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# DSC CHARACTERIZATION OF POLYETHYLENE COMPOSITES WITH RECYCLED PET-BASED POLAR WAX

**G.A. Bozorboeva[1], M.G. Alimukhamedov[2], A.B. Jurayev[3], N.Z. Dusiyorov[4], A. Norqobilov[5]**
Department of High-Molecular Compounds and Plastics, Tashkent Institute of Chemical Technology[1,2,3]
Nanostructured Composite Polymer Materials Laboratory, Institute of Polymer Chemistry and Physics of the Academy of Sciences of the Rep.Uzb.[4,5]

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***Abstract**. In this study, hydroxyl-containing oligomers were synthesized from secondary PET (sPET) via alcoholysis with diethylene glycol (DEG) and further reacted with stearic acid ($C_{17}H_{35}COOH$) to produce a polar synthetic wax. This wax was used as a filler in polyethylene (PE) composites (PE grades I-1561 and F-0320). Differential scanning calorimetry (DSC) analysis showed that the wax modified the crystallinity and thermal behavior of the materials, particularly enhancing the thermal stability of I-1561-based composites. The results demonstrate the potential of the synthesized wax as a high-value additive derived from recycled materials. The data suggest that the synthesized polar wax is not merely a recycled byproduct but serves as a functional, value-added processing aid with broad industrial potential.*

***Keywords:** secondary PET (sPET), alcoholysis, diethylene glycol (DEG), polymer composites, polyethylene (PE), synthetic wax, differential scanning calorimetry (DSC).*

## INTRODUCTION

Producing composite materials with enhanced properties by blending various polymers is considered highly cost-effective method [1]. The main advantage lies in the fact that the resulting composite forms a phase-integrated mixture, where the components are inseparable on the macroscopic level. This feature allows for the creation of high-performance polymer blends. One of the most accumulated polymer wastes today is polyethylene terephthalate (PET), with an estimated 50–60 thousand tons of this plastic type imported annually into Uzbekistan. Efficient recycling of such volume of waste is among the urgent problems requiring a solution [2].

According to numerous scientific studies, the use of various waxes with high crystalline structure as phase-change materials (PCMs) is a promising approach [3]. One such wax is paraffin, which, either in pure form or blended with linear low-density polyethylene (LLDPE), crystallizes and melts below 80°C [4]. Based on isothermal crystallization results from Differential Scanning Calorimetry (DSC) analysis, molten paraffin wax acted as a solvent for LLDPE at those temperatures. Increasing the wax mass fraction in the blend led to a reduction in both melting and crystallization temperatures. This indicates that paraffin wax acts as an effective plasticizer, increasing LLDPE chain mobility and lowering the initial crystallization temperature. During synthesis, LLDPE fractions separate at the molecular level, as they crystallize during the cooling of the melt, forming phases composed of highly linear fractions with higher melting points. Simultaneously, the wax implants in the amorphous regions. As cooling proceeds (within 70–105°C), shorter wax chains start segregating into the amorphous parts of polyethylene [8]. At lower crystallization temperatures, wax co-crystallizes with broader LLDPE chains due to the similarity in chain length and lamellar thickness [5].

*Djokovic et al.* investigated the physical properties of a Fisher-Tropsch wax and low-density polyethylene (LDPE) blend. According to DSC analysis, the polymer and wax chains co-crystallized, which contributed to improved material properties. The thermally unstable wax chains were shielded by the thermally stable PE chains, enhancing the overall crystalline structure. However, when the wax concentration exceeded 20%, the melting temperature decreased, suggesting phase separation. This dual effect of wax on LDPE resulted in enhanced crystallinity at low concentrations, but reduced viscosity at high concentrations due to shorter wax chains in the amorphous phase [6].

*Esmaeilzade et al.* studied the blending of secondary polyethylene-derived wax with high-density polyethylene (HDPE). It was observed that up to 12.05% wax content, no phase separation occurred, and both components co-crystallized. At higher wax concentrations, phase separation appeared, and PE/wax crystals could no longer maintain a single-phase structure. Their findings suggest that adding up to 10% wax improves the mechanical and thermal properties of the resulting composites [7].

