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Restoration and investigation of the structural and performance properties of waste polyethylene terephthalate through recycling and modification

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Восстановление и исследование структурных и эксплуатационных свойств отходов полиэтилентерефталата путём переработки и модификации

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Abstract

This study evaluates restoration of the structural and performance properties of post-consumer polyethylene terephthalate (PET) through mechanical recycling and reactive modification with pyromellitic dianhydride (PMDA). PET beverage-container waste was sorted, washed, shredded, dried, re-extruded, and granulated. Recycled PET formulations containing 0.25–1.00 wt.% PMDA were prepared and compared with unmodified regranulated PET. Pellet yield, moisture content, density, and melt flow rate (MFR) were assessed as processing and material indicators. The MFR of unmodified regranulated PET was 24.6 g/10 min and decreased progressively with increasing PMDA content, reaching 9.8 g/10 min at 1.00 wt.% PMDA. Pellet yield reached a maximum of 95.1 % at 0.50 wt.% PMDA and decreased to 93.9 % at 1.00 wt.%. Moisture content ranged from 0.016 to 0.018 %, while density increased from 1.342 to 1.353 g/cm³. Considering MFR, pellet yield, and visual appearance together, the formulation containing 0.50 wt.% PMDA showed the most balanced processing performance; the 0.75 wt.% formulation may be considered when lower melt flow is required. The results provide a basis for further molecular, thermal, and mechanical characterization to verify chain extension and determine the application potential of the modified recycled PET.

Аннотация

В статье исследуется восстановление структурных и эксплуатационных свойств полиэтилентерефталата (ПЭТ), полученного из потребительских отходов, посредством механической переработки и реакционной модификации пиромеллитовым диангидридом (PMDA). Отходы ПЭТ-бутылок сортировали, промывали, измельчали, сушили, повторно экструдировали и гранулировали. Образцы вторичного ПЭТ с содержанием PMDA 0,25–1,00 мас.% сравнивали с немодифицированным регранулятом. В качестве основных технологических и материаловедческих показателей оценивали выход гранул, влажность, плотность и показатель текучести расплава (ПТР). Для немодифицированного регранулята ПТР составил 24,6 г/10 мин и последовательно снижался с увеличением содержания PMDA до 9,8 г/10 мин при 1,00 мас.% модификатора. Наибольший выход гранул — 95,1 % — получен при содержании PMDA 0,50 мас.%; при 1,00 мас.% он снизился до 93,9 %. Влажность образцов составляла 0,016–0,018 %, плотность увеличивалась с 1,342 до 1,353 г/см³. Совместная оценка ПТР, выхода и внешнего вида гранул показывает, что состав с 0,50 мас.% PMDA обеспечил наиболее сбалансированные технологические показатели. Результаты обосновывают дальнейшее молекулярное, термическое и механическое исследование модифицированного вторичного ПЭТ для подтверждения удлинения цепей и определения областей его применения.

Keywords: waste polyethylene terephthalate, recycled PET, mechanical recycling, reactive extrusion, pyromellitic dianhydride, melt flow rate.

Ключевые слова: отходы полиэтилентерефталата, вторичный ПЭТ, механическая переработка, реакционная экструзия, пиромеллитовый диангидрид, показатель текучести расплава.

Introduction

Currently, the rapid growth in the production and consumption of polymeric materials has made the effective management of plastic waste, its recycling as a secondary raw material, and its reintegration into resource circulation important environmental and scientific-technological challenges. Globally, a substantial proportion of plastic waste is accumulated in landfills or released into the environment, while recycling rates remain relatively low. This situation reinforces the need to transition from the conventional linear “production–consumption–waste” model toward the principles of a circular economy [1, 2]. This issue is also highly relevant to Uzbekistan. According to official data, 14.8 million tonnes of municipal solid waste have been generated in the country, of which only 6.1 % has been recycled [3]. Recent data indicate that plastic waste accounts for approximately 15 % of municipal solid waste, with an average of 1.8 million tonnes of plastic waste generated annually, of which only about 6.6 % is recycled [4]. Therefore, the advanced recycling of polymer waste, including polyethylene terephthalate (PET) waste, the production of high-quality secondary raw materials, and the expansion of their reuse opportunities are of particular importance for ensuring environmental sustainability and resource efficiency in Uzbekistan.

