hot-press at a temperature of 150 °C and a pressure of 3 MPa. The amount of the filler in the HDPE polymer matrix was gradually increased from 40 to 70 wt% (by 10 wt% increment). The experimental design is given in Table 1. The tensile test specimens in the form of a dog bone were punched out from the thin panels with dimensions of 200 mm x 200 mm x 1 mm.

Determination of Tensile Properties of the Composite

Tensile testing was carried out on a universal testing machine (Gotech UAI-7000 M) with a testing speed of 1 mm/min at a temperature of 23±1°C according to ISO 527 standard. The water absorption of the specimens with a size of 3 mm x 3 mm x 1 mm was determined by exposure in water for 24 h at the room temperature. The density of the composites was measured on an H-200L density meter with an ultra-high resolution of 0.001 g/cm^3.

Table 1. Experimental Design

Specimen Code	Filler Content (%wt)	HDPE	Recycled HDPE
		Particle size (μm)	Particle size (μm)
1	40	630	630
2	40	630 to 1000	1000
3	40	> 1000	> 1000
4	50	630	630
5	50	630 to 1000	1000
6	50	> 1000	> 1000
7	60	630	630
8	60	630 to 1000	1000
9	60	> 1000	> 1000
10	70	630	630
11	70	630 to 1000	1000
12	70	> 1000	> 1000

DSC Analysis

Melting and crystallization behavior of the composite specimens produced by adding Tetra Pak® into HDPE in different sizes (630, 1000, and >1000 μm) and in different ratios (40%, 50%, 60%, and 70%) were determined on Perkin Elmer-8000 device by heat flow method. Its calibration was performed with zinc (Zn), indium (In) and tin (Sn) metals. DSC analysis was performed according to the ASTM D3418 standard. Amounts of 7 to 10 mg of the composite specimen were weighed and put into a pan. First, the specimens were heated up to 200 °C with a heating rate of 10 °C and when the temperature reached 200 °C, it was kept for 2 min. Then, the specimens were cooled to 10 °C with a cooling rate of 10 °C /min to achieve dynamic crystallization. After waiting for 2 min, the atmosphere was heated up to 200 °C with the same heating rate. All these processes were applied at a flow rate of 50 mL/min in nitrogen environment to prevent oxidation effect. The degree of the crystallization was determined using the enthalpy value obtained from the second melting graph curve in the Eq. 1 (Hristov and Vasileva 2003),

$$X_c(\%) = (\Delta H_m/\Delta H_{om}) \times 100 \tag{1}$$

where ΔH_m is the melting enthalpy (J/g) value calculated from the curve obtained during the second melting, and ΔH_{om} is the melting enthalpy of the 100% crystalline form of HDPE (289 J/g).

Morphological Analysis

The evaluation of dispersion and distribution of Tetra Pak® particles in the polymer matrix composite was carried out by SEM analysis. The specimens were first immersed in liquid nitrogen and then broken. The broken surface was covered with gold coating by applying the spraying method. Clear SEM images were obtained because the gold-plated surface increased the electrical conductivity. A JEOL Neo Scope JSM-5000 brand SEM with an acceleration voltage of 10 kV was used for the images.

RESULTS AND DISCUSSION

Tensile Properties

The tensile strength, tensile modulus, and elongation at break of the composites are given in Table 2. As expected, the tensile modulus of the specimens improved with increasing amount of the Tetra Pak® filler. The tensile modulus of the specimens was greatly increased by the increased amount of the Tetra Pak® filler while the tensile strength moderately decreased. For example, the tensile strength, tensile modulus, and elongation at break of the specimens with 40 wt% Tetra Pak® particles were 17.2, 1370, and 2.8 MPa, respectively, while they were found to be 14.1, 2350, and 0.7 MPa when the amount of the Tetra Pak® content reached to 70 wt% in the HDPE matrix. The results of the tensile modulus showed that the modulus of the composite was strongly affected by the modulus of each constituent. The addition of a rigid material into the polymer, for example cellulose fibers, enhances the stiffness of polymer. The Tetra Pak® particles improved the stress transfer from the HDPE to the reinforcing material which was the particles as well the deflection of the composites.