## MATERIALS AND METHODS

In our study, synthetic polar waxes of polyester type were synthesized using PET bottle waste as the primary raw material. In the first stage, the process involved mechanical crushing, washing, and drying of post-consumer PET, which was then reacted with diethylene glycol (DEG), a polyhydric alcohol. The sPET: DEG molar ratio varied from 1:0.1 to 1:0.4. To prevent thermal degradation of sPET, the synthesis temperature was kept below 220°C. The reaction was conducted under continuous stirring with zinc acetate ($Zn(Ac)_2$) used as a catalyst at 0.5% relative to sPET. The alcoholysis process lasted approximately 6–8 hours with intense stirring. The resulting products were analyzed using various physicochemical methods.

In the next step, the oligomers obtained from alcoholysis of sPET with DEG in different ratios were reacted with stearic acid ($C_{17}H_{35}COOH$) in a 1:1 molar ratio via polycondensation. As the DEG concentration decreased, the synthesized waxes became more viscous and exhibited poorer physical properties. A higher DEG content in the oligomer yielded waxes with improved physicochemical characteristics as per GOST standards.

For the production of polymer composites from the synthesized polar polyester wax and two grades of polyethylene: I-1561 *($M_w$ = 200,000–400,000 g/mol; $\rho$ = 0.961 g/mL)* and F-0320 *($M_w$ = 100,000–400,000 g/mol; $\rho$ = 0.9180 g/mL)*, manufactured at the Shurtan Gas Chemical Complex, the extrusion method was used. The wax was first ground into powder form. Since the PE was in granule form, 1–1.5% of motor oil (relative to the total mass) was added to improve compatibility during mixing. A fixed 8% mass fraction of wax was blended into the PE and fed into an extruder. The extrusion temperature ranged between 171.2°C and 194.8°C, depending on the melting points of the wax and PE grades, with a screw rotation frequency of 3.0 Hz. The output blend was collected in granule form.

## RESULTS AND DISCUSSIONS

Based on Differential Scanning Calorimetry (DSC) analysis, the thermal behavior of polyethylene grades I-1561 and F-0320 with the addition of synthesized wax was investigated. The pure PE I-1561 sample exhibited a melting peak around 125°C, which is characteristic for this high-density polyethylene. The incorporation of wax caused an upward shift in the melting peak, reaching a maximum at specific ratios. The stability of the crystalline lattice in the pure I-1561 was only slightly affected by the wax, indicating its limited disruption potential. The PE I-1561/wax composite is chosen as optimal specimen and its DSC thermogram is presented in Figure

1, while that of the PE F-0320/wax system is shown in Figure 2. The corresponding thermal characteristics of the remaining composite formulations are summarized in Table 1.

