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Original Paper

A New Approach to Recycling PET Bottles by Blending Polyethylene- Terephthalate and HDPE Using Chemical Reactions in One Step Process

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Abstract

The overall goal of this study is to propose a new approach to simplify the recycling process for producing high quality material from the waste of beverage bottles and other sources at a lower cost by using mechanical recycling and chemical reactions inside injection molding machines in a one-step process. The water and soft drink bottles are made of PET material, while the caps are made mainly of HDPE. The blends of PET and HDPE are incompatible. The resulted material has poor mechanical properties due to the immiscibility of polymer blend components, phase separation, and lack of interfacial adhesion. (HDPE-g-MA) was found to be an excellent reactive compatibilizer for the PET/HDPE blend. For the purpose of investigating the mechanical and morphological properties in this study, blends of rPET and virgin HDPE in weight compositions of 80/20, and 60/40 were modified with concentrations of 2 and 5 wt percent of (HDPE-g-MA). The addition of 4,4-Methylenebis (phenyl isocyanate) and compatibilizer increased the viscosity and molecular weight of the blends while also improving miscibility and interaction bonds between molecules, resulting in a significant reduction in interfacial tension and improved phase dispersion and adhesion via interpenetration and entanglements at the interface. The rPET segments have been grafted onto HDPE backbone chains. Since the crystallinity of HDPE is low, the crystallinity and crystallization temperature of rPET in the blends were reduced. The mechanical characteristics were improved by achieving uniform phase morphology. The main finding of this study is that the immiscible rPET/HDPE materials can be treated and processed in a single step inside the injection molding process. The one-step process is sufficient because it shows high *improvement in the mechanical characteristics, while the two-step process, namely the extrusion followed by injection molding, improves fewer mechanical characteristics and is more expensive.*

Keywords

Poly (ethylene terephthalate), High Density Polyethylene, PET recycling, injection molding, D.S.C., compatibilizer, chain extender, tensile testing, Rheology, SEM

1. Introduction

PET is a recyclable thermoplastic polymer that can be easily moulded. PET has become the main target for plastic recycling because of its wide use in the beverage industry. Both the overall performance and the affordable cost of PET make it an excellent choice in the packaging and manufacturing of soft drink bottles and other beverages, as well as the production of fiber. PET's advantages includes its high compressive and tensile strength, good chemical resistance, high impact resistance, transparency, fracture resistance, gas permeability resistance, and low cost. As a result of PET's resistance to biodegradation, large amounts of PET waste accumulate over time. HDPE has a high molecular weight, good stiffness, and chemical resistance to a wide range of chemicals. On the other hand, HDPE has poor weathering, high flammability, high thermal expansion, and is sensitive to stress cracking.

Polymer blending is the mixing of at least two polymers to generate a new material with different physical properties. Polymer blends can be broadly divided into three categories: 1. Immiscible or heterogeneous polymer blends: The constituent polymers exist in separate phases, and their glass transition temperatures are observed. 2. Compatible polymer blends are macroscopically displayed as having homogeneous physical characteristics as a result of sufficiently strong interactions between the component polymer phases. 3. Miscible or homogeneous polymer blends: These mixtures are often made from polymers with similar chemical structures, resulting in a polymer blend with a single-phase structure and one glass transition temperature is observed. HDPE and PET are immiscible polymers in the molten state, and their blends lead to high interfacial tension, low interfacial adhesion, and poor mechanical properties. To improve these properties, it is necessary to prevent the development of segregated phases during the mixing stage by introducing a third component in situ that acts as a compatibilizer at the interface between the two polymers. This kind of treatment has been investigated in many researches (Manita et al., 2002; Chen et al., 2018., Taghavi1 et al., 2018; Lusinchi et al., 2000; Shahrajabian et al., 2019; Pracella et al., 2002.).

PET and HDPE can be combined in post-consumer water and soft drink bottles as well as multilayer food packaging. Every year, billions of tonnes of post-consumer food packaging are created. Only a small percentage of this waste is recycled, with the rest being incinerated or disposed of. Chemical recycling and mechanical recycling are two important techniques for recycling waste PET.

Chemical methods are used to obtain the initial monomers in the polymer structure, which are then used to rebuild PET and other chemicals. The primary disadvantage of PET chemical recycling is its high cost. Separation, washing, drying, and melting are common methods for mechanically recycling

PET waste. It is critical to maintain a high molecular mass in the stages following mechanical recycling. Multiple rounds of reprocessing (recycling) may alter the molecular structure and molecular weight of PET, affecting the rheological and mechanical properties of the material. The generated rPET is very brittle, which is the main disadvantage of mechanical recycling. As a result, combining rPET with other polymers, particularly polyolefin, may result in a significant mechanical and processability equilibrium. Currently, the addition of an effective compatibilizer containing functional groups (e.g. carboxyl, anhydride, epoxy, etc.) capable of reacting with the other polymer component, is used during the melt processing to obtain a new product with good rheology and morphology using extrusion and pelletizing production units to obtain a compatible blend of rPET/HDPE. This addition can solve the problem of incompatibility between the two polymers (polyesters and polyolefin). Numerous efforts have been made to improve the methods for separating and purifying plastic components from industrial and municipal wastes. However, the cost of manufacturing recycled polymers is frequently higher than that of virgin products.

Blending scrap polymers under appropriate conditions may provide an alternative route for marketing recycled compounds with acceptable cost-performance characteristics and application potential in the packaging, domestic, and engineering sectors. Generally, reprocessing commingled scrap materials using conventional manufacturing methods is not economically viable. Recycling success is highly dependent on the development of new technologies and manufacturing processes for polymer systems composed of multiple components. Despite the high cost of component separation, the production of value-added compounds through the melt blending of post-consumer plastics appears to be economically viable when considering the extensive application potential of reactive injection moulding processes. Many studies have demonstrated that melt blending can be used to obtain good and consistent properties for plastic waste (Ahmadlouydarab et al., 2020; Chen et al., 2015; Torres et al., 2000; Abdul Razak et al., 2013; Abdelbary & Seileem, 2021).

