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Quality Sustainability Of Recycled High Density Polyethylene (HDPE)

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Abstract

The main way to reduce the generated large amounts of polymer waste is their recycling, their return to the processes of processing and reuse. The main and most commonly used method is mechanical recycling, in which some of the characteristics of the processed material deteriorate. It is known that the properties of the recyclate are most significantly affected by the homogeneity and the degree of aging of the end-of-life plastic products. Since polypropylene is a major pollutant in waste high-density polyethylene, this study tracked to what extent and what content of polypropylene leads to a decrease in the properties of the regranulate. For this purpose, the authors tracked the rheological and mechanical characteristics of high-density polyethylene with polypropylene impurities. The percentage content of polypropylene was established, which affects the mechanical properties of the regranulate. The processability, quality and homogeneity of the collected high-density polyethylene after use were studied, as well as the possibilities for improving the quality of the granulate produced from it. Data from a study of the behavior of fresh high-density polyethylene with admixtures of polypropylene recyclate in a certain percentage were compared with those of high-density polyethylene collected after use. In this way, the working group established the influence of the processing process on the rheology and mechanical properties of the recyclate and the degree of degradation of the polymers.

Keywords: High density polyethylene, polypropylene, mechanical recycling, properties, waste.

INTRODUCTION

The development of the industry over the past 20 years, both nationally and globally, has led to a continuous increase in the amount of recycled polymers. This in turn is the reason for the generation of large amounts of polymer waste. Polyethylene (PE) is one of the most versatile commercial polymers. Its semi-crystalline nature allows it to be processed over a wide temperature range. The crystalline phase of the polymer provides strength, while the amorphous phase provides flexibility [1]. PE is classified as a viscoelastic material. It is characterized by a high strength-to-density ratio, good chemical resistance, and resistance to high temperatures. Its melting point is generally higher than most other polymers in its weight class. This makes it easily processed. It has excellent tensile strength, a low coefficient of friction, and low moisture absorption. These properties make it one of the largest pollutants in nature due to the large-scale production of virgin PE [2]. The main way to reduce the need for fresh material in the production of polymer products and protect the environment is to recycle waste and return it to the processing processes. According to the latest data, this will contribute to reducing the carbon footprint by 15% by 2050, taking into account the current production rates [3]. Research and development worldwide is focused on developing more environmentally

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friendly methods for producing polymers from recycled material. The production of virgin high-density polyethylene (HDPE) is based on the raw material ethylene, which is obtained from natural gas through a technique called "cracking". This method, although fundamental, is energy-intensive and results in a significant carbon footprint. Mechanical recycling is one way to mitigate the carbon footprint, as products are ground, designed, remelted and then processed into new products, creating a closed-loop life cycle. This process significantly reduces the need to produce virgin material [4]. Within the framework of circular economy policies, the European Parliament, through a number of project proposals, stimulates the development and implementation of effective technologies for the recovery and reuse of polyolefins, by creating a regulatory framework that will provide guidance on the requirements for the properties of the recycled material and its respective application in various areas of industry, such as the production of household appliances, disposable utensils, food contact foils, automotive and aircraft manufacturing, medicine and pharmacy.

The increased interest in plastics recycling is the result of three important trends.

1. The increase in the production and use of plastics;
2. The constantly increasing prices of crude oil;
3. The growing need to reduce harmful emissions in order to protect the environment and transform the conventional linear economy of the plastics industry into a circular one [5-9].

