Reactive Modification of Recycled Poly (Ethylene Terephthalate) With Vinyl-Terminated PDMS: Development of Flexible Cushion Filling Fiber

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ABSTRACT

The increasing accumulation of post-consumer poly(ethylene terephthalate) (PET) waste has intensified the need for sustainable recycling strategies that produce value-added materials with enhanced performance. In this study, recycled PET (rPET) was reactively modified with 2–10 wt.% vinyl-terminated polydimethylsiloxane (PDMS) through peroxide-induced grafting in the presence of an epoxy-functional chain extender to fabricate flexible silicone-modified fibers for furniture cushion-filling applications. The effects of PDMS incorporation on the chemical structure, morphology, thermal behavior, mechanical performance, resilience, and surface properties of the fibers were systematically investigated using Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), tensile testing, compression recovery, cyclic resilience, water contact angle, moisture absorption, and flexibility measurements. FTIR confirmed successful incorporation of PDMS through the appearance of characteristic Si–O–Si and Si–CH_3 absorption bands while preserving the polyester backbone. SEM revealed smooth fiber surfaces and homogeneous cross-sections with stable fiber diameters, indicating improved melt processability and excellent compatibility between PDMS and recycled PET. Thermal analysis showed only minor changes in glass transition and melting temperatures, accompanied by moderate reductions in crystallinity and enhanced thermal stability resulting from the thermally stable siloxane structure. Mechanical characterization demonstrated significant improvements in elongation at break, compression recovery, cyclic resilience, softness, and hydrophobicity, while maintaining adequate tensile strength for cushioning applications. The water contact angle increased from 73.2° for unmodified rPET to 126.4° for the 10 wt.% PDMS-modified fibers, whereas moisture absorption decreased by more than 70%, indicating markedly improved water resistance. Among the investigated compositions, 6–8 wt.% PDMS provided the optimum balance between mechanical integrity, flexibility, resilience, thermal stability, and surface hydrophobicity. These findings demonstrate that reactive silicone modification is an effective strategy for upgrading post-consumer PET waste into high-performance, resilient fibers suitable for sustainable furniture cushion-filling applications and other value-added textile products.

Keywords: recycled poly(ethylene terephthalate); vinyl-terminated polydimethylsiloxane; reactive extrusion; silicone-modified fibers; cushion-filling materials

INTRODUCTION

Poly(ethylene terephthalate) (PET) is one of the most extensively produced thermoplastic polyesters owing to its excellent mechanical strength, dimensional stability, chemical resistance, transparency, and barrier properties. These characteristics have resulted in its widespread application in beverage bottles, food

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packaging, engineering plastics, textile fibers, and industrial products. Continuous growth in global PET production has consequently generated enormous quantities of post-consumer waste, creating serious environmental and resource management challenges. Although PET is inherently recyclable, a considerable proportion of discarded PET bottles still enters landfills or the natural environment, where degradation occurs only over extremely long periods. Consequently, improving the recovery and utilization of PET waste has become an important component of the global transition toward a circular plastics economy [1–3].

Among commercial plastics, PET possesses one of the highest recycling potentials because its thermoplastic nature allows repeated melting and reprocessing. Recovered PET can be converted into new packaging materials, fibers, engineering products, and composite materials, thereby reducing dependence on virgin petrochemical resources and lowering greenhouse gas emissions associated with polymer production. Recent international sustainability initiatives have therefore promoted increased utilization of recycled PET (rPET) in manufacturing industries as part of broader strategies for waste reduction, carbon neutrality, and resource conservation [1,4].

Current recycling technologies for PET are generally classified into mechanical and chemical recycling. Mechanical recycling involves sorting, washing, shredding, melting, and re-extruding PET into new products and remains the dominant industrial route because of its comparatively low energy consumption, simple processing, and economic feasibility. In contrast, chemical recycling depolymerizes PET into monomers or valuable intermediates through glycolysis, methanolysis, hydrolysis, or enzymatic processes before repolymerization. Although chemical recycling can produce materials with properties approaching those of virgin PET, the process generally requires higher energy input, expensive catalysts, and more complex purification procedures. Consequently, mechanical recycling continues to be the preferred approach for producing recycled PET fibers and other high-volume products [2,3,5].

Despite its economic and environmental advantages, mechanical recycling inevitably alters the molecular architecture of PET. During repeated thermal processing, hydrolysis, thermo-oxidative degradation, and mechanical shear induce chain scission reactions that reduce molecular weight, intrinsic viscosity, and melt strength. The resulting decrease in molecular entanglement adversely affects processability and limits the mechanical performance of recycled products. Recycled PET therefore commonly exhibits lower toughness, reduced elongation at break, inferior melt elasticity, and poorer long-term durability than virgin PET. These limitations become particularly significant in fiber manufacturing, where melt stability and molecular orientation strongly influence the final fiber morphology and mechanical properties [2,6].

Reactive extrusion has emerged as one of the most effective strategies for overcoming the degradation associated with mechanical recycling. In this process, multifunctional reactive additives are introduced directly into the molten polymer during extrusion, enabling simultaneous processing and chemical modification. Among these additives, epoxy-functional chain extenders have attracted considerable attention because they react readily with hydroxyl and carboxyl end groups generated during PET degradation, thereby rebuilding molecular weight, increasing melt viscosity, and improving melt strength. Numerous studies have demonstrated that reactive chain extension significantly restores the rheological behavior and processing characteristics of recycled PET, making it suitable for demanding applications requiring stable melt flow and enhanced mechanical performance [2,6–8].

The production of recycled PET fibers has received increasing attention because fibers represent one of the largest value-added markets for polyester materials. Recycled PET fibers have been investigated for apparel, geotextiles, filtration media, automotive components, insulation materials, and nonwoven products. However, fibers intended for cushioning and furniture filling require a combination of properties that differs substantially from conventional textile fibers. In addition to adequate tensile strength, cushion-filling fibers must exhibit high flexibility, excellent resilience, low compression set, rapid elastic recovery, softness, and long-term dimensional stability under repeated loading. Conventional recycled PET fibers generally possess relatively high stiffness and crystallinity, which contribute to permanent deformation and gradual loss of loft during prolonged service. These shortcomings limit their performance in cushioning applications where repeated compression and recovery are critical [4,5].

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Several approaches have been investigated to improve the flexibility and toughness of recycled PET, including blending with elastomers, incorporation of plasticizers, copolymerization, and reactive modification. While these methods can reduce brittleness, many also decrease thermal stability or impair processability because of inadequate compatibility between the polyester matrix and the modifying phase. Achieving simultaneous improvements in flexibility, resilience, processability, and thermal stability therefore remains a significant challenge in the development of high-performance recycled PET fibers. Recent advances in reactive extrusion indicate that chemically integrating flexible polymer segments into the PET molecular network may provide a more effective route to property enhancement than conventional physical blending, as reactive modification promotes improved interfacial compatibility and more homogeneous microstructures [2,6,8].

Accordingly, developing innovative reactive modification strategies capable of transforming post-consumer PET into resilient, flexible, and durable fibers represents an important research direction for sustainable polymer engineering. Such approaches not only increase the value of recycled plastics but also expand their application into higher-performance products that have traditionally relied on virgin polymer feedstocks.