1.1 Research Aim

The main aim of this research is to develop and implement a cost-effective new scientific production technology method that allows compatibilization of immiscible rPET and HDPE blends through injection moulding. In terms of industrial application, this processing method is critical because it demonstrates that blends with good morphological and mechanical properties can be produced in a single processing step. Simplifying recycling processes can increase the desire of manufacturers to rely on waste plastic material produced in their own facilities, which can expand the spectrum of recycling producers. This can be accomplished by avoiding sorting and skipping the costly extrusion and pelletizing steps, which are only available at huge recycling facilities. This includes the direct use of rPET/HDPE flakes with additives in injection moulding, which results in additional cost savings and broadens the range of stakeholders involved in waste plastic. The generated raw material has better rheology, morphlogy, chemical, thermal, and mechanical properties because it has only been subjected to one thermal cycle.

2. Experimental

2.1 Materials

In this study, three types of materials were characterized and investigated: 1- post-consumer PET bottles. The scrap of PET has been collected in a heterogeneous deposit that has been contaminated by many different types of PET bottles. The scrap PET was gathered from a variety of sources, including colored and colorless post-consumer bottles. These bottles were mechanically broken down into 2mm flakes. They were then washed and dried for three hours at 150 degrees Celsius to get the moisture content down to less than 0.005% of the original weight. 2- virgin HDPE was supplied by OQ. It has a melting point of 131 degrees Celsius and a density of 0.965 g/cm3. 3- The chemical extender 4,4-Methylene bis (phenyl isocyanate) 98 percent was supplied by Aldrich, Germany, 4- (HDPE-g-MA) was supplied by Aldrich-Germany as well.

2.2 Samples Preparation

Two different techniques were used to prepare the samples:

2.2.1 Injection Molding (One Step Processing)

The samples were prepared by injecting the blended materials in one step with and without additives.

2.2.2 Extrusion & Pelletizing and Injection Molding (Two-step Processing)

It includes extrusion and pelletizing, followed by injection molding. The samples were prepared by mixing different ratios of rPET flakes and HDPE virgin in the presence of the additives inside twin extruders and pelletizing units to obtain equal-sized pellets. Then the pellets were used in situ injection molding to obtain standard test samples. For both techniques, the samples were injected inside a mould insert to obtain a standard tensile test specimen (ISO 527 type 1A). GUC Plastic Academy (PIMC) employed a Systec Sumitomo Demag 160 tonne-250 gm shot size injection moulding machine to run this experiment. The samples were prepared according to the compositions described in Table 1. The process parameters were kept fixed during preparing the samples. Barrel temperature: 240–265 ℃—Screw speed: 100 revolutions per minute, Injection speed: 70 mm/s Cooling time: 12 seconds, injection time: 3 seconds, hold pressure: 2 seconds, total cycle time: 24 seconds. As elementary results, concentrations of rPET/HDPE of 60/40 and 80/20 were chosen to investigate the effects of additional additions to the blends according to the composition ratios described in Table 2. The samples (A1, A2, A3, and A4) were prepared using a fixed ratio of 0.25 wt percent (phenyl-isocyanate) as a chain extender and a concentration of 2 and 5% (HDPE-g-MA) as a compatibilizer.

Table 1. Compositions of the Samples without Additives (wt. %)

Sample	rPET %	HDPE %	(HDPE-g-MA) %	(phenyl -isocyanate)%
S1	100	0	0	0
S2	0	100	0	0
S3	20	80	0	0
S4	40	60	0	0
S5	60	40	0	0
S6	80	20	0	0

Table 2. Compositions of the Samples with Additives (wt. %)

Sample	rPET %	HDPE %	(PP-g-MA) %	(phenyl -isocyanate) %
A1	80	20	2	0.25
A2	80	20	5	0.25
A3	60	40	2	0.25
A4	60	40	5	0.25

3. Characterization Techniques

3.1 Tensile Testing

Tensile properties were obtained using the Zwick/Roel-Z100 tensile testing machine at a test speed of 10 mm/min. To ensure the integrity of the tests, the straightness of the grips was checked during each set-up, as was the load associated with gripping (30-50 N).

3.2 SEM Analysis

Using a scanning electron microscope (JEOL JSM 6510lv), we were able to examine the blends' morphology and determine the influence of the compatibilizer and additives on the microstructure development required for efficient rPET dispersion in HDPE. The samples were fractured prior to measurement by submerging them in liquid nitrogen for 5 minutes.

3.3 Infrared Spectroscopy

FTIR was used to measure the absorbance bands of the different samples. The measurements were performed using an ANICOLT IS10 FTIR spectrometer controlled by the OMNIC 8 computer software (Thermo Electron Scientific Instruments Corporation, Madison, Wisconsin, USA). The samples were prepared using an "Aspecac" press instrument, with a 7 mm diameter cutting die applied at a pressure of 2 tonnes. All spectra were taken with 8 cm^{-1} resolution in the range of 4000-500 cm^{-1}.

3.4 Differential Scanning Calorimetry (DSC)

DSC was used to measure the thermal transition temperatures (Tg and Tm) and heat content Δ H with subsequent cooling and heating cycles (10 C▫/min) ranging from 0 to 300 C▫ and the degree of the crystallinity of the different phases of the blend.

3.5 Rheology

The rheological characteristics of the samples were investigated using a capillary rheometer. The experiment was used to indicate the changes in melt viscosity induced by multiple processing steps.

3.5.1 Sample Preparation

The samples were prepared as injection molded dog-bone shapes (ISO 527 type 1A). A circular disc of diameter 12 mm and a thickness of 2 mm were cut with a die of diameter 12 mm from both ends of the dog bone shape.

3.5.2 Rheometer Measurement

Parallel stainless steel plates with a diameter of 25 mm (original ETC plates from Bohlin) were installed in the rheometer. The standard approach for determining the zero gap distance is followed. The sample disc, with a diameter of 12 mm, is centrally located in the bottom plate. With a 3 mm gap spacing, the setup is heated to 270 C▫. When the temperature reaches 250 C▫, the separation distance is reduced to 0.7 mm. The chamber is then opened repeatedly for a brief period of time and the gap distance changed until the whole gap is filled. For a 0.4 mm gap distance, the shear rate is changed between a high shear rate of 20/s and a low shear rate of 0.05/s. This range has a total of 21 measurement points that are evenly dispersed on a logarithmic scale. The measurement takes around 200 seconds. The run was done twice after each temperature measurement. Each run lasts around 15 minutes. The temperatures for oscillatory measurement were 270 degrees Celsius, and the thermal equilibrium duration was 1 minute. The frequency was varied between 0.5Hz and 50Hz while maintaining a constant strain of 0.5 percent. On a logarithmic scale, the 21 distinct frequencies were spread evenly. The test was conducted at low frequency, increased to high frequency, and then returned to low frequency. The gap is opened and the PET sample is withdrawn after the measurement (Abdelbary & Seileem, 2021).