Mechanical recycling is one of the most effective and commonly used methods for processing polymer waste. However, the recycling industry has to deal with problems related to the difficulty of predicting and obtaining constant, uniform mechanical properties depending on the origin of the material and the recycling process used [10]. The use of recycled plastics is a good alternative, but in most cases, the material is subjected to repeated reprocessing. This leads to a change in the structure and mechanical properties of the material [11]. The properties of mechanically recycled polymers do not remain the same due to degradation by thermal processes, mechanical stresses and oxidation during their processing. Degradation affects the molecular structure and therefore the mechanical properties of the material [12, 13] thus quality is the main issue when working with mechanically recycled products [14]. In addition to mechanical, rheological properties also change [15-17]. For example, some authors report that HDPE shows different behavior during extrusion. At higher temperatures, longer chains are subjected to tensile and elongational stresses [18, 19]. The presence of oxygen leads to thermo-oxidative processes and rupture of the polymer chains, while shorter chains are less sensitive to mechanical stresses but are more prone to branching [20]. Other authors report that chain branching is a dominant factor up to about 30 extrusion cycles. Blending virgin and recycled HDPE up to 70%, recycled content can improve mechanical and rheological properties, bringing them closer to those of virgin HDPE. Blending medium density polyethylene (MDPE) with recycled (r) HDPE in a 50/50 ratio has shown very good impact resistance. Another experimental study reported that increasing the weight content of rHDPE can significantly reduce the flexural and tensile strengths of v/rHDPE blends [21]. Although HDPE is relatively easy to recycle, the heat and stresses of the processing process can change the structure and color of the polymer, which in turn leads to a decrease in the rheological and mechanical properties of the recycled material. Antioxidants, light stabilizers and impact modifiers are commonly used to improve the recyclability and performance of recycled HDPE. There are reports of the use of metal soaps, mineral agents and metal oxides to neutralize catalyst residues left over from polymer production. Primary antioxidants are used to scavenge free radicals that are formed when polymer chains are broken down by high processing temperatures. Secondary antioxidants are used to intervene during the polymer melt phase [22-25]. All this leads to the conclusion that the properties of the recycled material cannot be predicted. Different applications require specific material properties, which necessitates the introduction of different additives. Recycled HDPE has many industrial applications, for example in construction for the production of geomembranes. HDPE geomembranes (GSE HD) require exceptional chemical resistance, mechanical properties, resistance to cracking, resistance to heat aging. GSE HD must have excellent resistance to UV radiation considering their outdoor use [26]. For the application of the material in drinking and wastewater pipes, in gas distribution systems, anticorrosion properties and high tensile strength, high chemical resistance, resistance to microorganisms and corrosion, resistance to UV rays are relied on [27]. For the production of bottles, containers and bags for food, the requirements for HDPE are not limited to organoleptic properties. The material must have hardness, impact resistance and a low degree of deformation, moisture impermeability, which are achieved with special additives - stabilizers, modifiers, etc. All this must be taken into account in the recycling processes and the design of new formulations [28]. For each specific polymer, it is necessary to study the influence of processing processes, material aging, differences between recycled and virgin material from both chemical and mechanical points of view. In accordance with the above, our study focused on investigating the influence of different amounts of PP admixture on the density, impact strength and rheology of the recycled material.

METHODS AND MATERIALS

Materials

In this work, an analysis of the mechanical and rheological properties of virgin high-density polyethylene (vHDPE), brand Polypropylene Buplen Homopolymer 6131, used for the production of household appliances, garden furniture, thin-film parts with complex configurations, for high-speed high-pressure casting of parts for medical and technical purposes [29] and recycled high-density polyethylene (rHDPE) with impurities of recycled polypropylene (rPP) was carried out.

TEST METHODS

The following standardized methods of testing and analysis were used:

1. *Mass-flow rate (MFR) and melt volume index (MVR) in accordance with EN ISO 1133*-The melt flow rate measurements were made on an Instron MF 20 digital instrument, under the following conditions:For vHDPE and rHDPE - temperature 190^{o} C and pressure with a load of 2.16 kg.For vPP and rPP - temperature 230^{o} C and pressure with a load of 2.16 kg.;
2. *Differential scanning calorimetry (DSC) in accordance with EN ISO 11357-3*; The analysis was performed on a Polyma instrument, DSC21400A-0339-L. The measurements were performed according to the above-cited standard. The mass of the samples was 5.0 ± 0.1 mg. The heating and cooling rate was 20 C/min. All measurements were performed in a nitrogen atmosphere. The results are presented as average values of three replicates.
3. *Ash content in accordance with EN ISO 3451-1, method A* - direct calcination; Method "A" was used - direct calcination, i.e. by burning the organic matter and heating the residue at high temperature until a constant mass is reached by burning in a muffle furnace model: AproTerm6L. The ash content is expressed as a percentage of the mass and is calculated by the formula:
   $(m_1/m_0) \times 100\% = A\%$

Where:
m_0 is the mass, in grams, of the dried test sample;
m_1 is the mass, in grams, of the ash obtained;
A% is the result of the ash content.