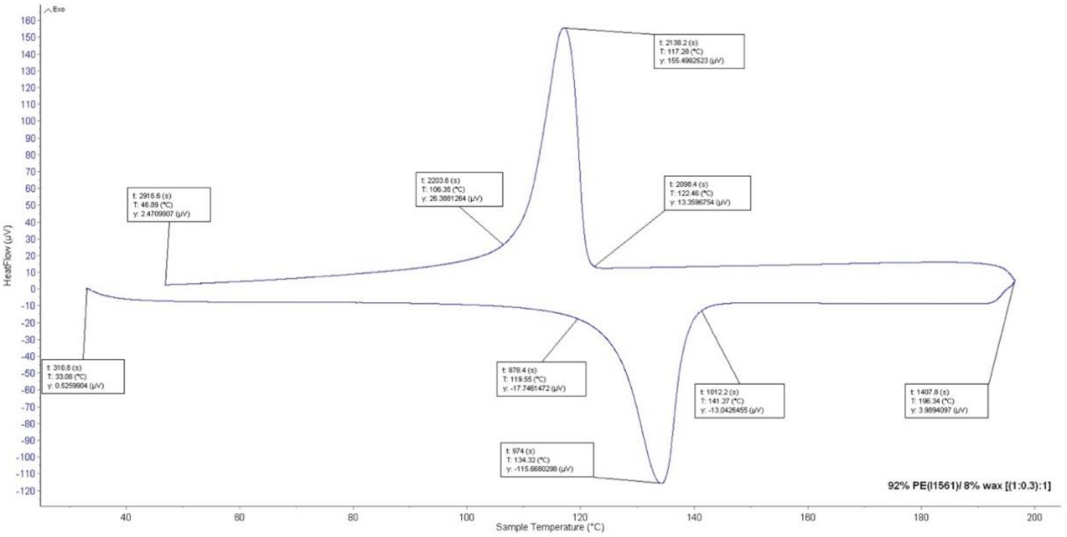

***Figure 1. DSC thermogram of [(1:0,3):1]$_{wax}$/I-PE based composite***

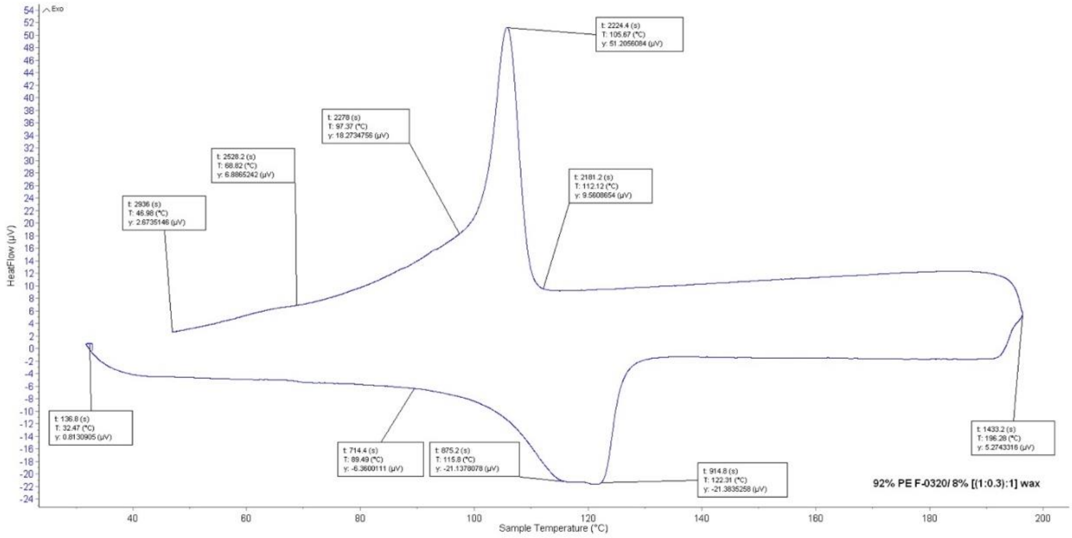

***Figure 2. DSC thermogram of [(1:0,3):1]$_{wax}$/F-PE -based composite***

For the PE F-0320 sample, which has lower average crystallinity and thermal resistance, melting initiated at 105°C and concluded at 125°C, with a peak melting temperature of approximately 114°C. Upon the addition of wax, all melting temperatures decreased, with the peak melting temperature in all composite samples falling within the range of ~105.5–106.2°C, and the melting end point shifting down to ~110.7–112.1°C. These changes reflect partial destabilization of the polyethylene crystalline lattice due to intermolecular interactions with the wax.

The increase in melting temperature observed in the PE I-1561 composition, which contains wax with (1:0.3):1 ratio indicates co-crystallization or stabilization of PE crystallites due to structural ordering. However, as the DEG content in the wax increased further (up to 1:0.4), partial lattice disruption occurred, lowering the melting temperature ($T_m$). Moreover, both the onset crystallization temperature and crystallization peak temperature of PE I-1561 composites increased with rising DEG concentration in the wax.