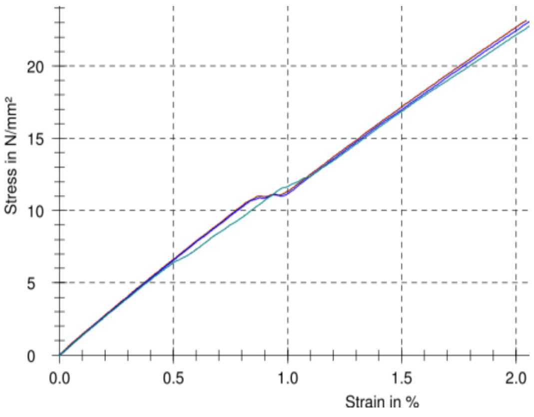


Figure 1. rPET Stress-Strain Curve

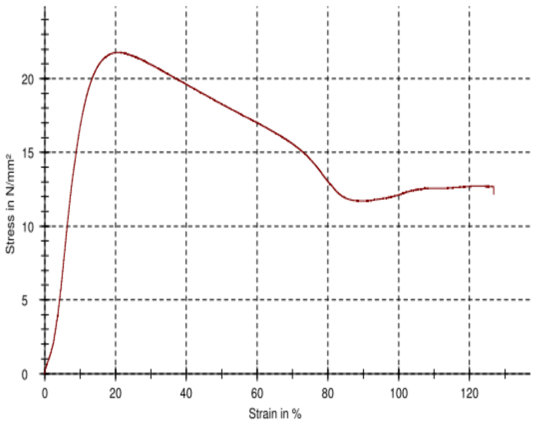


Figure 2. HDPE Stress-Strain Curve

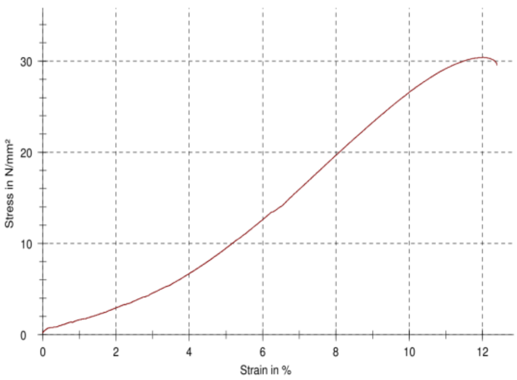


Figure 3. Stress Strain Curve of Incompatibilized Blend of rPET80/HDPE20-0.25% EX

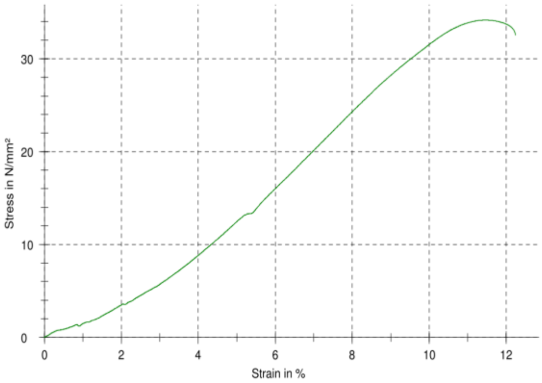


Figure 4. Stress Strain Curve of Compatibilized Blend of rPET80/HDPE20-2%C-0.25% EX

4. Results & Discussions

4.1 Tensile Test Results

The tensile test results are illustrated in Table 3. The average maximum strength measured for rPET is 21.78N/mm^2. The measured strain percent is 2%, which indicates that the sample (S1) has very low ductility and high brittleness, and a significant rate of degradation occurred. These measurements are shown in Figures 1 and 2. The blend sample rPET80/HDPE20 without additives (S3) reached a maximum stress of 30.41 N/mm^2, a strain percent of 11.09, and an E modulus of 260.07 N/mm^2. The rPET 80/HDPE20-0.25%EX-2%C blend sample exhibited the greatest tensile strength of 34.17 N/mm^2 among all samples, and a strain percent of 11.48 is shown in Figure 4. When the amount of compatibilizer in the sample (A4) was increased to 5 wt. percent, the strain percent reached 14.68, but the tensile strength decreased to 26.5 N/mm^2. Figure 5 shows a chart that demonstrates the effect of the blend ratio of rPET/HDPE without additives on the mechanical properties. The maximum strain percent value was obtained at a blend ratio of 40/60. Figure 6 shows stress and strain curve comparisons between three different samples with zero, 2 wt, and 5 wt of rPET 40/HDPE 60 blends. The sample 40/60 with the addition of 2% compatibilizer achieved the highest value for this trend, with a value of 27.62 N/mm^2, while the max E modulus reached 227.44 N/mm^2 with the addition of 5% compatibilizer. Figure 7 shows the results of the tensile tests. It depicts the addition of 2 and 5% wt compatibilizer to rPET 80/HDPE 20. The maximum tensile strength reached 34.17 N/mm^2 and 310.57 for the modulus with the addition of 2 wt percent. The resultant values were the highest among all samples, while the addition of 5 wt percent of compatibilizer for the same blend ratio decreased the max stress to reach 26.5 N/mm^2, but the strain percent increased to reach 14.68. These results mean that 2 wt percent is adequate to obtain the max stress at rPET80/HDPE20, and 5% can help to increase the ductility when it is needed according to the application. The comparison between the result samples A1, A1^{-}, A2, A2^{-}, A3, A3^{-}, A4, A4^{-} led to the overall conclusion that applying one step processing can achieve better mechanical properties within 8% to 17%, which is illustrated in Table 3.