Polyethylene terephthalate is a thermoplastic polyester characterized by high mechanical strength, chemical resistance, transparency, and favorable processing properties. It is widely used in the production of beverage containers, packaging materials, films, synthetic fibers, and various technical products [5]. Since a substantial proportion of PET products becomes waste after use, the development of efficient methods for recycling this polymer is essential. Mechanical recycling of PET waste, involving sorting, washing, shredding, drying, extrusion, and granulation, is one of the practical approaches that enables the polymer to be reused as a secondary raw material [5, 6]. However, during repeated thermomechanical processing of PET, thermal, hydrolytic, and thermo-oxidative degradation of the macromolecular chains occurs under the influence of elevated temperature, residual moisture, oxygen, and mechanical shear forces. Consequently, a decrease in molecular weight and intrinsic viscosity (IV), as well as changes in melt rheological behavior, crystallization, thermal stability, and physicomechanical properties, can occur [6, 7]. Therefore, regranulation of waste PET alone is insufficient to fully restore the original performance characteristics of the material.

One promising approach to overcoming this problem is the targeted modification of recycled PET (rPET). In particular, the use of chain-extending compounds during reactive extrusion can promote reactions between the functional end groups of PET macromolecules, thereby increasing chain length and molecular weight and improving melt viscosity and mechanical properties [8, 9]. In this process, the type and concentration of the modifier, the moisture content of PET, extrusion temperature, and other processing parameters are critical factors determining the final structure and properties of the material.

From this perspective, the restoration and investigation of the structural and performance properties of waste polyethylene terephthalate through recycling and modification constitute an important scientific and practical challenge. The present study involves a comparative investigation of virgin PET, waste PET, regranulated rPET, and modified rPET samples, determination of the degradation mechanisms occurring during recycling, and restoration of the properties lost during processing through targeted modification. In this context, a comprehensive evaluation of the

structural, rheological, thermal, and physicomechanical characteristics of PET is essential for establishing the “processing conditions–structure–property” relationship and developing the scientific and technological basis for producing regenerated PET granules with improved properties.

Materials and Methods

Colorless post-consumer polyethylene terephthalate (PET) waste originating from beverage packaging was selected as the primary material for the study. Unprocessed virgin PET (vPET) granules were used as a comparative reference material. Waste PET containers were separated from polypropylene/polyethylene caps, labels, adhesive residues, and other foreign components and subsequently shredded into flakes measuring approximately 8–12 mm. The samples were cleaned in a warm washing solution and rinsed with distilled water until a neutral pH was achieved. The washed PET was initially dried at 120 °C for 2 h and subsequently dried in a vacuum dryer at 150 °C for 4 h. Prior to extrusion, the moisture content was controlled to achieve, as far as practicable, a level below 0.02 wt.% (200 ppm).

Seven sample groups were prepared for the experiments: V-PET, virgin PET used as the reference material; W-PET, washed and dried waste PET; R-PET, regranulated PET produced without a modifier; and P025, P050, P075, and P100, recycled PET modified with 0.25, 0.50, 0.75, and 1.00 wt.% pyromellitic dianhydride (PMDA), respectively.

PET regranulation was carried out using a laboratory-scale twin-screw extruder. The initial temperature profile of the extruder zones was set at 240–250–260–265 °C, while the die temperature was maintained at 260–265 °C. Screw rotation speeds within the range of 80–120 rpm were investigated. Since prolonged exposure of the material to elevated temperatures may cause additional thermal degradation, the residence time in the extruder was maintained, as far as possible, within 2–4 min. The R-PET sample was processed under the same conditions without the addition of any reactive modifier and was used as the control material for comparison with the subsequently modified samples.

To prepare the P025–P100 samples, dried R-PET granules were dry-blended with 0.25, 0.50, 0.75, and 1.00 wt.% PMDA, respectively, followed by reactive extrusion. The process was carried out directly in the PET melt without a separate solvent medium. Where technically feasible, the extruder feed section and processing system were protected with an inert nitrogen (N_2) atmosphere to minimize oxidative degradation. The extrudate was cooled, pelletized into granules approximately 3–4 mm in size, and redried before analysis.

The molecular characteristics of PET were evaluated by determining its intrinsic viscosity (IV) using solution viscometry. An approved solvent system suitable for PET and compliant with the laboratory safety requirements and the applicable standard method was employed. The same solvent system, polymer concentration, and measurement temperature were maintained for all V-PET, W-PET, R-PET, and M-PET samples to ensure comparability of the results. The melt flow rate (MFR) was determined under selected standard conditions appropriate for PET, and the effects of polymer degradation and chain extension on the melt-flow behavior were evaluated.

Results and Discussion

At the initial stage of the study, particular attention was focused on recovering waste polyethylene terephthalate as a secondary raw material, producing regranulated PET, and evaluating the effect of a chain-extending modifier on the properties of recycled PET. Colorless PET containers were selected as the waste feedstock and separated from caps, labels, and other foreign contaminants. The PET waste was washed, shredded into flakes measuring approximately 8–12 mm, and thoroughly dried to minimize the adverse effect of residual moisture during subsequent processing.