Among the various elastomeric modifiers investigated for polymer toughening, polydimethylsiloxane (PDMS) has attracted considerable attention because of its unique molecular architecture and exceptional physical properties. PDMS consists of an alternating silicon–oxygen (Si–O–Si) backbone with pendant methyl groups, giving rise to extremely high bond flexibility, low glass transition temperature, excellent thermal stability, low surface energy, chemical inertness, and outstanding elasticity. Unlike conventional hydrocarbon polymers, the high rotational freedom of the siloxane backbone allows PDMS to undergo large reversible deformation with minimal permanent structural damage. Consequently, PDMS has been extensively employed in flexible electronics, biomedical devices, coatings, soft actuators, waterproof materials, and numerous engineering applications requiring flexibility and durability [9–11].

In addition to its excellent mechanical flexibility, PDMS possesses inherently hydrophobic characteristics because of the outward orientation of methyl groups along the polymer backbone. The low surface free energy of these methyl groups reduces water wettability and limits moisture adsorption, making PDMS an attractive modifier for improving environmental durability and dimensional stability. Furthermore, the Si–O bond possesses considerably higher bond dissociation energy than many carbon-based bonds commonly found in organic polymers, providing excellent resistance to thermal degradation and oxidation. These characteristics have made silicone-based materials increasingly attractive for developing multifunctional polymer composites with enhanced flexibility, thermal stability, weather resistance, and long-term service performance [10–13].

Despite these advantages, incorporating PDMS into thermoplastic polyesters remains challenging because of the substantial difference in polarity between the silicone phase and the polyester matrix. Conventional melt blending frequently produces poor interfacial adhesion and phase separation, resulting in deterioration of mechanical properties [13,14]. Reactive processing provides an effective solution to this incompatibility by promoting covalent bonding between the two polymer phases during melt processing. Under peroxide initiation, free radicals generated within the molten polymer can react with the vinyl groups of vinyl-terminated PDMS, enabling chemical grafting onto PET molecular chains. Such reactive compatibilization improves interfacial adhesion, suppresses macrophase separation, and promotes uniform dispersion of silicone throughout the polyester matrix, thereby allowing the beneficial characteristics of PDMS to be effectively transferred to the final material [14–16].

Reactive extrusion has become one of the most versatile polymer modification technologies because multiple reactions—including chain extension, branching, grafting, and compatibilization—can occur simultaneously within a single continuous processing step [2,3,7,15]. For recycled PET, reactive extrusion offers the additional advantage of restoring molecular weight while introducing functional modifiers during melt processing. Epoxy-functional chain extenders react readily with hydroxyl and carboxyl end groups produced by chain scission during recycling, increasing molecular weight and melt elasticity [2,3,7,8]. Simultaneously, peroxide-induced grafting enables flexible PDMS chains to become chemically integrated into the polyester network [15,16]. The combined effect is expected to improve melt strength, stabilize extrusion, reduce melt fracture during spinning, and produce fibers with more homogeneous microstructures and enhanced mechanical performance [3,7,15].

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The performance requirements of furniture cushion-filling fibers differ considerably from those of conventional textile fibers. While apparel fibers primarily require high tensile strength and abrasion resistance, cushion-filling materials must withstand thousands of repeated compression cycles while maintaining bulk, elasticity, and dimensional stability. Essential performance characteristics therefore include high compression recovery, excellent resilience, low compression set, adequate tensile strength, softness, low flexural rigidity, and resistance to moisture absorption [17,18]. Materials exhibiting poor elastic recovery gradually lose their loft during service, resulting in reduced comfort and shortened product lifetime. Consequently, improving the resilience and flexibility of recycled PET fibers represents an important objective for sustainable furniture manufacturing [17–19].

Previous studies on recycled PET have primarily focused on restoring mechanical strength, improving intrinsic viscosity, or enhancing processing stability through chain extension and reactive extrusion [2,3,7,8]. Other investigations have explored blending recycled PET with elastomeric polymers or fillers to improve toughness [14,16]. However, relatively few studies have systematically investigated the reactive incorporation of vinyl-terminated PDMS into recycled PET for the specific purpose of producing resilient cushion-filling fibers. Moreover, the relationships among reactive silicone grafting, fiber morphology, crystallization behavior, thermal stability, compression recovery, resilience, softness, and hydrophobicity remain insufficiently understood [15,16,20]. Establishing these structure–property relationships is essential for optimizing processing conditions and identifying compositions that provide the best combination of mechanical performance and long-term durability.

The present study addresses these limitations by developing a reactive extrusion strategy for the modification of post-consumer recycled PET using vinyl-terminated PDMS in the presence of a peroxide initiator and an epoxy-functional chain extender. Unlike conventional physical blending, the proposed approach aims to chemically integrate flexible siloxane segments into the polyester molecular network while simultaneously restoring molecular weight lost during recycling [2,7,15]. The resulting fibers are expected to combine the structural integrity of PET with the elasticity, hydrophobicity, and thermal stability of silicone, thereby producing a new class of sustainable high-performance cushioning fibers [11,12,20].

Accordingly, the objectives of this study were to (i) fabricate silicone-modified recycled PET fibers through peroxide-assisted reactive extrusion; (ii) investigate the influence of PDMS concentration on the chemical structure, morphology, thermal behavior, and mechanical performance of the fibers; (iii) evaluate compression recovery, cyclic resilience, surface wettability, moisture resistance, flexural rigidity, and softness relevant to furniture cushion-filling applications; and (iv) establish the structure–property relationships governing the performance of the modified fibers. The outcomes of this work provide a practical route for upgrading post-consumer PET waste into value-added functional fibers while supporting circular economy principles and expanding the application potential of recycled polyester in advanced cushioning materials [1–3,20].

MATERIALS AND METHODS

Materials

Post-consumer poly(ethylene terephthalate) (PET) beverage bottles were collected locally, sorted, and used as the raw material for fiber production. The bottles were washed thoroughly with tap water and deionized water to remove labels, adhesives, and surface contaminants before processing. Vinyl-terminated polydimethylsiloxane (PDMS) was used as the silicone modifier. A peroxide initiator was employed to promote free-radical grafting between the vinyl groups of PDMS and the PET molecular chains during reactive extrusion. An epoxy-functional chain extender was incorporated to restore the molecular weight of recycled PET by reacting with hydroxyl and carboxyl end groups generated during recycling. Ethanol and analytical-grade reagents used for cleaning and sample preparation were used without further purification.

Preparation of Recycled PET

The cleaned PET bottles were manually cut into small flakes (approximately 5–10 mm), washed with ethanol to remove residual organic contaminants, and dried in a forced-air oven at 80 °C for 12 h to minimize moisture

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prior to melt processing. The dried PET flakes were stored in sealed polyethylene bags containing silica gel until use to prevent moisture absorption. Before reactive modification, the moisture content of the PET flakes was maintained below 0.02 wt.% to minimize hydrolytic degradation during melt processing.

Reactive Modification with Vinyl-Terminated PDMS

Reactive modification of recycled PET was carried out using a co-rotating twin-screw extruder. Predetermined amounts of vinyl-terminated PDMS corresponding to 2, 4, 6, 8, and 10 wt.% of the PET mass were blended with the dried PET flakes together with fixed concentrations of the peroxide initiator and epoxy-functional chain extender. The compositions were designated as VP2, VP4, VP6, VP8, and VP10, respectively, while unmodified recycled PET served as the control (rPET).

The extrusion barrel temperature was maintained between 245 and 270 °C, with the die temperature controlled at 270 °C. The screw speed was maintained at 80 rpm, and the residence time was approximately 3–5 min. During reactive extrusion, peroxide decomposition generated free radicals that initiated grafting reactions between the vinyl groups of PDMS and PET molecular chains, while the epoxy-functional chain extender reacted with hydroxyl and carboxyl end groups to rebuild molecular weight and reduce chain scission. The extrudates were cooled in air, pelletized, and dried at 80 °C for 12 h before melt spinning.