Table 3. Stress and Strain Results of Different Blends

Sample	Material	Max Stress N/MM^2	Strain%	E modulus N/MM^2
S1	rPET	23.05	2	--
S2	HDPE	21.78	120	235.89
S3	rPET80/HDPE20	30.41	11.09	260.07
S4	rPET60/HDPE40	23.63	13.89	218.25
S5	rPET40/HDPE60	21.36	66.56	245.76
S6	rPET20/HDPE80	21.65	127.57	257.98
A1	rPET80/HDPE20-0.25% EX -2%C	34.17	11.48	310.57
A1^{-}	rPET80/HDPE20-0.25% EX -2%C	31.58	10.31	280.17
A2	rPET80/HDPE20-0.25% EX -5%C	26.5	14.68	226.30
A2^{-}	rPET80/HDPE20-0.25% EX -5%C	26.01	12.29	227.44
A3	rPET60/HDPE40-0.25% EX -2%C	27.62	10.79	224.16
A3^{-}	rPET60/HDPE40-0.25% EX -2%C	23.22	10.97	242.29
A4	rPET60/HDPE40-0.25% EX -5%C	25.02	12.14	227.44
A4^{-}	rPET60/HDPE40-0.25% EX -5%C	23.63	13.89	218.25

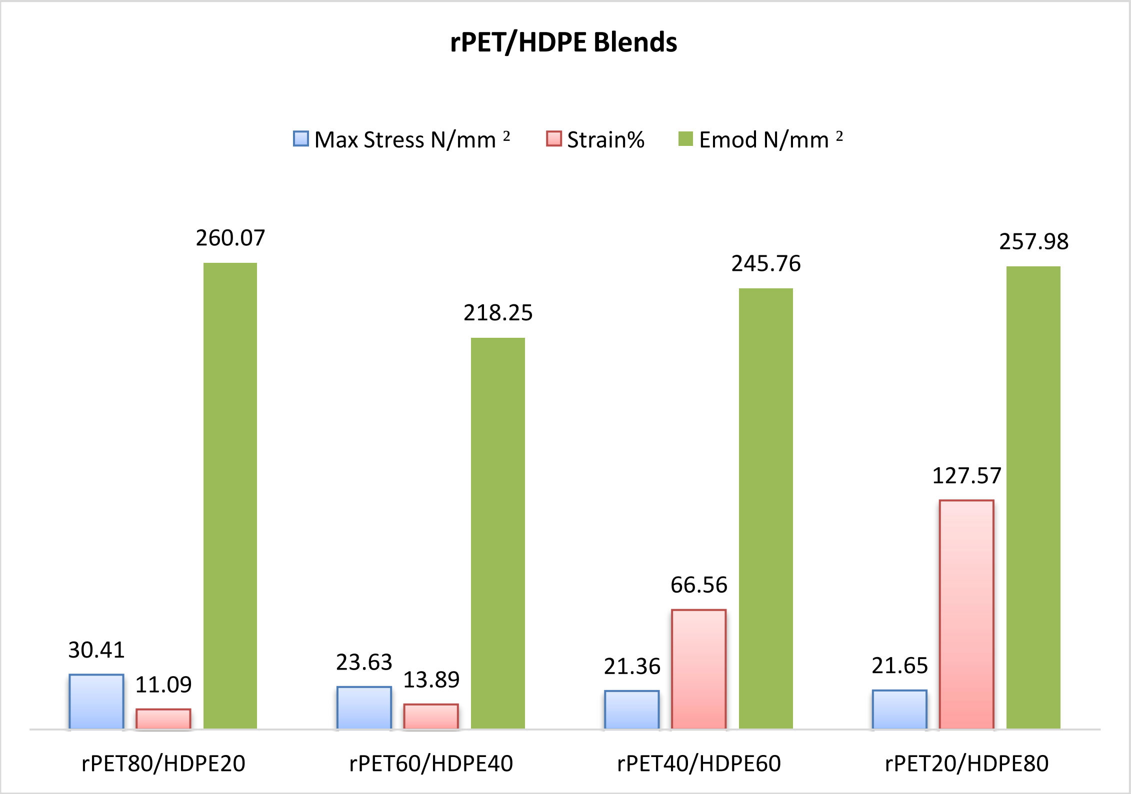


Figure 5. Effect of rPET/HDPE Blend Concentrations on Mechanical Properties

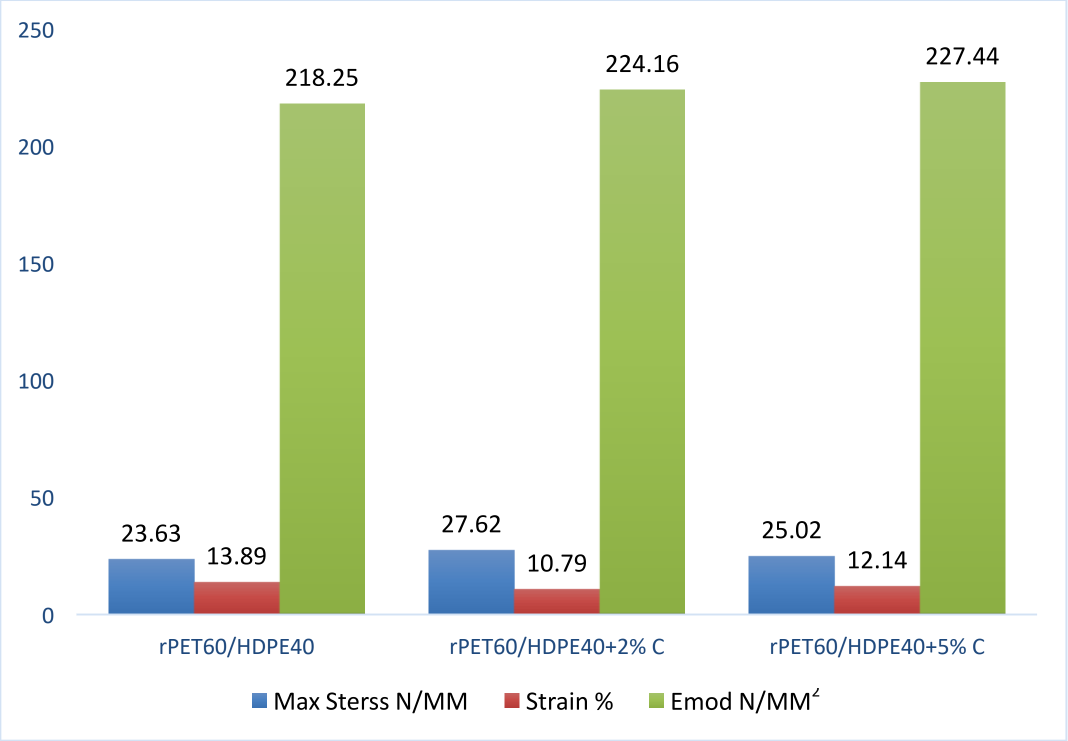


Figure 6. The Effect of Addition 2 and 5% HDPE-g-MA Compatibilizer on Mechanical Properties of rPET60/HDPE40