For the experimental study, the samples were designated as follows: V-PET – virgin PET; W-PET – washed and dried waste PET; R-PET – regranulated PET produced without a modifier; and P025, P050, P075, and P100 – rPET samples modified with 0.25, 0.50, 0.75, and 1.00 wt.% pyromellitic dianhydride (PMDA), respectively.

Regranulation of Waste PET. Initially, waste PET was regranulated by extrusion without the addition of a modifier. This experiment was conducted to obtain the R-PET control sample and to evaluate the processability of waste PET under the selected extrusion conditions. The main temperature zones of the extruder were maintained within the range of 240–250–260–265 °C, the die temperature was set at 260–265 °C, and the screw rotation speed was varied within the range of 80–120 rpm. Stable melting of the dried PET in the extruder and the formation of a continuous and uniform extrudate were considered the principal technological criteria for assessing the stability of the regranulation process.

The resulting extrudate was cooled and pelletized into granules approximately 3–4 mm in size. As a result, the R-PET base material was obtained and subsequently used for the modification experiments.

Effect of Chain Extender Content

Pyromellitic dianhydride (PMDA) was selected as the chain extender to compensate for the reduction in PET macromolecular chain length caused by degradation during recycling. To determine the optimal modifier content, the PMDA concentration was varied from 0.25 to 1.00 wt. %. The compositions and sample designations used in the experimental study are presented in Table 1.

Table 1. Composition and designation of rPET samples containing different amounts of PMDA

Sample	rPET, wt.%	PMDA, wt.%	Sample Type
R-PET	100,00	0	Control
P025	99,75	0,25	Modified
P050	99,50	0,50	Modified
P075	99,25	0,75	Modified
P100	99,00	1,00	Modified

As far as practicable, identical drying and extrusion conditions were maintained for all samples. This approach made it possible to attribute the observed differences primarily to the PMDA concentration rather than to variations in processing temperature or residual moisture content.

At this stage of the study, prior to conducting advanced instrumental and structural analyses, it was considered appropriate to determine the primary characteristics of the produced granules, including pellet yield, visual appearance, color, pellet size, density, moisture content, and melt flow rate (MFR/MFI).

Table 2. Pellet yield, moisture content, density, melt flow rate, and visual appearance of PMDA-modified rPET samples

Sample	Pellet Yield, %	Moisture Content, %	Density, g/cm³	MFR, g/10 min
R-PET	94,2	0,018	1,342	24,6
P025	94,5	0,017	1,345	20,8
P050	95,1	0,016	1,349	16,3
P075	94,8	0,016	1,351	12,7
P100	93,9	0,017	1,353	9,8

The obtained results demonstrated that increasing the PMDA content in rPET had different effects on the granulation process, moisture content, density, melt flow rate, and visual appearance of the pellets. In particular, the systematic decrease in MFR can be regarded as one of the key findings indicating a significant effect of the chain-extending modifier on the rheological behavior of the rPET melt.

For the unmodified R-PET sample, the pellet yield was 94.2 %, whereas the incorporation of 0.25 wt.% PMDA increased the yield to 94.5 % for P025. A further increase in PMDA content to 0.50 wt.% resulted in a pellet yield of 95.1 % for P050. Compared with R-PET, the 0.9 percentage-point increase in pellet yield observed for P050 indicates that extrudate formation and granulation proceeded relatively steadily in the presence of a moderate amount of the modifier.

When the PMDA content was increased to 0.75 wt.%, the pellet yield was 94.8 %, while at 1.00 wt.% PMDA it decreased to 93.9 %. The decrease observed for P100 may be associated with substantial changes in the flow behavior of the polymer melt. At a high modifier concentration, increased chain extension and branching may increase melt viscosity, thereby hindering material flow through the extruder die. Therefore, from the standpoint of product yield alone, the P050–P075 composition range appeared to be more technologically favorable.

The moisture content of the samples ranged from 0.016 to 0.018 %. The moisture content was 0.018 % for R-PET, 0.017 % for P025, and decreased to 0.016 % for both P050 and P075. For P100, a value of 0.017 % was recorded. Overall, the differences among the samples were small, indicating that the initial moisture levels of the modified and unmodified samples were comparable. This parameter is particularly important because PET is susceptible to hydrolysis, and elevated moisture content may promote hydrolytic chain scission of the macromolecules during high-temperature extrusion. Therefore, the low and comparable moisture levels among the samples allow the effect of the modifier to be evaluated with relatively limited interference from variations in moisture content.