4. *Density in accordance with EN ISO 1183-1, method A;* An immersion method was used for solid plastics that do not contain voids (except powders).

The density was calculated using the formula:

$$\rho s = \frac{ms,A \times \rho IL}{ms,A - ms,IL}$$

Where:
ms, A – apparent mass of the sample body in air, in g;
ms, IL - apparent mass of the sample in impression fluid, in g;
ρIL – density of the impression fluid in g/cm^3;
The results are expressed as the average of three measurements.

5. *Mechanical properties*: Charpy impact strength in accordance with EN ISO 179 -1; Samples with dimensions: 80mm (length) x 10mm (width) x 4mm (thickness) were extruded on a laboratory extruder, corresponding to Notched Charpy impact test specimens EN ISO 179 -1/1eA. 10 test specimens were tested.
6. *Tensile properties. Part 2*: Test conditions for plastics for moulding and extrusion, EN ISO 527-2:2012

Tensile test results according to EN ISO 527-2 were obtained at room temperature and a constant machine speed of 50 mm/min. Five test specimens were tested from each sample. The final result is the average of the five measurements.

RESULTS AND DISCUSSION

In this experiment, samples from different batches of mechanically recycled HDPE were initially separated for characterization. The amount of PP contained in the recyclate was determined by DSC analysis. The minimum PP content in the recyclate was 3%, the maximum was 8%. The results are presented in Table 1.

The next step of the experiment was to test specimens made of virgin high-density polyethylene (vHDPE), intended for household products. Rheological, mechanical parameters, ash content and density were monitored. The results are presented in Table 2.

The results in Table 2 are compared with the results of testing rHDPE with different rPP contents in the composition.

The following Figures present the results of the examination of these batches.

From the graph in Figure 1 it is observed that in batch 1, which has 3% rPP, the density is 0.958 g/cm³. In batches from 2 to 5 the density decreases to 0.955 g/cm³ to 0.952 g/cm³, and in batch 6 it reaches 0.943 g/cm³. The difference between batch 1 and batch 6 is a 1.6% decrease in the value, while the difference between the density of the virgin material and that of batch 6 is 1.3%. It could be said that the change in this characteristic is insignificant when varying different rPP contents in the recycled HDPE.

Table 1. RPP content in different batches of mechanically recycled HDPE.

Batches	rPP content, %	r HDPE content, %
1	3	97
2	4	96
3	5	95
4	6	94
5	7	93
6	8	92

Table 2. vHDPE Test Results:

Characteristic	Value	Test method
Density	0,955 g/cm³	BDS EN ISO 1183
Mass-Flow Rate at190°C /2,16 kg	0,27 g/10 min	BDS EN ISO 1133
Tensile strength	31 MPa	BDS EN ISO 527-2
Elongation at break	750%	BDS EN ISO 527-2
Ash Content	0,02%	BDS EN ISO 3451
Shore Hardness	67 (Shore D)	BDS EN ISO 868