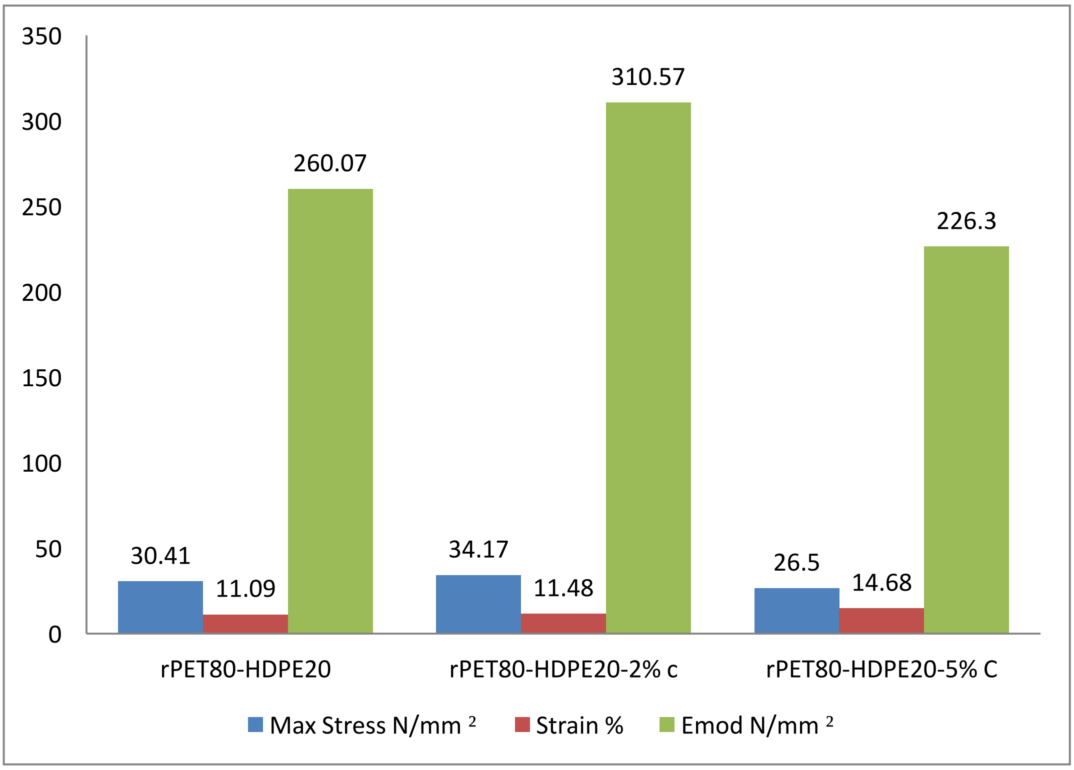


Figure 7. The Effect of Addition 2 and 5% HDPE-g-MA Compatibilizer on Mechanical Properties of rPET60/HDPE40

4.2 Morphology Results

The Scanning Electro-Microscope (SEM) micrographs of the rPET80/HDPE20 blend without compatibilizer, show that this blend has irregular shapes and dimensions, are shown in Figure 9.

This immiscible blend led to microvoids at the interfaces between the continuous and dispersed phases, resulting in phase separation. The SEM micrograph demonstrates the significant degree of incompatibility. The dispersed particles are enormous, ranging in diameter from a few microns to around ten microns, and therefore do not bond to the matrix. As a result, the two phases of a rPET/HDPE blend adhered poorly (Techawinyutham et al., 2021).

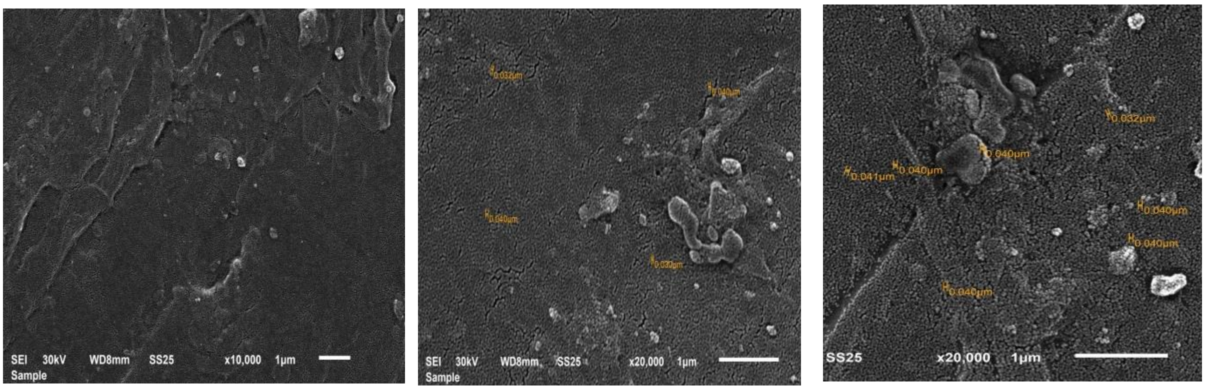
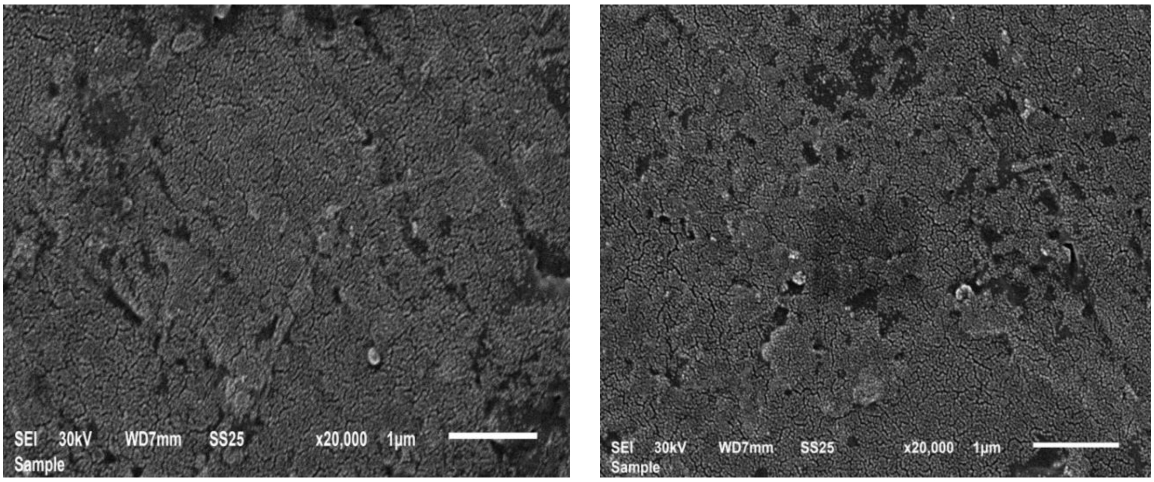