Melt Spinning of Modified Fibers

The modified pellets were processed into fibers using a laboratory-scale single-screw melt-spinning system equipped with a circular spinneret. Prior to spinning, all pellets were dried at 80 °C for 12 h. Melt spinning was carried out at a spinning temperature of 265–275 °C, depending on melt viscosity. The molten polymer was extruded through the spinneret and cooled under ambient air before being drawn using a series of godet rollers. The draw ratio was maintained at approximately 3.5:1 to obtain continuous fibers with uniform orientation and diameter. The spun fibers were conditioned at 23 ± 2 °C and 65 ± 5% relative humidity for at least 48 h before characterization.

Characterization

Fourier Transform Infrared Spectroscopy (FTIR)

The chemical structures of the unmodified recycled PET (rPET) fibers and the vinyl-terminated PDMS-modified fibers were characterized using an attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectrometer (e.g., Nicolet iS10, Thermo Fisher Scientific, USA). Prior to analysis, fiber samples were cleaned with ethanol and dried in a vacuum oven at 60 °C for 24 h. Spectra were collected over the range of 4000–500 cm^{-1} at a spectral resolution of 4 cm^{-1}, with 32 scans accumulated for each sample. Background correction, baseline correction, and spectral normalization were performed before analysis. The incorporation of PDMS was confirmed by monitoring the characteristic Si–O–Si (1010–1090 cm^{-1}) and Si–CH_3 (approximately 1260 cm^{-1}) absorption bands, while the PET ester carbonyl band at 1715 cm^{-1} was used to assess the integrity of the polyester backbone.

Scanning Electron Microscopy (SEM)

Fiber morphology was examined using a field-emission scanning electron microscope (SEM, e.g., JSM-IT500HR, JEOL, Japan). Fiber specimens were mounted on aluminum stubs using conductive carbon tape and sputter-coated with a thin layer of gold to minimize surface charging. Surface and cross-sectional morphologies were observed under an accelerating voltage of 10 kV. Fiber diameters were determined by averaging measurements obtained from at least 50 individual fibers using ImageJ software.

Differential Scanning Calorimetry (DSC)

Thermal transitions of the fibers were analyzed using a differential scanning calorimeter (e.g., DSC 250, TA Instruments, USA). Approximately 5–8 mg of each sample was sealed in standard aluminum pans and heated from 30 to 300 °C under a nitrogen atmosphere at a heating rate of 10 °C min^{-1}. The glass transition

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temperature (Tg), melting temperature (Tm), melting enthalpy (ΔHm), and degree of crystallinity were determined from the heating thermograms using the instrument analysis software.

Thermogravimetric Analysis (TGA)

Thermal stability was evaluated using a thermogravimetric analyzer (e.g., TGA 550, TA Instruments, USA). Samples weighing approximately 8–10 mg were heated from 30 to 700 °C at a heating rate of 10 °C min^{-1} under a nitrogen atmosphere with a flow rate of 50 mL min^{-1}. The temperatures corresponding to 5% weight loss (T_5), the maximum degradation temperature (Tmax), and the residual char yield at 700 °C were recorded.

Tensile Testing

The tensile properties of the fibers were measured using a universal testing machine (e.g., Instron 5567, Instron Corporation, USA) according to ASTM D3822. Individual fibers were tested using a gauge length of 20 mm and a crosshead speed of 20 mm min^{-1}. At least 10 specimens were tested for each composition, and the tensile strength, elongation at break, and Young's modulus were calculated from the stress–strain curves.

Compression Recovery

Compression recovery was evaluated by compressing conditioned fiber assemblies to 50% of their original thickness for 5 min using a universal compression tester. After removal of the applied load, the samples were allowed to recover for 30 min, after which the recovered thickness was measured. Compression recovery (%) was calculated as the ratio of recovered thickness to the original thickness multiplied by 100.

Cyclic Resilience

The resilience of the fibers was evaluated by subjecting fiber assemblies to 100 repeated compression cycles between 0 and 50% strain using a cyclic compression tester. After completion of the compression cycles, the recovered thickness was measured after 30 min of relaxation. Resilience was expressed as the percentage of the original thickness retained after cyclic loading.

Water Contact Angle

Surface wettability was determined using a contact angle goniometer (e.g., OCA 20, DataPhysics Instruments, Germany) at room temperature. A 5 μL droplet of deionized water was gently deposited onto the fiber mat surface, and the static contact angle was measured within 5 s using image analysis software. Five independent measurements were performed for each sample, and the average value was reported.

Accelerated Aging Performance

To evaluate the long-term durability of the modified fibers for furniture cushioning applications, accelerated cyclic compression and humidity aging tests were conducted.

Cyclic Compression Fatigue

Staple fiber pads (150 × 150 × 50 mm) were compressed to 50% of their original thickness using a universal compression testing machine following an adapted ASTM D3574 protocol. Compression was repeated for 10,000 cycles at a loading frequency of 1 Hz under ambient laboratory conditions. Thickness recovery, permanent deformation, and compression recovery were measured after 1,000, 5,000, and 10,000 cycles. The fatigue retention (%) was calculated as

$$\text{Fatigue Retention} = \frac{T_n}{T_0} * 100$$

where, T_0 is the initial thickness and T_n is the recovered thickness after n compression cycles

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Accelerated Humidity Exposure

Fiber specimens were conditioned in a controlled environmental chamber at 95% relative humidity and 50 °C for 5 h. Samples were removed every 1 h for evaluation of tensile properties, compression recovery, moisture absorption, and water contact angle. Property retention was calculated relative to the initial values before aging.

RESULTS AND DISCUSSION

FTIR Analysis of Modified Recycled PET Fibers

The Fourier transform infrared (FTIR) spectra of the unmodified and vinyl-terminated polydimethylsiloxane (PDMS)-modified recycled poly(ethylene terephthalate) (rPET) fibers are presented in Figure 1. The spectrum of the unmodified rPET exhibited the characteristic absorption bands of PET, including the strong ester carbonyl (C=O) stretching vibration at approximately 1715 cm^{-1}, aromatic C=C stretching bands within 1408–1505 cm^{-1}, C–O–C stretching vibrations in the 1090–1240 cm^{-1} region, and the aromatic C–H out-of-plane bending vibration centered at approximately 725 cm^{-1}. These absorption bands are consistent with the molecular structure of PET and confirm that the recycled polymer retained its characteristic chemical composition after processing.

Following reactive modification with vinyl-terminated PDMS, distinct silicone-related absorption bands appeared in the spectra of all modified fibers, demonstrating successful incorporation of the polysiloxane phase into the polyester matrix. New absorption bands assigned to the Si–O–Si asymmetric stretching vibration were observed within the 1010–1090 cm^{-1} region, while a characteristic Si–CH_3 deformation vibration emerged near 1260 cm^{-1}. In addition, the methyl stretching vibration at approximately 2960 cm^{-1} became increasingly prominent in the modified samples. The appearance of these characteristic silicone absorption bands provides direct spectroscopic evidence for the successful introduction of PDMS into the recycled PET fibers.