Table 2. Water Absorption and Mechanical Properties of the HDPE Composites with Different Size and Content of the Tetra Pak® Filler

Composite Composition (wt%)	Property	Unit	Particle Size of the Filler (µm)		
			< 630	630 to 1000	> 1000
40% Tetra Pak+60% HDPE	Tensile strength	MPa	17.2	17.2	16.6
	Modulus tensile	MPa	1370	1310	1230
	Elongation at break (%)	%	2.8	3.2	2.4
	Water absorption (24-h)	%	1.1	0.6	1.3
	Density	kg/m^3	1115	1150	1088
50% Tetra Pak+50% HDPE	Tensile strength	MPa	16.5	16.1	15.8
	Modulus tensile	MPa	1950	1680	1550
	Elongation at break (%)	%	1.2	1.5	2
	Water absorption (24-h)	%	1.8	2.0	2.1
	Density	kg/m^3	1121	1175	1305
60% Tetra Pak+40% HDPE	Tensile strength	MPa	14.3	13.1	13.3
	Modulus tensile	MPa	1990	1790	1640
	Elongation at break (%)	%	0.8	1.1	1.4
	Water absorption (24-h)	%	3.3	3.9	4.1
	Density	kg/m^3	1185	1195	1194
70% Tetra Pak+30% HDPE	Tensile strength	MPa	14.1	13.1	13.2
	Modulus tensile	MPa	2350	1840	1820
	Elongation at break (%)	%	0.7	1.1	1.1
	Water absorption (24-h)	%	4.6	5.7	5.9
	Density	kg/m^3	1283	1286	1292

The decrease in the particle size increased the tensile strength and tensile modulus at the same loading level of the Tetra Pak®. The tensile strength of the specimens decreased as a function of the increasing particle content due to the fact that the polymer matrix was not enough to bond the Tetra Pak® particles as a binder. Similarly, the amount of the HDPE matrix as an adhesive was not sufficient for the interfacial bond when the number of the Tetra Pak® particles increased. This was due to the fact that overall surface area of the smaller particles was higher than that of the larger particles. According to the results of the tensile properties, it can be said that the best results were found in the Tetra Pak® size which was less than 630 µm.

The elongation at break of the specimens decreased as a function of increasing Tetra Pak® filler. The reduction in the elongation at break of the specimens having a higher content of the Tetra Pak® filler may be explained by the low elongation of Tetra Pak® filler that restricted the flow of HDPE matrix. Tetra Pak® filler contains aluminum foil and paper layer. The ductility of the HDPE composites with Tetra Pak® filler was noticeably decreased.

Water absorption of the biocomposites increased from 1.1 to 4.6% when the amount of the Tetra Pak® filler content increased from 40 to 70 wt% in the HDPE (Table 2). This result was expected because the filler contained the paper layer prepared from wood pulp. The addition of the Tetra Pak® filler into the HDPE matrix increased the water permeability. This behavior depended on the Tetra Pak® content and its particle size. Higher water absorption of the HDPE with a high amount of the particles of the used beverage carton reflected that the lignocellulosic fibers with the hydroxyl groups were not well encapsulated by the polymer matrix. Some of the lignocellulosic fibers were easily accessible to water. The lower water absorption of the composites with a low amount of the used beverage carton could be because the fibers were fully covered by the melted thermoplastic material, which protected them from water. The increased particle size of the Tetra Pak® increased the water absorption of the HDPE composites at all the loading levels. For example, at the 50 wt% content of the Tetra Pak®, the water absorption of the composites having a size of lower than 630 µm Tetra Pak® was 1.2%, while it was 2.1% for the composites with a size of higher than 1000 µm of Tetra Pak®. When the loading level of the Tetra Pak® filler was increased in the HDPE, the total surface area of the particles increased, causing more of the hydroxyl groups to be exposed to the water. The hydrophilic nature of the natural fibers proportionally increased the water absorption of the composites with higher Tetra Pak® loading.