Figure 9. SEM micrographs of Incompatibilized rPET80/HDPE20 Blends



(a) 2% Compatibilizer (b) 5% Compatibilizer

Figure 10. SEM micrographs of Compatibilized rPET80/HDPE20 Blends

On the other hand, by adding 2% of the compatibilizer (HDPE-g-MA) to rPET80/HDPE20 blend demonstrated a more uniform distribution of particles with a diameter of only a few microns and the interfacial adhesion was increased. Figure 10 (a) shows a high degree of compatibility between the two phases. The particle dimension of dispersed rPET domains was further reduced in the blend as the compatibilizer ratio was increased to 5% as shown in Figure 10 (b). The concentration of the compatibilizer had an effect on the dispersion of the blend. Incompatible blends have larger particle sizes and lower interfacial adhesion. Higher dispersion phases have smaller particle sizes and a higher adhesion between the interfaces (Lusinchi et al., 2000). The produced blends had better interfacial adhesion to the rPET matrix phase. Because of the greater function reactivity of the compatibilizer, the interfacial tension and agglomerate suppression are reduced, and the molecular structure of the compatibilized blend is smaller than that of the incompatibilized blend (Dimitrovaa et al., 2000).

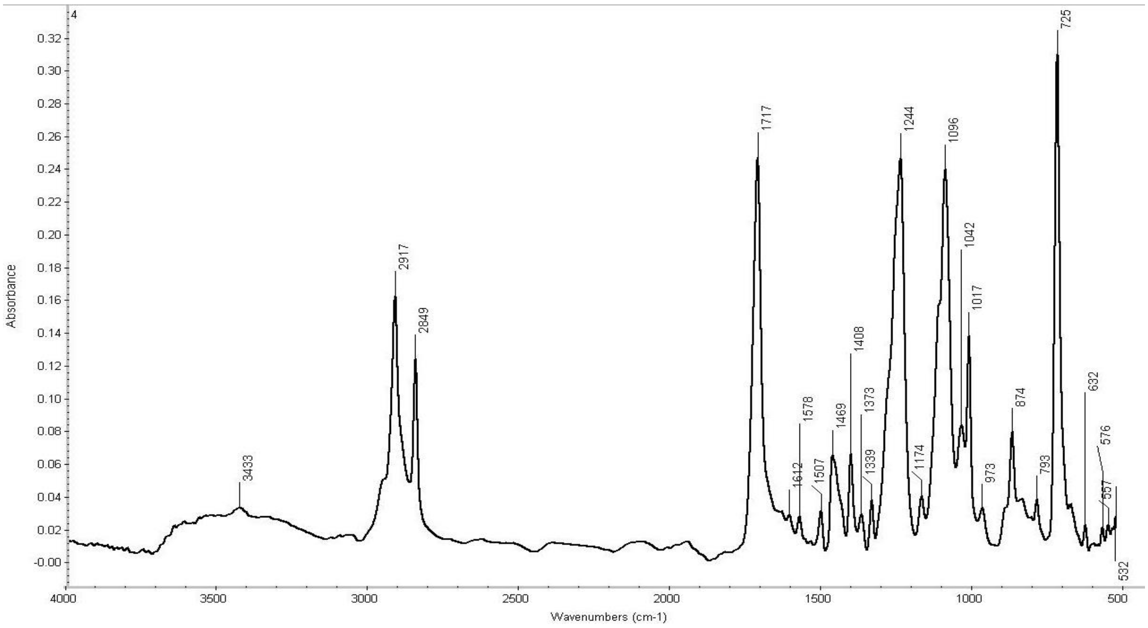


Figure 8. Spectroscopy Results of Compatibilized rPET80/PET20-2%C +0.025%EX

4.3 Infrared Spectroscopy Results

The grafting reaction on molten HDPE was carried out in a closed system within the injection moulding process by adding 2 and 5 wt percent of HDPE-g-MA compatibilizer. The melt blend was prepared in a single step by injecting the polymers in the presence of additives to generate (ISO 527 type 1A) samples. The closed system was used to avoid MA volatilization into the atmosphere. Figure 8 demonstrates the absorption of MA and two peaks located at 725 and 1469 cm^{-1}, which correspond to polyethylene's characteristic absorption bands, confirming that grafting of MA onto HDPE in the molten state occurred (Techawinyutham et al., 2021).

4.4 DSC Results

The DSC second heating and cooling curves of incompatible and compatible rPET/HDPE blends are shown in Figures 11 and 12. Tables 4 and 5 illustrate the transition temperatures and crystallinity changes due to the effect of comptatibilization and chain extension on rPET and HDPE. The melting and recrystallization temperatures of the compatibilized blend increased from Tm (128.42 to 131.45 ℃) and from Tc (113.74 to 119.37 ℃) for HDPE. For rPET Tm was slightly increased from (248.97 to 249.05 ℃) and Tc was increased from (113.74 to 119.37 ℃). Furthermore, the enthalpy for rPET (ΔHm) was decreased from 28.33 to 26.01 J/gk, while the HDPE ΔHm was increased from 12.29 to 52.54 J/g. There are two recrystallization peaks in the incompatibilized blend for HDPE, which are due to the rearrangement process of the crystalline fraction, which is responsible for the presence of these two peaks. Although imperfect small crystals formed a constant crystallization temperature in the first endothermal temperatures, the second endothermal peak at different curves represents the fusion of more perfect crystals in the second endothermal temperatures (Abdelbary & Seileem, 2021).The crystallinity degree of the incompatibilized rPET phase was found to be 5.5%, and it decreased in the compatibilized rPET phase to be 4%. For the HDPE phase, the degree of crystallinity had a slight decrease from 4% to 3% in the compatibilized phase.