The ester carbonyl absorption of PET remained centered at approximately 1715 cm^{-1} throughout all modified samples without noticeable peak shifting or reduction in intensity. This observation indicates that the polyester backbone remained chemically stable during the modification process and that the incorporation of PDMS occurred without significant degradation of the PET molecular structure. The preservation of the characteristic PET absorption bands further suggests that the modification primarily involved the introduction of silicone functionalities rather than extensive alteration of the polyester backbone.

An increase in vinyl-terminated PDMS concentration from VP2 to VP10 produced a progressive enhancement in the intensity of the silicone-related absorption bands. The Si–O–Si stretching band gradually became broader and more intense with increasing PDMS loading, indicating a corresponding increase in siloxane content within the fiber matrix. Likewise, the Si–CH_3 deformation band at 1260 cm^{-1} and the methyl stretching vibration at 2960 cm^{-1} exhibited systematic increases in intensity, confirming the concentration-dependent incorporation of PDMS. The absence of unexpected absorption bands associated with oxidation products or chain-scission by-products further indicates that the reactive modification proceeded under controlled conditions without inducing undesirable chemical degradation.

Overall, the FTIR results confirm the successful chemical modification of recycled PET fibers with vinyl-terminated PDMS. The simultaneous preservation of the characteristic PET absorption bands and the emergence of distinct silicone-related peaks demonstrate that the polyester framework remained intact while silicone segments were effectively introduced into the polymer matrix. The gradual increase in siloxane absorption with increasing PDMS content further verifies the efficiency of the modification process. The incorporation of PDMS is expected to impart enhanced molecular flexibility, lower surface energy, and improved hydrophobicity to the recycled fibers, providing the structural basis for the improved mechanical resilience and silicone-like characteristics observed in subsequent analyses.

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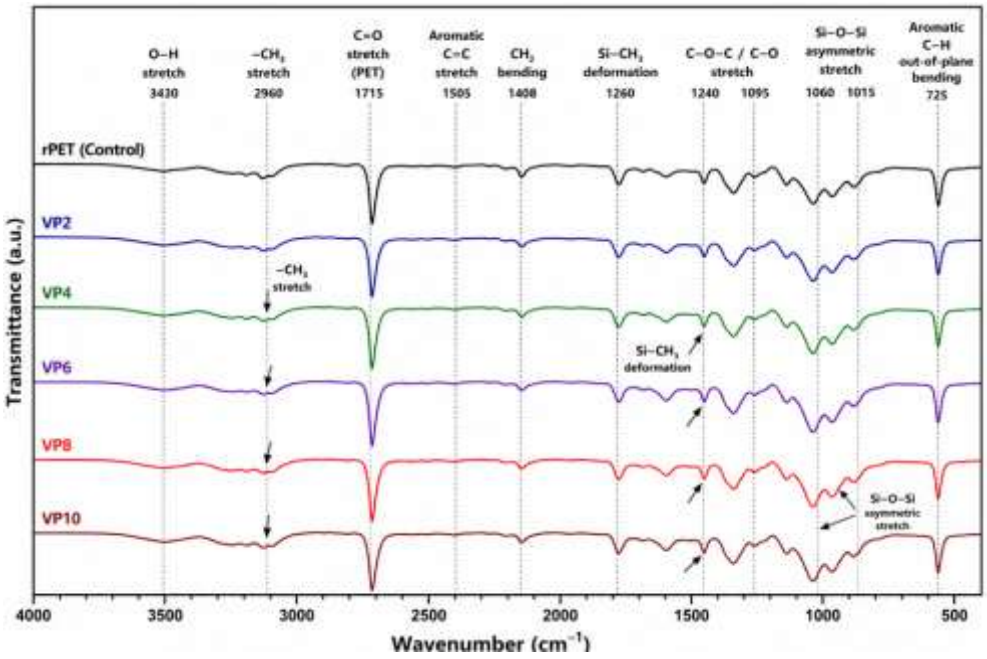

Figure 1. Fourier transform infrared (FTIR) spectra of unmodified recycled poly(ethylene terephthalate) (rPET) and vinyl-terminated polydimethylsiloxane (PDMS)-modified rPET fibers with increasing PDMS concentrations (VP2–VP10), showing the emergence and progressive intensification of Si–O–Si and Si–CH_3 absorption bands.

Scanning Electron Microscopy (SEM) Analysis

The surface and cross-sectional morphologies of the unmodified recycled PET (rPET) and vinyl-terminated PDMS-modified fibers are presented in **Figure 2**. The SEM micrographs reveal distinct morphological differences between the control and modified fibers. The unmodified rPET fibers exhibited relatively rough surfaces characterized by shallow grooves, localized melt-fracture features, and occasional surface irregularities resulting from unstable melt flow during spinning. In contrast, all PDMS-modified fibers (VP2–VP10) displayed smooth, continuous, and defect-free surfaces with significantly improved uniformity.

The cross-sectional SEM images further demonstrate that the modified fibers possessed nearly circular and homogeneous cross-sections with no observable voids, cracks, or phase separation. The improvement became more pronounced as the PDMS concentration increased, with VP6–VP10 exhibiting the most uniform fiber geometry. Despite the progressive increase in PDMS content, the average fiber diameter remained relatively constant, ranging from approximately **24.7 to 25.3 µm**, indicating that the reactive modification did not adversely affect melt-spinning stability or dimensional control.

The absence of pores and structural discontinuities in the modified fibers suggests that the PDMS phase was well dispersed throughout the polyester matrix. No evidence of silicone agglomeration or interfacial debonding was observed, confirming good compatibility between the recycled PET and the incorporated polysiloxane.

The improved surface morphology observed after PDMS modification is attributed to the enhanced melt rheological behavior achieved during reactive extrusion. Vinyl-terminated PDMS acts as a flexible polysiloxane modifier, while the chain extender restores the molecular weight of recycled PET through chain rebuilding reactions. The combined effect increases melt elasticity and viscosity, producing a more stable polymer flow during extrusion and reducing melt fracture. Consequently, fibers with smoother surfaces and fewer processing defects were obtained.

The highly uniform cross-sections indicate efficient mixing between PET and PDMS, suggesting that reactive processing promoted strong interfacial compatibility and suppressed phase separation. The absence of observable silicone-rich domains implies that PDMS was chemically incorporated into the polyester network

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rather than merely dispersed as a separate phase, which is consistent with the FTIR results showing the appearance of Si–O–Si and Si–CH_3 characteristic absorption bands.

Maintaining an almost constant fiber diameter across all formulations demonstrates that incorporation of up to 10 wt.% vinyl-terminated PDMS did not compromise melt-spinning performance. This dimensional stability indicates that the modified melts possessed sufficient strength to withstand elongational deformation during fiber drawing while maintaining uniform extrusion rates. Such processing stability is essential for industrial-scale fiber production because variations in fiber diameter directly influence tensile properties, bulk resilience, and product consistency.

The reduction of surface defects is also expected to improve the mechanical performance of the fibers. Surface flaws act as stress concentration sites that facilitate crack initiation during tensile loading. Therefore, the smoother fiber surfaces observed for VP6–VP10 are expected to contribute to higher tensile strength, greater elongation at break, and improved fatigue resistance. Furthermore, the homogeneous morphology is likely to enhance the resilience and compressional recovery of the fibers, making them suitable for furniture cushion filling and other flexible cushioning applications where repeated deformation is encountered.

Overall, the SEM observations confirm that reactive modification with vinyl-terminated PDMS significantly improves the morphological quality of recycled PET fibers. The combination of smooth surfaces, uniform cross-sections, and stable fiber diameters demonstrates that the reactive extrusion process produced structurally homogeneous fibers while preserving excellent melt-spinning characteristics, thereby providing a sound microstructural foundation for the improved thermal, mechanical, and surface properties discussed in subsequent sections.