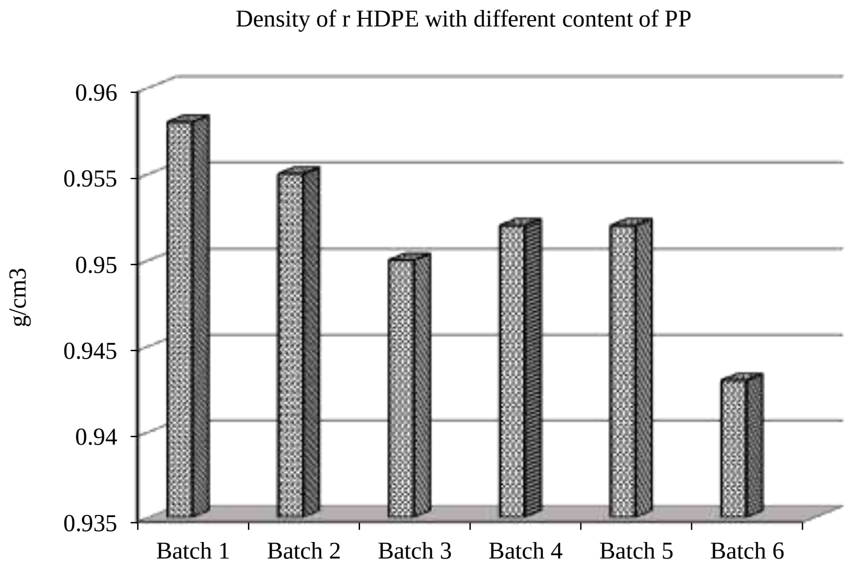

Figure 1. Density measurement of rHDPE with Different rPP Content in The Composition, in accordance with EN ISO 1183-1, Method A.

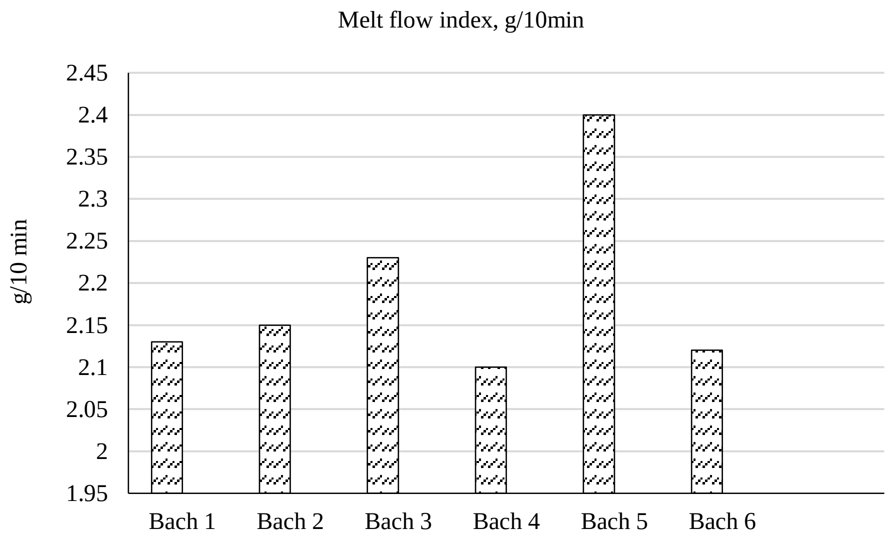

Figure 2. Measurement of melt flow rate (MFR) in accordance with EN ISO 1133-1 of rHDPE with different rPP content in the composition.

The data from Figure 2 show that at 3% PP admixture in the composition of rHDPE, the melt flowability is the same as at 8% PP content. At 5% PP the melt index by mass is 2.23 g/10min, and at 7% PP content it is 2.4g/10min. That is, a change is observed that does not follow any dependence. These variations in the results are most likely due to the influence of other impurities in the recyclate or multiple processing of the material.

Figure 3 represents the change in the Charpy impact strength of rHDPE with different rPP content, it is noticeable that with increasing rPP content, the impact strength decreases. At 3% rPP the value is 27.16 kJ/m2, and at 8% the strength has decreased to 25.8 kJ/m2, which is a 5% decrease in the indicator. Compared to HDPE, PP has a lower impact strength. This could be attributed to the fact that PP is prone to brittleness due to its sensitivity to oxidation, as well as processing cycles.