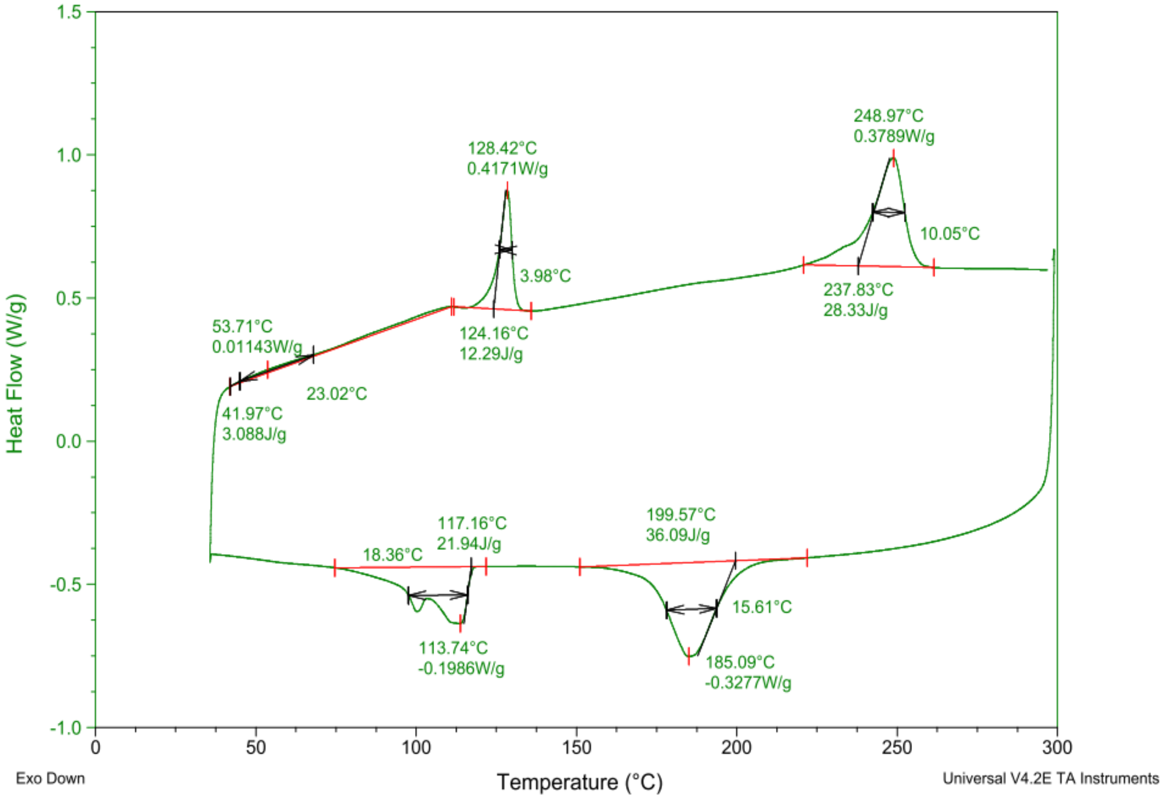


Figure 11. DSC Curve of Incompatibilized rPET80-HDPE20

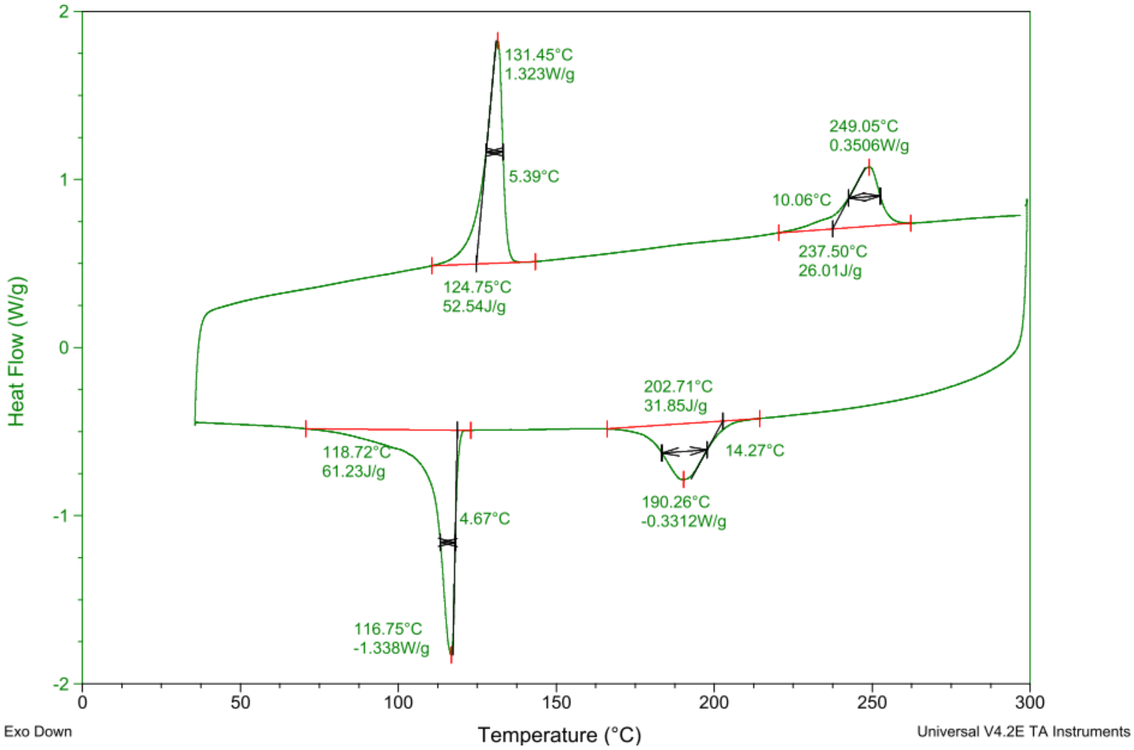


Figure 12. DSC Curve of Compatibilized rPET80-HDPE20

Table 4. DSC Results for rPET80-HDPE20 Blend

Material Crystallinity%	Tm °C	Tc °C	ΔHc J/gk	ΔHm J/gk
HDPE 4%	128.42	113.74	21.94	12.29
R PETR 5.5%	248.97	185.09	36.09	28

Table 5. DSC Results for Compatibilized rPET80-HDPE20 Blend

Material Crystallinity%	Tm °C	Tc °C	ΔHc J/gk	ΔHm J/gk
HDPE 3%	131.45	119.37	61.23	52.54
r- PETR 4%	249.05	190.26	31.85	26.01

4.5 Rheology Results

Figure 13 illustrates that the first run of the recycled PET material at shear rates ranging from 0.05 to 20 (1/S) produced a viscosity range of 50 to 60 (Pas), while the second run at the same shear rate produced a viscosity range of 32 to 48 (Pas). The third run with the same sample under the same running conditions achieved a viscosity range between 28 and 39 (Pas). This experiment demonstrates the effect of re-melting on the viscosity range and, consequently, on the molecular weight. These results demonstrates the benefit of recycling materials by being processed in a single step to create a high-quality material.