A small but systematic increase in density was also observed. The density of R-PET was 1.342 g/cm³, whereas the corresponding values for P025, P050, P075, and P100 were 1.345, 1.349, 1.351, and 1.353 g/cm³, respectively. The overall increase in density for P100 relative to R-PET was approximately 0.82 %. However, this change cannot be attributed solely to chain extension. The

density of PET is also influenced by the degree of crystallinity, thermal history, cooling rate, and internal morphology of the pellets. Therefore, until this trend is confirmed by additional structural or thermal analyses, the observed density variation should be interpreted as a supplementary indicator rather than direct evidence of chain extension.

The most pronounced changes were observed in the MFR values. The unmodified R-PET sample exhibited an MFR of 24.6 g/10 min. Upon the addition of 0.25 wt.% PMDA, the MFR decreased to 20.8 g/10 min. Increasing the PMDA concentration to 0.50 wt.% further reduced the MFR to 16.3 g/10 min, while values of 12.7 and 9.8 g/10 min were obtained at PMDA concentrations of 0.75 and 1.00 wt.%, respectively.

Compared with R-PET, the MFR decreased by approximately 15.4 % for P025, 33.7 % for P050, 48.4 % for P075, and 60.2 % for P100. These results demonstrate a clear inverse relationship between PMDA content and the melt flowability of rPET.

Variation in MFR with PMDA content: R-PET – 24.6 g/10 min; P025 – 20.8 g/10 min; P050 – 16.3 g/10 min; P075 – 12.7 g/10 min; P100 – 9.8 g/10 min.

The decrease in MFR indicates an increase in melt viscosity. In the PET system, such behavior may be attributed to reactions between PMDA and the terminal functional groups of PET macromolecules, resulting in chain extension and potentially the formation of branched macromolecular structures.

The dependence of MFR on PMDA concentration revealed a particularly interesting technological trend for the P050–P075 samples. For P050, although the MFR decreased by approximately one-third relative to R-PET, the pellet yield remained the highest among the investigated samples, at 95.1 %. For P075, the MFR decreased further to 12.7 g/10 min, while the pellet yield remained relatively high at 94.8 %. Thus, within this concentration range, the increase in melt viscosity did not result in a substantial deterioration of the granulation process.

A different behavior was observed for the P100 sample. As the MFR decreased to 9.8 g/10 min, the pellet yield simultaneously declined to 93.9 %, accompanied by more pronounced yellowing of the material and increased difficulty in melt flow. These observations suggest that modification with 1.00 wt.% PMDA may be excessive from the standpoint of technological processability. Excessively high melt viscosity may increase pressure within the extruder, destabilize melt flow, and reduce granulation efficiency. The increased yellow coloration may be associated with thermal or thermo-oxidative processes occurring during high-temperature processing; however, its exact origin cannot be conclusively established without additional analysis.

The visual appearance of the pellets also changed depending on the modifier concentration. R-PET was slightly yellowish and transparent, whereas the P025 and, particularly, P050 samples exhibited more homogeneous and smooth pellets. A slight yellowish coloration was observed for P075, while the yellowing became more pronounced for P100. In this respect, P050 exhibited the most favorable combination of visual appearance and granulation stability.

A comprehensive evaluation of the results indicates that the P050 and P075 formulations are the most promising among the investigated compositions. For P050, the pellet yield reached a maximum of 95.1 %, while the MFR decreased from 24.6 to 16.3 g/10 min, corresponding to a reduction of 33.7 %. P075 exhibited a further substantial decrease in melt flow rate; however, the onset of yellowish coloration was observed in the pellets. Although P100 exhibited the lowest MFR, it was also characterized by reduced granulation efficiency and deterioration in visual appearance.

Based on these results, the PMDA concentration range of 0.50–0.75 wt.% can be identified as the most promising range for further investigation. If the principal criterion is achieving an appropriate balance among granulation stability, visual appearance, and sufficiently high melt viscosity, the P050 formulation containing 0.50 wt.% PMDA may be considered the preliminary optimum composition. Nevertheless, determination of the final optimum formulation requires further evaluation of mechanical properties and, where necessary, molecular and thermal characteristics.

Conclusion

Overall, increasing the PMDA concentration from 0 to 1.00 wt.% resulted in a decrease in the MFR of rPET from 24.6 to 9.8 g/10 min, demonstrating the pronounced effect of the modifier on the melt rheological behavior of the polymer. At the same time, the deterioration in processability observed at a PMDA concentration of 1.00 wt.% indicates the existence of an optimum concentration range for the chain-extending modifier.

Based on the obtained results, the P050 formulation containing 0.50 wt.% PMDA can be considered the most technologically balanced composition, providing a favorable combination of melt rheological behavior, granulation stability, and pellet quality. In contrast, the P075 formulation containing 0.75 wt.% PMDA may be regarded as a promising composition for applications requiring higher melt viscosity.

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