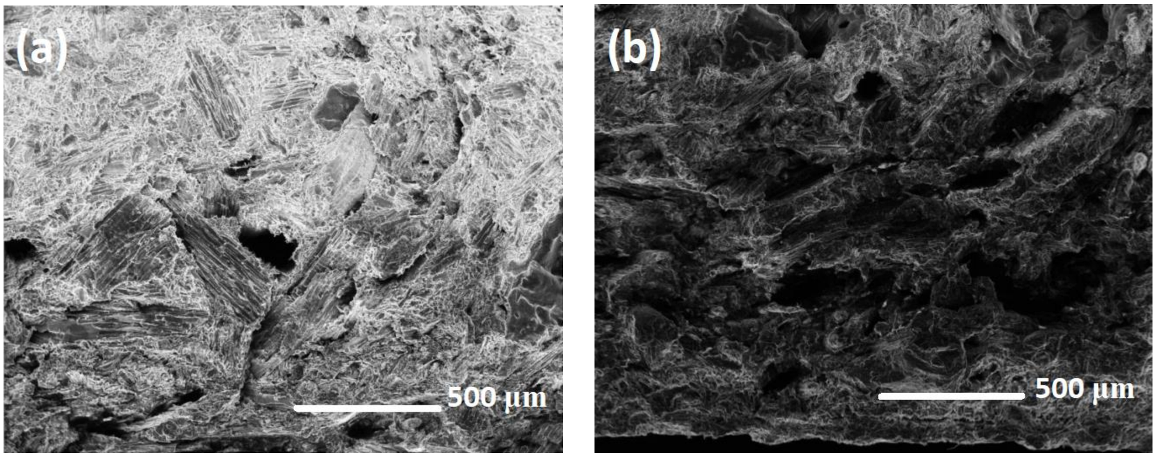

Fig. 1. SEM images of HDPE/Tetra Pak® composites. A) 40 wt% filler content. B) 70 wt% filler content (SEM magnification x60).

The HDPE has no hydrophilic character because of its non-polar hydrophobic nature. Previous studies reported that the increase in the loading level of fillers above a certain level in the polymer matrix reduced the tensile strength of the thermoplastic composites due to the poor interfacial bonding between the filler and the HDPE matrix (Ayrilmis *et al.* 2008, 2013b; Tufan and Ayrilmis 2016). Furthermore, the increased number of microgaps in the HDPE composites as a function of increasing amount of the Tetra Pak® filler caused higher water absorption. This was confirmed by the SEM images. The SEM micrographs of the HDPE composites produced from the lowest (40 wt%) and highest (70 wt%) particle content of the Tetra Pak® are shown in Fig. 1. The number of microgaps increased with the amount of the Tetra Pak® particles in the HDPE matrix (Fig 1B).

The physical and mechanical properties of the composites produced from Tetra Pak® filler and recycled HDPE are given in Table 3. The size of the Tetra Pak® filler was more than 1000 μm. The tensile modulus of the specimens increased with the addition of the filler while the tensile strength decreased. Especially, when the amount of the particle was increased from 60 wt% to 70 wt%, a considerable increase in the tensile modulus of the specimens was determined. This can be explained by the fact that the tensile modulus of the wood fibers is significantly higher than that of the HDPE (Ayrilmis and Jarusombuti 2011).

The tensile properties of the composites produced with the virgin HDPE were higher than those of the specimens produced with the recycled HDPE. Water absorption in the specimens with the recycled HDPE was higher than the composites containing the virgin HDPE.