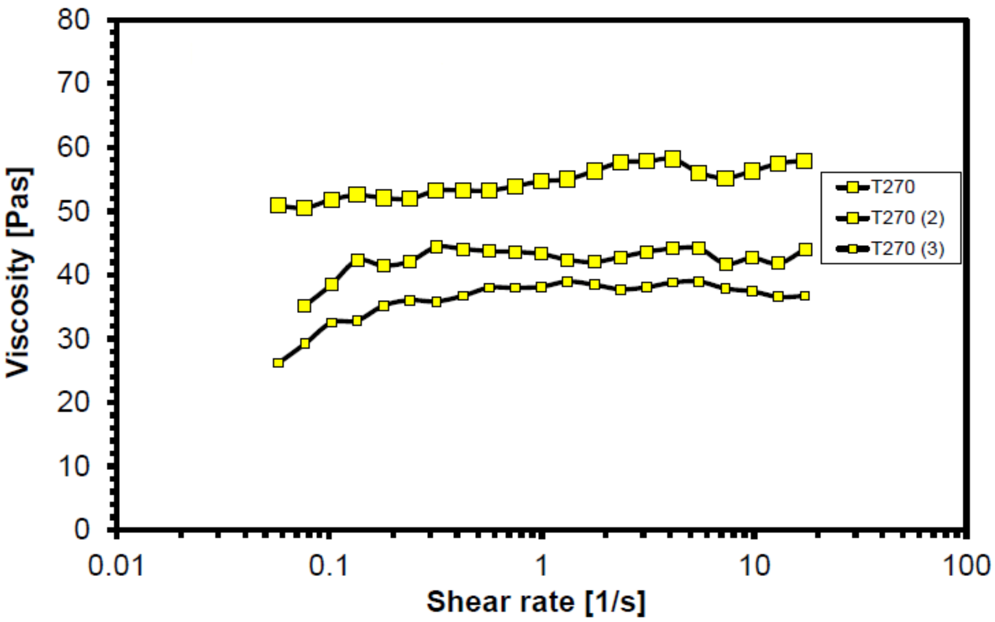


Figure 13. Simulating the Effect of Thermal History and Reprocessing on rPET Material and Its Effect on Viscosity and Molecular Weight

5. The New Recycling Process

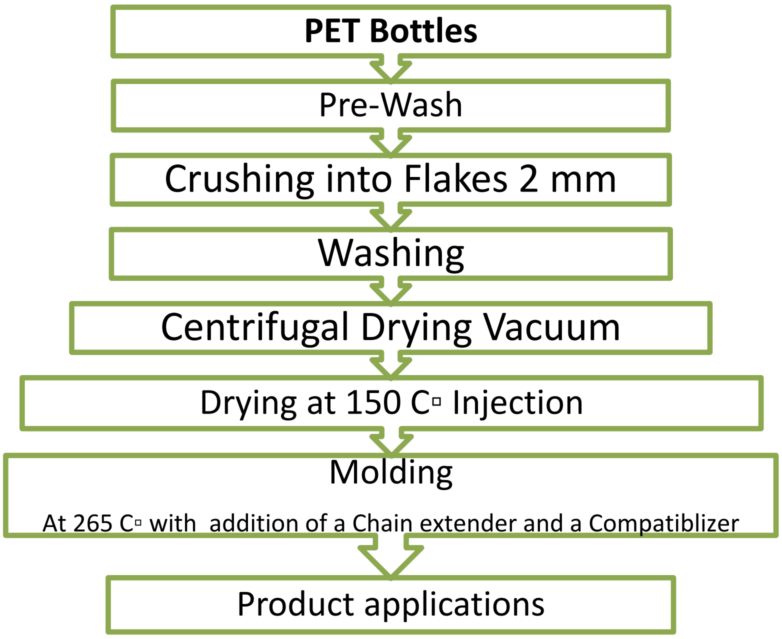


Figure 14. A Flow Chart of the New Recycling Process

The new recycling procedure includes washing PET bottles with hot air and steam to remove contaminants such as PVC labels, tape adhesive substances, and any organic material. The waste PET bottles are then shred without sorting into equal-sized flakes of 2 mm, followed by centrifugal drying and vacuum drying at 150 C▫ for 3 hours (Abdelbary & Seileem, 2022). The generated flakes are directly used in injection molding in an isolated interface, and adding reactive additives includes chain extender and a compatibilizer. The complete work flow of the new recycling process is described in Figure 14. The reactive chemicals interact to create homogeneity between rPET and HDPE, while the chain extender will chemically react with PET molecules to restore the viscosity and molecular weight of the rPET chains to its virgin status. In a single step, high-quality injected products can be obtained from post-consumer PET bottle waste. This new approach can help recycling stations reduce capital costs by eliminating the high cost of sorting, extrusion, and pelletizing units, thus lowering the price of recycled material. The newly created material will be sold in the form of flakes without being treated at recycling stations. It is the role of plastic manufacturers to incorporate additives into the injection molding process. This method can be applied to rPET waste material by adding the chain extender. The sorting process will be included, but the extrusion and pelletizing steps should be skipped.

6. Conclusions

The proposed approach can help to avoid the disadvantages of mechanical recycling due to the effect of reprocessing in different steps, and the same results as chemical recycling in the restoration of the virgin status of waste materials can be achieved at a lower cost with minimal energy consumption. The incorporation of the chain extender and compatibilizer during the melt processing in situ injection molding resulted in recycled rPET/HDPE bottles having nearly the same molecular weight and viscosity as virgin materials, and can also be applied to PET waste after sorting to restore viscosity and molecular weight to virgin status. This method is extremely cost-effective because it cuts costs on materials, reduces the initial investment, and uses low energy. Mechanical and morphological properties obtained in a single thermal processing step outperform those obtained in two thermal processing steps. Moreover, the mechanical and morphological characteristics are greatly improved.

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