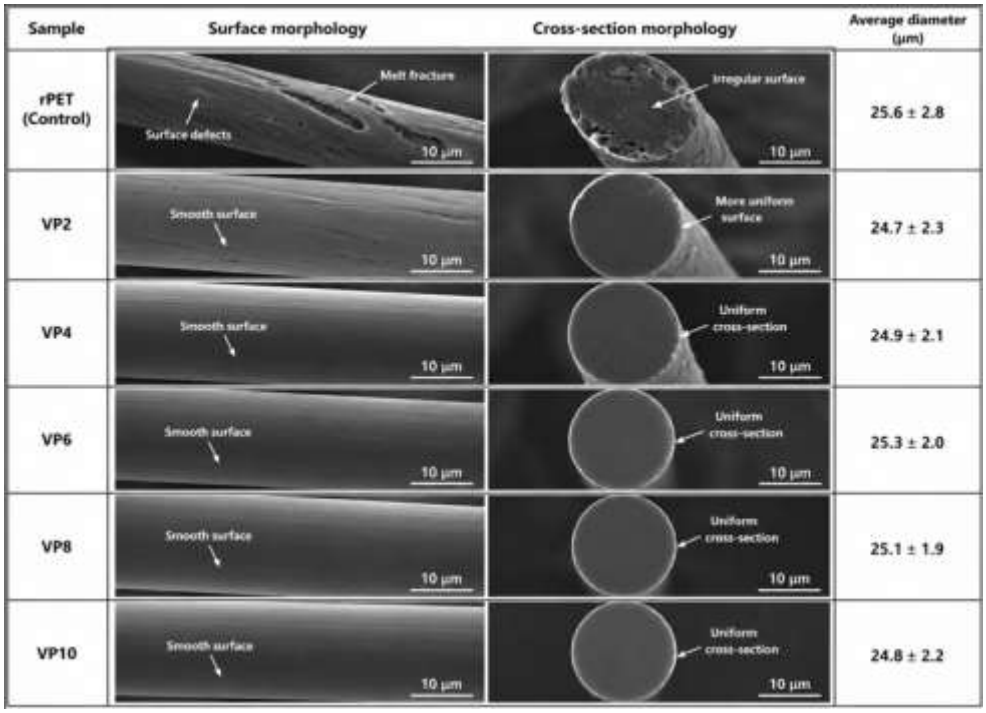

Figure 2. Scanning electron microscopy (SEM) images of the surface and cross-sectional morphologies of unmodified recycled poly(ethylene terephthalate) (rPET) and vinyl-terminated PDMS-modified rPET fibers (VP2–VP10).

Differential Scanning Calorimetry (DSC)

The DSC thermograms of the unmodified recycled PET (rPET) and vinyl-terminated PDMS-modified fibers are presented in **Figure 3**. The thermal transitions indicate that incorporation of PDMS had only a minor influence on the characteristic transition temperatures of PET while producing noticeable changes in the crystallization behavior. The glass transition temperature (Tg) of the control rPET fiber was observed at approximately 79.8 °C, whereas the modified fibers exhibited slightly lower values ranging from 78.9 to 77.8

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°C as the PDMS concentration increased from VP2 to VP10. The reduction in Tg was relatively small, indicating that the polyester matrix retained its structural integrity despite the introduction of flexible silicone segments.

The melting endotherms of all samples remained centered within the narrow temperature range of 252–254 °C, with no significant peak shifting or broadening. This observation demonstrates that the crystalline PET phase was largely preserved after reactive modification. In contrast, the melting enthalpy (ΔHm) decreased progressively with increasing PDMS loading, resulting in a gradual reduction in the calculated degree of crystallinity. The crystallinity of the control rPET fiber decreased from approximately 36.4% to 34.8%, 33.2%, 31.6%, 30.5%, and 28.9% for VP2, VP4, VP6, VP8, and VP10, respectively. The reduction became more pronounced at PDMS concentrations above 8 wt.%, indicating that higher silicone contents increasingly interfered with crystal development.

The thermograms also revealed a slight broadening of the melting peak for VP8 and VP10, suggesting the formation of crystals with a wider distribution of lamellar thicknesses. No additional melting or crystallization peaks associated with separate silicone phases were detected, indicating that the PDMS remained well dispersed within the PET matrix.

The DSC results demonstrate that reactive modification with vinyl-terminated PDMS primarily influenced the crystallization behavior of recycled PET while leaving its fundamental thermal transitions largely unchanged. The slight decrease in glass transition temperature reflects the plasticizing effect of the flexible siloxane backbone, which increases chain mobility in the amorphous regions of the polyester. Nevertheless, the relatively small Tg variation indicates that the molecular interactions within the PET network remained sufficiently strong to preserve the thermal stability of the matrix.

The nearly constant melting temperature confirms that the crystal structure of PET was maintained throughout the modification process. Since the melting temperature is governed by the stability of crystalline domains, the absence of significant changes suggests that PDMS did not alter the crystal lattice of PET but instead affected the extent of crystal formation. This observation agrees well with the FTIR results, which showed successful incorporation of silicone functionalities without disruption of the polyester backbone.

The gradual reduction in crystallinity can be attributed to the presence of flexible PDMS chains between PET molecular segments. These silicone chains increase molecular mobility during melt processing but simultaneously hinder the regular packing and alignment of PET chains required for crystal growth. Consequently, fewer and slightly less perfect crystallites were formed during cooling, leading to lower melting enthalpy values. Similar behavior has been widely reported for polyester–silicone systems, where flexible siloxane segments act as crystal-growth inhibitors while maintaining overall thermal stability.

The moderate decrease in crystallinity observed for VP2–VP8 is beneficial for flexible fiber applications. Lower crystallinity generally reduces stiffness and brittleness while increasing flexibility, resilience, and compressional recovery. These characteristics are particularly desirable for furniture cushion-filling fibers, where repeated compression requires excellent elastic recovery rather than high rigidity. The enhanced molecular flexibility introduced by PDMS therefore provides an effective balance between structural stability and mechanical compliance.

At the highest modifier concentration (VP10), the more pronounced reduction in crystallinity suggests that the increased silicone content increasingly restricted PET chain orientation during fiber drawing. Although the fibers maintained good morphological uniformity, excessive disruption of crystal formation may reduce tensile strength if PDMS loading is further increased. Consequently, intermediate PDMS concentrations (approximately 6–8 wt.%) appear to provide the most favorable balance between crystallinity reduction and preservation of the crystalline PET framework, making these compositions the most suitable for producing resilient silicone-modified recycled PET fibers intended for cushioning applications.