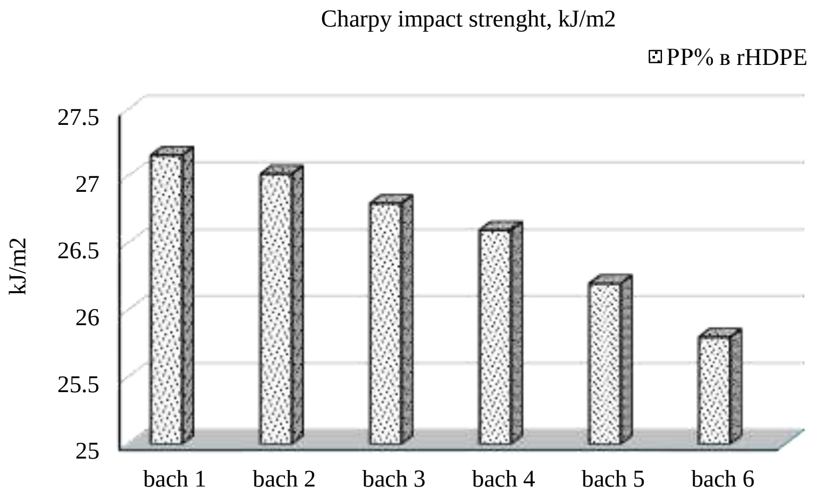

Figure 3. Change in charpy impact strength in accordance with EN ISO 179 -1, depending on The PP content in the recyclate.

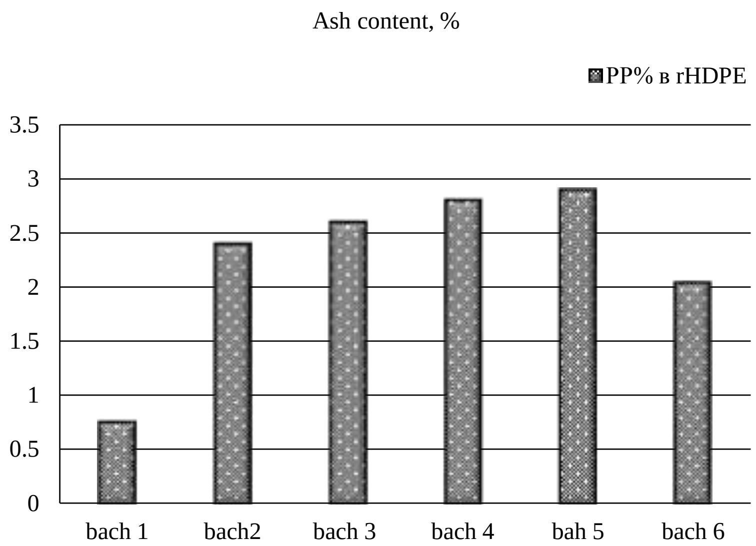

Figure 4. Change in ash content in accordance with EN ISO 3451-1, method a – direct calcination depending on the amount of pp in the recyclate.

The graph in Figure 4 shows the change in ash content with different amounts of rPP in the recyclate. At 3% rPP there is 0.75% ash in the recyclate, from 4% rPP to 7% rPP the content increases by 0.6%, and at 8% rPP the value is 2.04%. These data confirm the fact that the ash content in the recycled material is not affected by the different percentage of rPP in the composition.

Figure 5 shows the change in tensile strength depending on the content of rPP in the recyclate.

Figure 5 presents the results of the change in tensile strength due to the change in the percentage of rPP in the composition of rHDPE. The difference between batch 1 with 3% rPP and batch 6 with 8% rPP is a decrease in the value by 24%.

Figure 6 presents the change in elongation at break depending on the content of rPP in the recycled material.

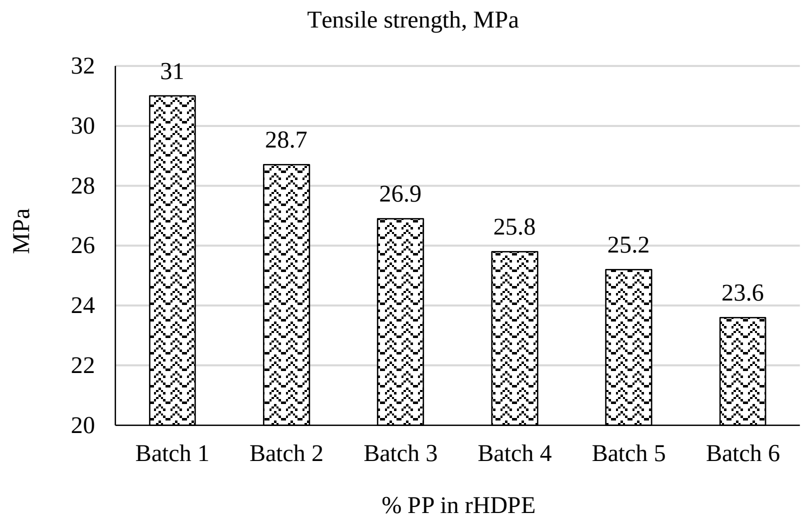

Figure 5. Change in tensile strength in accordance with EN ISO 527-2, depending on The rPP Content in the recyclate.