***Table 1.***

***DSC Analysis Results of Polyethylene Grades (F-0320 and I-1561) and their Composites Containing 92% PE / 8% Wax***

| Sample | Onset Melting Temp., °C | Peak Melting Temp., °C | End Melting Temp., °C | Crystallization Temp., °C | Crystallization Temp., °C | ΔH Melting Enthalpy, J/g |
|---|---|---|---|---|---|---|
| Pure PE (I-1561) | 112.0 | 125.0 | 140.0 | 114.0 | 112.0 | ~190 |
| (1:0.1):1 $_{wax}$/I-PE | 119.48 | 134.10 | 140.01 | 117.83 | 122.58 | ~150–170 |
| (1:0.2):1 $_{wax}$/I-PE | 115.50 | 138.88 | 146.70 | 117.00 | 122.32 | ~160–170 |
| (1:0.3):1 $_{wax}$/I-PE | 119.55 | 141.27 | 141.27 | 117.28 | 122.46 | ~150–160 |
| (1:0.4):1 $_{wax}$/I-PE | 119.50 | 134.10 | 140.00 | 119.48 | 122.58 | ~150–170 |
| Pure PE (F-0320) | 105.0 | 114.0 | 125.0 | 117.0 | 122.0 | ~80–90 |
| (1:0.1):1 $_{wax}$/F-PE | 97.32 | 105.44 | 110.82 | 117.10 | 122.29 | ~65–75 |
| (1:0.2):1 $_{wax}$/F-PE | 98.53 | 105.45 | 110.71 | 114.28 | 121.30 | ~60–70 |
| (1:0.3):1 $_{wax}$/F-PE | 97.37 | 105.67 | 112.12 | 115.80 | 122.31 | ~55–65 |
| (1:0.4):1 $_{wax}$/F-PE | 96.68 | 106.22 | 111.33 | 115.60 | 122.45 | ~55–60 |

In contrast, the polar polyester wax considerably reduced the crystalline phase fraction in PE F-0320 composites, affirming the amorphous nature of the wax. In these composites, the polyester fragments disrupted the crystalline lattice significantly, lowering the crystallization temperature ($T_c$) and decreasing crystallization intensity, which suggests inhibition of the crystallization process. The introduction of amorphous components hindered the regular packing of polyethylene macromolecules, reducing crystallinity. The crystallization onset in PE F-0320/wax composites ranged between ~114–117°C, closely aligning with values of pure PE. The crystallization peaks remained consistently near ~122°C across all samples. Despite reduced melting temperatures, the crystallization capability was preserved.

In the PE I-1561/wax composites, the wax acted as a nucleating agent, accelerating the onset of crystallization in the polymer matrix. The melting enthalpy (ΔH) in all composites decreased relative to pure I-1561 (~190 J/g), ranging from ~150–170 J/g depending on the ratio. This reduction in ΔH indicates a decrease in overall crystallinity due to partial replacement of the crystalline matrix by amorphous wax. For pure PE F-0320, ΔH was ~80–90 J/g, consistent with a material of high crystallinity. As wax content increased, ΔH declined steadily—from ~65–75 J/g at (1:0.1):1 to ~55–60 J/g at (1:0.4):1—indicating progressive loss of crystallinity. Low-temperature transitions (glass transition temperature, $T_g$) were observed in the range of 46–48°C, confirming the presence of amorphous wax and its weak dependence on composition changes.

## CONCLUSION

The addition of 8% wax to PE I-1561 resulted in increased melting temperature and stable crystallization behavior up to a critical DEG concentration (maximum at (1:0.3):1), demonstrating improved thermal stability. However, further increases in DEG content (up to 1:0.4) led to reduced $T_m$, indicating disruption of crystalline order. The overall crystallinity of the composites decreased relative to pure PE. In PE F-0320, the incorporation of 8% wax led to a decrease in melting temperature, reflecting disruption of macromolecular crystalline order. Despite the destabilizing effects of wax, stable crystallization parameters were maintained, indicating preserved crystallization capacity.

Based on DSC results, composite materials synthesized using waxes with low DEG molar content derived from sPET were found to offer optimal thermal stability and structural compatibility. These compositions demonstrated improved crystallization behavior due to elevated melting temperatures and favorable crystalline formation.

PE I-1561/wax composites are suitable for packaging, electrical insulation, and applications requiring controlled thermoplasticity and high thermal resistance. PE F-0320/wax composites are applicable in packaging, flow-modifying additives, or thermally stable coatings requiring modified structure and thermal endurance.

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