Table 3. Recycled HDPE Composites with Tetra Pak® Particles

Property	**Tetra Pak Filler Content (%wt)**			
	40	**50**	**60**	**70**
Tensile strength (MPa)	16.3	15.8	13.1	12.9
Modulus tensile (MPa)	1540	1650	1660	1940
Elongation at break (%)	2.2	1.7	1.4	1.1
Water absorption (24-h) (%)	2.1	3.2	5.3	6.9
Density (kg/m^3)	1134	1190	1217	1233

DSC Analysis

The DSC data of the composites produced by adding Tetra Pak® with 630 μm dimensions are given in Table 4. The DSC analysis of composites produced by adding 40 to 70% of Tetra Pak® filler into the HDPE (particle size: 630, 1000, and >1000 μm) and recycled HDPE (particle size: >1000 μm) were performed. Melting and cooling curves were obtained in the DSC diagrams. The melting and crystallization peaks of these curves are presented in Figs. 2 to 4. The crystal form formation of the composites took place in the usual way, and no new crystal form was formed. There were differences between melting and crystallization temperature peaks depending on particle sizes. Therefore, the results were evaluated according to particle size. Cold crystallization starts (T_c onset), end (T_c end set), peak (T_c), cold crystallization enthalpy (ΔH_c), melting temperature (T_m), melting enthalpy (ΔH_m), and crystallization degree (X_c) of composites determined.

When the amount of the Tetra Pak was increased from 40 to 70 wt%, the values of the melting temperature (T_m) (132.8-131.7 °C) and melting enthalpy (ΔH_m) values (199.7 to 97.6 J g^{-1}) decreased. Especially, when the amount of the Tetra Pak increased from 60 wt% to 70 wt% in the HDPE matrix, the enthalpy value of cold crystallization (ΔH_c) and enthalpy value of melting (ΔH_m) decreased sharply (Table 4). The degree of crystallization decreased from 69.1 to 33.8% with the addition of 40% and 70% Tetra Pak, respectively. However, cold crystallization temperature peak values (114.6 to 115.7 °C) increased.

Table 4. DSC Data of HDPE Composites with Tetra Pak® Particles

Tetra Pak Content (%)	Particle Size (μm)	$T_{c\ \mathrm{onset}}$ (°C)	$T_{c\ \mathrm{endset}}$ (°C)	$T_{c\ \mathrm{peak}}$ (°C)	ΔH_c (J g^{-1})	T_m (°C)	ΔH_m (J g^{-1})	X_c (%)
40	630	118.9	107.3	114.6	199.6	132.8	199.7	69.1
50	630	1189	108.1	114.8	169.1	132.5	190.1	65.8
60	630	119.1	108.3	115.4	166.9	132.4	180.9	62.6
70	630	118.9	108.4	115.7	94.1	131.7	97.6	33.8
40	1000	118.9	103.8	113.1	186.1	132.5	184.0	63.7
50	1000	118.7	105.3	113.2	180.8	132.4	179.3	62.0
60	1000	118.2	106.0	113.8	146.3	132.2	111.8	38.7
70	1000	118.3	109.7	115.0	116.4	130.7	85.9	29.7
70	> 1000	119.8	110.8	117.3	132.9	133.5	129.8	44.9

Note: $T_{c\ \mathrm{onset}}$: cold crystallization peak onset temperature, $T_{c\ \mathrm{peak}}$: cold crystallization peak maximum temperature, ΔH_c: enthalpy value of cold crystallization, T_m: melting temperature, ΔH_m: enthalpy value of melting, X_c: crystallinity degree

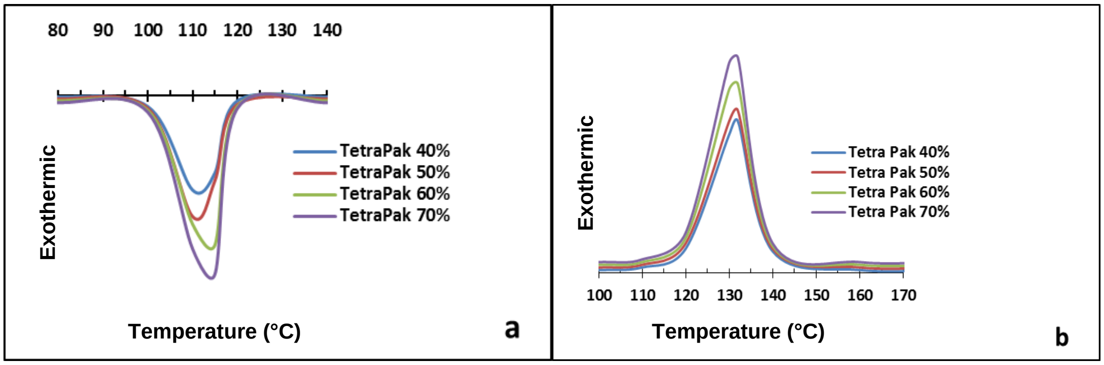

Fig. 2. DSC diagram of HDPE-Tetra Pak (particle size 630 μm) composites: (a) melting and (b) crystallization diagram