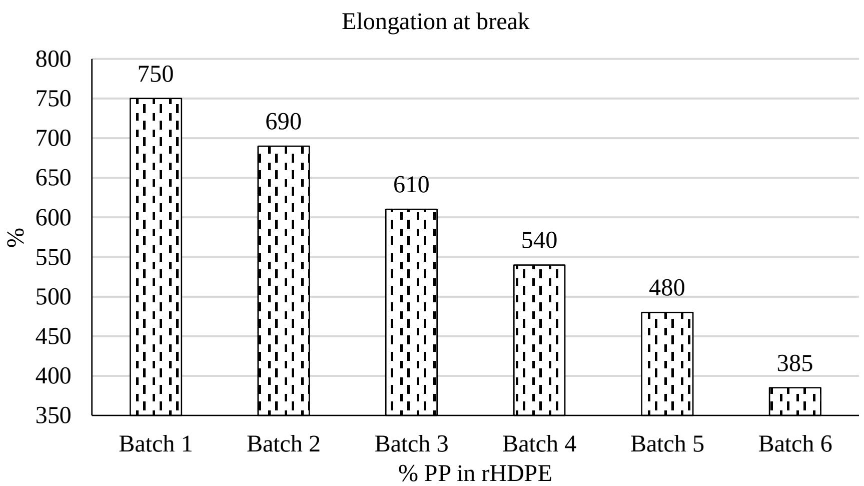

Figure 6. Change in elongation at break in accordance with EN ISO 527-2, depending on The PP content in the recyclate.

As can be seen from the graph, the difference between the elongation at break of batch 1 with 3% rPP in the composition and the elongation at break of batch 6 with 8% rPP in the composition is 48%.

The reduction in the indicators in Figure 5 and Figure 6 is probably due to both the processes of breaking and branching of the polymer chain that occur during the processing process, or possibly crosslinking during the reprocessing of the compound [30].

After the type of polypropylene contained in the recycled rHDPE material was established, tests were performed and the results of rPP and vPP with the same properties and purpose were compared. The following table presents the test results.

The data in Table 3 show that recycled polypropylene has similar characteristics (melt index by mass, density and tensile strength) to the fresh material. The minor differences in the indicators are probably a consequence of thermal processing and atmospheric aging. The following graphs present the results of testing vHDPE and rHDPE with different rPP contents in the composition. The blends are compared in terms of “Charpy impact strength” and “melt index by mass”.

Table 3. RPP and VPP Test results.

Property	Unit	Value			Test method
		Requirement	vPP	rPP	
Melt flow rate at 230^{O}C and loading 2,16 kg.	g/10 min	16-25	17	19	ASTM D 1238 - 94a EN ISO 1133
Density at 23^{O}C	kg/m^3	898-905	899	901	ASTM D 792-91 EN ISO 1183-1, Method A
Tensile strength at flow, at 50 mm/min, not less than	MPa	-	29	27	ASTM D 638-94b EN ISO 527-2