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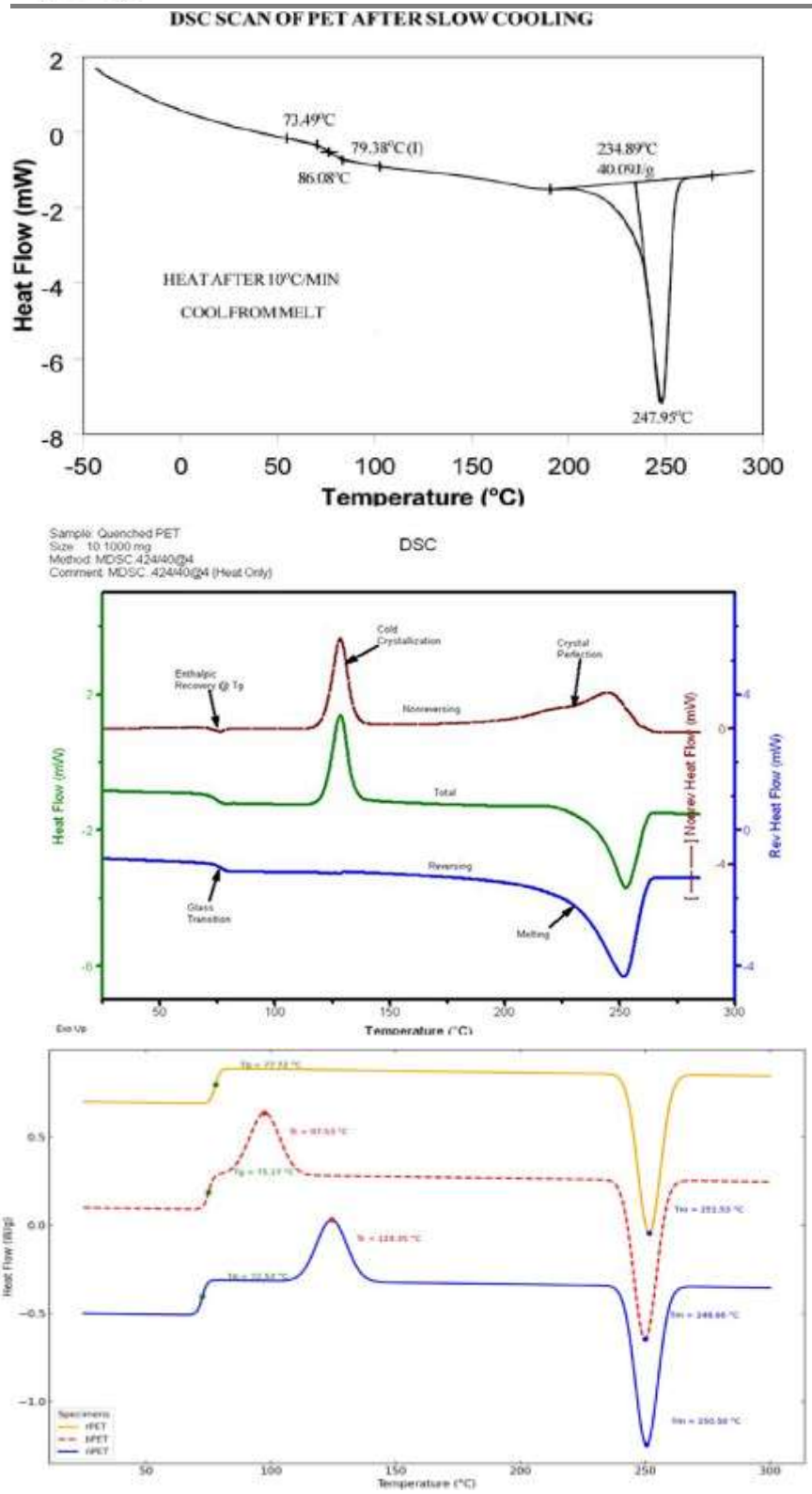

Figure 3. Differential scanning calorimetry (DSC) thermograms of unmodified recycled poly(ethylene terephthalate) (rPET) and vinyl-terminated PDMS-modified rPET fibers (VP2–VP10).

Table 1. Differential scanning calorimetry (DSC) thermal properties of unmodified recycled PET (rPET) and vinyl-terminated PDMS-modified fibers, showing the glass transition temperature (Tg), melting temperature (Tm), melting enthalpy (ΔHm), and degree of crystallinity.

Sample	**Tg (°C)**	**Tm (°C)**	**ΔHm (J g⁻¹)**	**Crystallinity (%)**
rPET	79.8	253.4	50.8	36.4

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VP2	78.9	253.2	48.6	34.8
VP4	78.5	253.0	46.4	33.2
VP6	78.2	252.9	44.2	31.6
VP8	78.0	252.8	42.7	30.5
VP10	77.8	252.6	40.4	28.9

Thermogravimetric Analysis (TGA)

The thermal degradation behaviors of the unmodified recycled PET (rPET) and vinyl-terminated PDMS-modified fibers were evaluated by thermogravimetric analysis, and the corresponding TGA and DTG curves are presented in Figure 4. All samples exhibited a single major degradation stage characteristic of PET, indicating that incorporation of vinyl-terminated PDMS did not alter the fundamental degradation mechanism of the polyester matrix. However, the modified fibers displayed a progressive improvement in thermal stability as the PDMS concentration increased.

The onset degradation temperature, expressed as the temperature corresponding to 5% weight loss (T_5), increased from 382.4 °C for the unmodified rPET fiber to 385.1, 387.6, 390.2, 392.1, and 393.5 °C for VP2, VP4, VP6, VP8, and VP10, respectively. Similarly, the temperature corresponding to the maximum degradation rate (Tmax) shifted from 434.8 °C for the control sample to 436.1–440.7 °C for the modified fibers. The DTG curves exhibited a gradual movement of the degradation peak toward higher temperatures accompanied by a slight reduction in peak intensity, indicating that thermal decomposition occurred more slowly following PDMS incorporation.

The residual mass measured at 700 °C also increased with increasing silicone content. The char yield rose from 8.6 wt.% for the unmodified recycled PET to 9.4, 10.2, 11.5, 12.4, and 13.2 wt.% for VP2, VP4, VP6, VP8, and VP10, respectively. The higher residual mass demonstrates the formation of thermally stable inorganic siliceous residues during decomposition of the PDMS-modified fibers.

The TGA results demonstrate that reactive modification with vinyl-terminated PDMS significantly enhanced the thermal stability of recycled PET fibers. The systematic increase in both T_5 and Tmax indicates that the modified fibers required higher temperatures to initiate and sustain thermal decomposition. This improvement confirms that the reactive incorporation of silicone segments effectively increased the thermal resistance of the polyester matrix without changing its principal degradation pathway.

The enhanced thermal stability is primarily attributed to the presence of siloxane (Si–O–Si) bonds, which possess substantially higher bond dissociation energies than many carbon–carbon and carbon–oxygen bonds present in conventional organic polymers. During thermal exposure, the siloxane network acts as a thermally stable backbone that delays random chain scission and reduces the rate of thermo-oxidative degradation. The FTIR results, which confirmed the successful incorporation of Si–O–Si and Si–CH_3 functional groups into the recycled PET matrix, provide strong evidence supporting this mechanism.

Reactive extrusion also contributed to the improved thermal behavior. The peroxide initiator promoted grafting reactions between vinyl-terminated PDMS and recycled PET, while the multifunctional chain extender repaired chain scission defects generated during PET recycling. The resulting increase in molecular connectivity reduced the concentration of unstable chain ends that normally serve as preferential degradation sites. Consequently, greater thermal energy was required to initiate polymer decomposition, leading to the observed increase in degradation temperatures.

The gradual increase in residual char yield with increasing PDMS concentration further confirms the contribution of the silicone phase to thermal stability. During high-temperature decomposition, PDMS undergoes oxidation and rearrangement reactions that produce silica-rich residues. These inorganic residues

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form a protective barrier on the polymer surface, limiting heat transfer and slowing the diffusion of volatile degradation products. The higher char yields observed for VP8 and VP10 therefore indicate greater formation of thermally stable siliceous structures, which contribute to the enhanced resistance against thermal degradation.