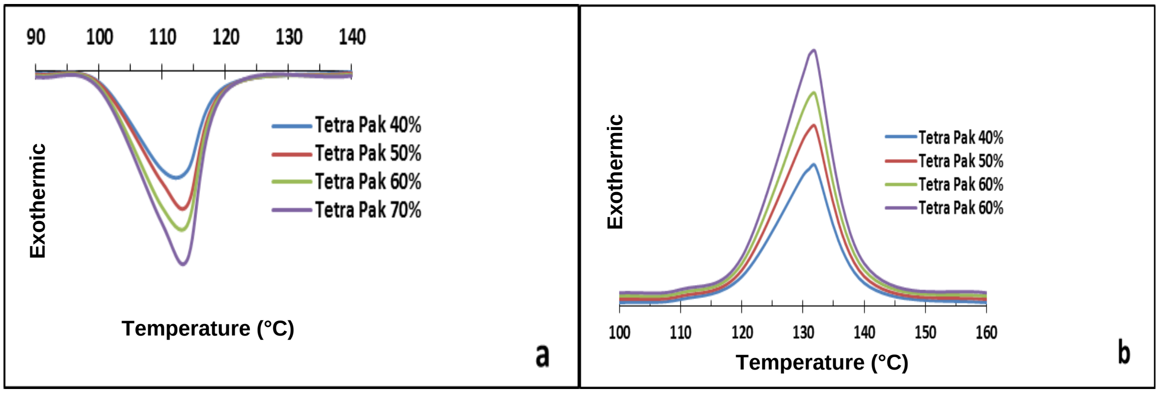

Fig. 3. DSC diagram of HDPE-Tetra Pak (particle size 1000 μm) composites: (a) melting and (b) crystallization diagram

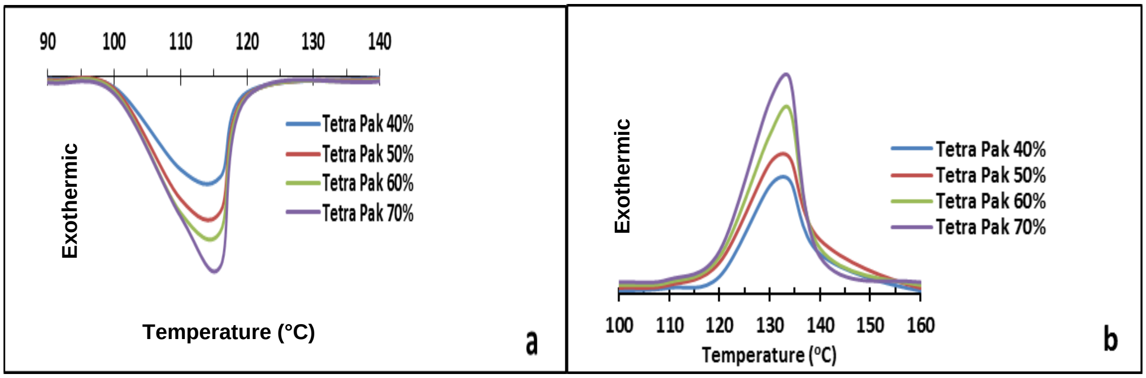

Fig. 4. DSC diagram of recycled HDPE-Tetra Pak (particle size >1000 μm) composites: (a) melting and (b) crystallization diagram

The DSC data obtained by adding 1000 μm Tetra Pak® are given in Table 4. Similar results were obtained with increasing particle size. As a result of increasing the addition of Tetra Pak® from 40% to 70%, T_m (132.5 to 130.7 °C), ΔH_m (184 to 85.9 J g^{-1}), ΔH_c (186.1 to 116.4 °C), and X_c (63.7% to 29.7%) decreased.