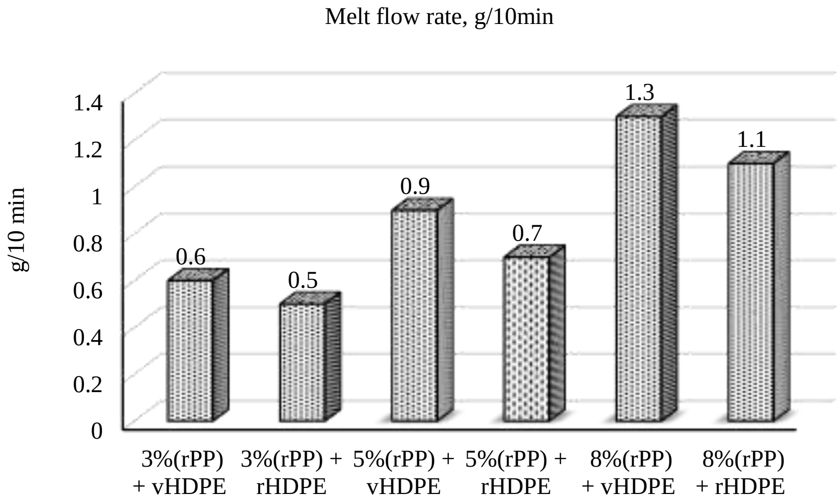

Figure 7. Measurement of Melt Flow Rate (MFR) in Accordance with EN ISO 1133-1 of rHDPE and vHDPE with Different rPP content in the composition.

In Figure7 we observe the change in rheology of rHDPE and vHDPE compositions with 3.5 and 8% rPP. The decrease in the indicators is respectively 16% for a composition with 3% rPP, 22% for a composition with 5% rPP and 15% for a composition with 8% rPP.

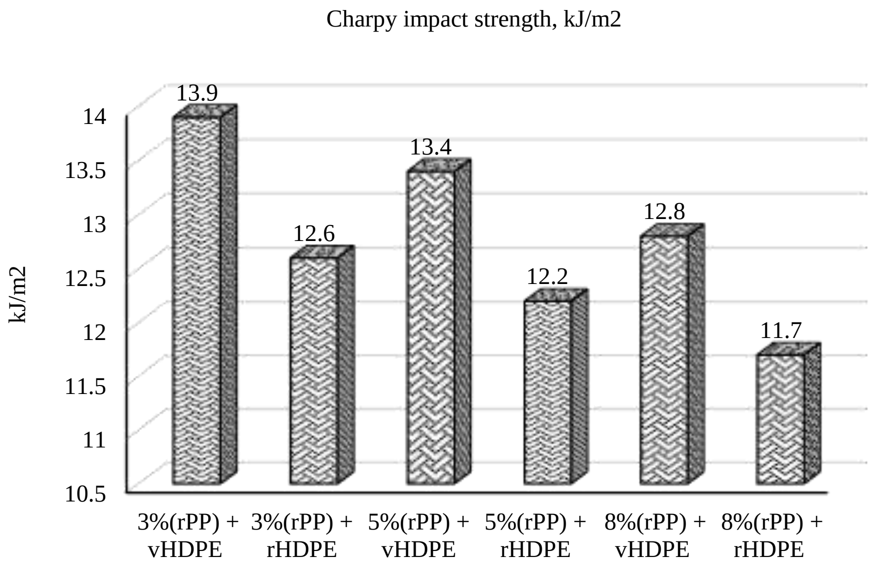

Figure 8. Change in charpy impact strength in accordance with EN ISO 179 -1, of rHDPE and vHDPE with different rPP content in the composition

The impact strength, as seen from the graph in Fig. 8, in rHDPE and vHDPE compositions with 3%, 5% and 8% rPP decreases by approximately 9% in all three compositions. Considering the results in Fig. 7 and Figure 8, it can be assumed that the decrease in performance in the mixtures with rPP and rHDPE, comparing them with rPP and vHDPE, is probably due to the contributions of heat treatment in the aging process of the material.

CONCLUSIONS

Experimental analysis shows that the relative density of the regranule, the ash content of the recycled material and the melt index by mass are not significantly affected by the different percentage of rPP in the composition.

The Charpy impact strength decreases with increasing rPP content in the rHDPE composition. Increasing the amount of rPP in the recycled HDPE composition has a significant impact on the tensile strength of the material.

The melt index by mass of a blend of recycled HDPE with rPP is reduced by an average of 18% compared to that obtained from a blend of virgin HDPE with rPP. A decrease of 9% in Charpy impact strength is observed between the two compositions. for rHDPE and rPP.

This information could serve as a basis for designing recycled HDPE products for various Industrial sectors.

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