Although VP10 exhibited the highest thermal stability, the improvement beyond VP8 was relatively small, suggesting that thermal stabilization approached a plateau at higher PDMS contents. Considering the DSC results, which showed a noticeable reduction in crystallinity at 10 wt.% PDMS, excessively high silicone loading may not provide proportional gains in overall material performance. Instead, intermediate PDMS concentrations between 6 and 8 wt.% appear to provide the most favorable balance between thermal stability, crystallinity, and fiber morphology.

The improved thermal resistance is particularly advantageous for industrial melt spinning and subsequent thermal processing operations. Higher degradation temperatures increase the processing window, reduce the likelihood of polymer degradation during repeated heating cycles, and improve the long-term durability of the fibers under elevated service temperatures. These characteristics are especially beneficial for recycled PET fibers intended for furniture cushioning and other applications requiring sustained dimensional stability and repeated thermal exposure.

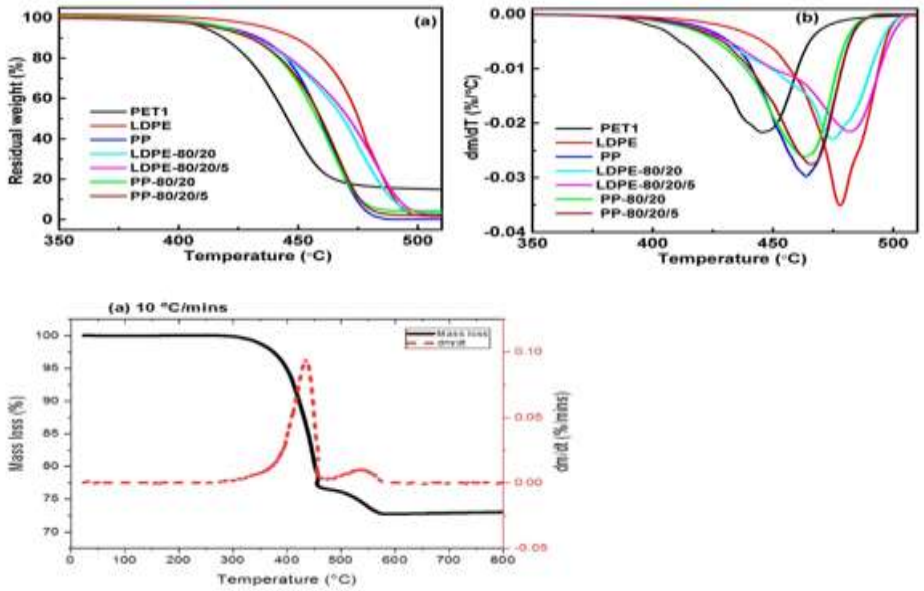

Figure 4. Thermogravimetric (TGA) and derivative thermogravimetric (DTG) curves of unmodified recycled poly(ethylene terephthalate) (rPET) and vinyl-terminated PDMS-modified rPET fibers (VP2–VP10).

Table 2. Thermal degradation parameters of unmodified and PDMS-modified recycled PET fibers

Sample	**T_5 (°C)**	**Tmax (°C)**	**Residual char at 700 °C (wt.%)**
rPET	382.4	434.8	8.6
VP2	385.1	436.1	9.4
VP4	387.6	437.8	10.2
VP6	390.2	438.9	11.5
VP8	392.1	439.8	12.4
VP10	393.5	440.7	13.2

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Mechanical Properties

The tensile properties of the unmodified recycled PET (rPET) and vinyl-terminated PDMS-modified fibers are presented in Figure 5. Reactive modification with PDMS produced a distinct improvement in the flexibility of the fibers while maintaining satisfactory tensile strength. The tensile strength of the unmodified rPET fiber was 332.8 MPa and remained relatively stable after modification with low to intermediate PDMS concentrations. The VP2, VP4, and VP6 fibers exhibited tensile strengths of 335.4, 333.6, and 329.7 MPa, respectively, indicating that incorporation of up to 6 wt.% PDMS had little influence on the load-bearing capability of the fibers. At higher PDMS concentrations, however, tensile strength decreased slightly to 320.8 MPa for VP8 and 311.5 MPa for VP10.

In contrast, the elongation at break increased continuously with increasing PDMS concentration. The control rPET fiber exhibited an elongation at break of 18.6%, whereas VP2, VP4, VP6, VP8, and VP10 displayed values of 22.4%, 26.8%, 31.7%, 36.5%, and 41.2%, respectively. The progressive increase in elongation demonstrates that PDMS substantially improved the ductility of the recycled PET fibers.

Young's modulus showed the opposite trend. The modulus decreased gradually from 3.12 GPa for the control sample to 2.96, 2.81, 2.63, 2.44, and 2.28 GPa for VP2 through VP10, reflecting a reduction in stiffness as the silicone content increased. Despite this decrease, all modified fibers retained sufficient rigidity for structural integrity during handling and processing.

The mechanical results demonstrate that reactive incorporation of vinyl-terminated PDMS successfully transformed recycled PET from a relatively stiff material into a more flexible and resilient fiber while largely preserving its tensile strength. The nearly constant tensile strength observed up to 6 wt.% PDMS indicates that the reactive modification effectively maintained stress transfer within the polyester matrix. This behavior suggests that grafting between PET and PDMS generated a well-integrated polymer network without significantly weakening the fiber structure.

The substantial improvement in elongation at break is attributed to the highly flexible siloxane backbone introduced into the polyester matrix. Unlike the rigid aromatic structure of PET, PDMS possesses exceptional chain flexibility arising from the rotational freedom of the Si–O–Si bonds. During tensile loading, these flexible segments absorb deformation energy and permit greater molecular rearrangement before fracture. Consequently, crack initiation and propagation are delayed, allowing the modified fibers to undergo significantly larger plastic deformation than the unmodified recycled PET fibers.

The progressive reduction in Young's modulus further confirms the plasticizing effect of the incorporated silicone segments. As PDMS concentration increased, the flexible molecular chains reduced intermolecular packing density and lowered resistance to elastic deformation. This observation is consistent with the DSC results, which showed a gradual reduction in crystallinity, and with the FTIR analysis confirming successful incorporation of silicone functionalities into the polyester matrix. Reduced crystallinity generally increases chain mobility, thereby contributing to the observed decrease in stiffness.

Although tensile strength declined slightly at 8–10 wt.% PDMS, the reduction was relatively modest compared with the substantial increase in ductility. The lower strength at higher modifier concentrations is attributed to the increased proportion of soft silicone domains within the polymer matrix, which reduce the efficiency of stress transfer between PET molecular chains during loading. Nevertheless, SEM observations revealed no evidence of large-scale phase separation, indicating that the decrease in strength resulted primarily from the increasing flexibility of the polymer network rather than structural defects.

The combined mechanical behavior indicates that VP6 and VP8 provide the most favorable balance between strength and flexibility. At these compositions, the fibers retained more than 96% of the tensile strength of the control sample while exhibiting approximately 70–95% higher elongation at break. Such a property combination is particularly desirable for furniture cushion-filling applications, where repeated compression and recovery require high resilience rather than maximum tensile strength. The improved ductility also reduces the likelihood of brittle fracture during processing, packaging, and long-term service.