DSC data obtained by recycled HDPE are given in Table 5. The results in the test samples produced from recycled HDPE with the addition of Tetra Pak® with particle sizes >1000 μm showed similar trends to the results of samples with particle sizes of 630 and 1000 μm. However, the values of all parameters were found to be higher in certain proportions in recycled HDPE specimens. By increasing the amount of Tetra Pak® addition from 40% to 70%, the values of T_m (133.9 to 133.5 °C), ΔH_m (216.3 to 129.8 J g^{-1}), ΔH_c (231.5 to 132.9 J g^{-1}), and X_c (74.8% to 44.9%) decreased. Similarly, T_c values (116.8 to 117.3 °C) increased, similar to the samples with 600 and 1000 μm sizes.

Depending on the increase in Tetra Pak® content, melting temperature and enthalpy values of the HDPE and recycled HDPE decreased. In all the composite groups produced by adding Tetra Pak®, the degree of crystallinity decreased as a function of Tetra Pak® addition as compared to the pure HDPE. The fiber fraction containing Tetra Pak® had an inhibitory influence on the crystallization of the polymer matrix (Colom *et al.* 2000; Stark and Matuana 2004). The reason was the dilution effect with the increase of Tetra Pak® content added to HDPE and recycled HDPE matrix (Amash and Zugenmaier 2000). The T_c increased slightly for all particle sizes.

Table 5. DSC data of Recycled HDPE/Tetra Pak® Composites

Filler Content (%wt)	Particle Size (μm)	$T_{c\ on\ set}$ (°C)	$T_{c\ end\ set}$ (°C)	$T_{c\ Peak}$ (°C)	ΔH_c (J g^{-1})	T_m (°C)	ΔH_m (J g^{-1})	X_c (%)
40	> 1000	120.7	110.0	116.8	231.5	133.9	216.3	74.8
50	> 1000	120.5	110.4	117.0	240.6	133.8	198.4	68.7
60	> 1000	120.4	110.5	117.1	141.8	133.6	142.0	49.1
70	> 1000	119.8	110.8	117.3	132.9	133.5	129.8	44.9

Note: $T_{c\ onset}$: cold crystallization peak onset temperature, $T_{c\ peak}$: cold crystallization peak maximum temperature, ΔH_c: enthalpy value of cold crystallization, T_m: melting temperature, ΔH_m: enthalpy value of melting, X_c: crystallinity degree.

The Tetra Pak® content caused this increase by acting as a heterogeneous nucleation within the HDPE and recycled HDPE matrix (Bouafif *et al.* 2009; Huang *et al.* 2015). In all specimens, the decrease in the X_c and ΔH_c values with the increase of Tetra Pak® content and size was noteworthy. The melting and crystallization values of the DSC diagrams depending on the particle sizes and Tetra Pak® content are given in Table 5.

CONCLUSIONS

1. The increasing content of the Tetra Pak® filler in the high-density polyethylene (HDPE) composites improved the tensile modulus while the tensile strength decreased. The particle size also had an impact on the tensile properties of the specimens. The tensile strength and modulus of the specimens decreased with decreasing particle size at the same loading level of the Tetra Pak® filler.
2. As the Tetra Pak® content increased, the melting temperature of the composite decreased but the crystallization temperature increased. The degree of crystallization decreased with increasing Tetra Pak® content. Increasing particle size negatively affected the crystallization degree and caused it to decrease. However, it was important that the HDPE maintained its crystalline form. In the recycled HDPE composites, the degree of crystallization decreased as the Tetra Pak® content increased, but this value was still noticeably higher than that of the crystallization degree of the HDPE.
3. The best tensile properties were found using Tetra Pak® with a size of less than 630 μm. The virgin HDPE showed better mechanical performance than recycled HDPE.

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Data Availability Statement

Data available on request from the authors.

Declaration of Conflicting Interests

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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