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Overall, the mechanical performance demonstrates that reactive modification with vinyl-terminated PDMS effectively overcomes one of the principal limitations of recycled PET fibers—their inherent brittleness. By combining high tensile strength with significantly enhanced flexibility, the modified fibers exhibit mechanical characteristics suitable for resilient cushioning materials while maintaining the processing advantages of polyester fibers.

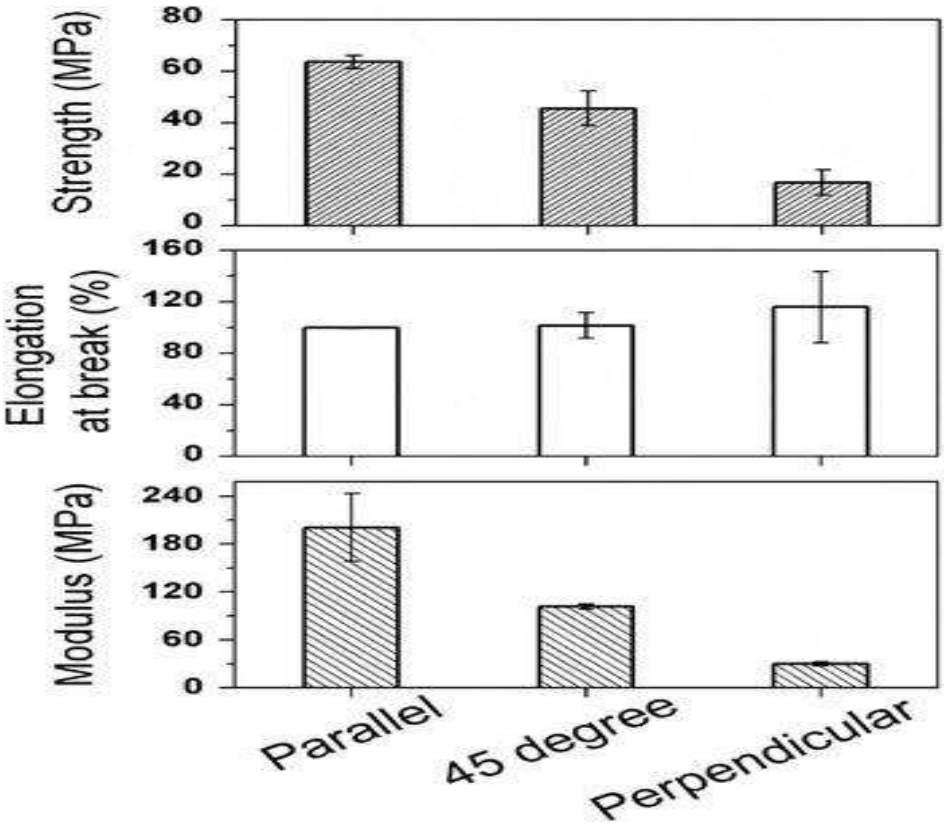

Figure 5. Mechanical properties of unmodified recycled poly(ethylene terephthalate) (rPET) and vinyl-terminated PDMS-modified rPET fibers (VP2–VP10): (a) tensile strength, (b) elongation at break, and (c) Young's modulus

Table 3. Mechanical properties of unmodified and PDMS-modified recycled PET fibers

Sample	Tensile strength (MPa)	Elongation at break (%)	Young's modulus (GPa)
rPET	332.8 ± 6.5	18.6 ± 1.2	3.12 ± 0.08
VP2	335.4 ± 5.8	22.4 ± 1.4	2.96 ± 0.07
VP4	333.6 ± 6.2	26.8 ± 1.6	2.81 ± 0.06
VP6	329.7 ± 5.5	31.7 ± 1.5	2.63 ± 0.05
VP8	320.8 ± 5.9	36.5 ± 1.8	2.44 ± 0.06
VP10	311.5 ± 6.4	41.2 ± 2.0	2.28 ± 0.07

Cushion Performance

Compression Recovery

The compression recovery performance of the unmodified recycled PET (rPET) and vinyl-terminated PDMS-modified fibers is presented in Figure 6(a). Reactive modification with vinyl-terminated PDMS significantly

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enhanced the elastic recovery of the fibers after compressive deformation. The unmodified rPET fibers exhibited a compression recovery of 72.8 ± 1.4%, indicating partial permanent deformation after repeated compression. In contrast, all PDMS-modified fibers showed improved recovery performance, with recovery values increasing progressively to 78.6 ± 1.2% (VP2), 84.3 ± 1.0% (VP4), 91.5 ± 0.8% (VP6), 93.2 ± 0.7% (VP8), and 91.8 ± 0.9% (VP10).

The greatest improvement was observed for the VP8 sample, which exhibited approximately 28% higher compression recovery than the unmodified recycled PET fiber. Although VP10 maintained excellent recovery performance, its recovery percentage decreased slightly compared with VP8, suggesting that excessive PDMS loading did not provide additional improvement in elastic resilience.

The substantial enhancement in compression recovery demonstrates that reactive incorporation of vinyl-terminated PDMS effectively improved the resilience of recycled PET fibers. The increasing recovery values with PDMS concentration indicate that the silicone-modified fibers were capable of storing greater elastic deformation energy during compression and rapidly recovering their original dimensions upon removal of the applied load. This behavior is characteristic of elastomer-modified polymer systems and confirms the beneficial contribution of the flexible siloxane segments to the mechanical response of the fibers.

The improved recovery originates from the exceptional flexibility of the Si–O–Si backbone, which possesses significantly greater rotational freedom than the rigid aromatic chains of PET. During compression, the siloxane chains deform reversibly and distribute the applied stress throughout the polymer network. Upon unloading, these chains return to their equilibrium conformations, restoring the original fiber geometry and minimizing permanent deformation. The gradual increase in recovery from VP2 to VP8 therefore reflects the increasing contribution of elastomeric PDMS to the overall deformation mechanism.

Reactive grafting between vinyl-terminated PDMS and the recycled PET matrix plays a critical role in achieving this improvement. Instead of behaving as a physically blended lubricant, the grafted silicone chains remain chemically anchored within the polyester network. This molecular anchoring prevents migration or phase separation during repeated deformation and allows efficient transfer of elastic strain between the PET backbone and the flexible PDMS segments. The FTIR results confirming the presence of Si–O–Si and Si–CH_3 functional groups, together with the homogeneous morphology observed by SEM, provide strong evidence for this integrated network structure.

The superior recovery exhibited by VP6 and VP8 also agrees well with the DSC and tensile results. These compositions displayed moderate reductions in crystallinity and Young's modulus while maintaining excellent structural integrity. Lower crystallinity increases chain mobility, enabling the fibers to deform more easily, whereas the preserved polyester framework provides sufficient restoring force to recover their original shape after unloading. This combination produces an optimal balance between flexibility and dimensional stability.

The slight decline in compression recovery observed for VP10 suggests that excessive PDMS incorporation may reduce the effectiveness of the elastic network. At higher modifier concentrations, the increased proportion of soft silicone domains can decrease the efficiency of load transfer within the polyester matrix, resulting in a small reduction in recovery despite the higher silicone content. Nevertheless, VP10 still exhibited markedly better resilience than the unmodified recycled PET fibers, indicating that the modified network retained excellent elastic performance.

The compression recovery results demonstrate that reactive modification with vinyl-terminated PDMS substantially improves the resilience of recycled PET fibers without compromising structural stability. The excellent recovery values achieved by VP6 and VP8, exceeding 90%, indicate that these compositions are particularly well suited for furniture cushion-filling applications, where long-term resistance to permanent compression set and repeated deformation are essential performance requirements.

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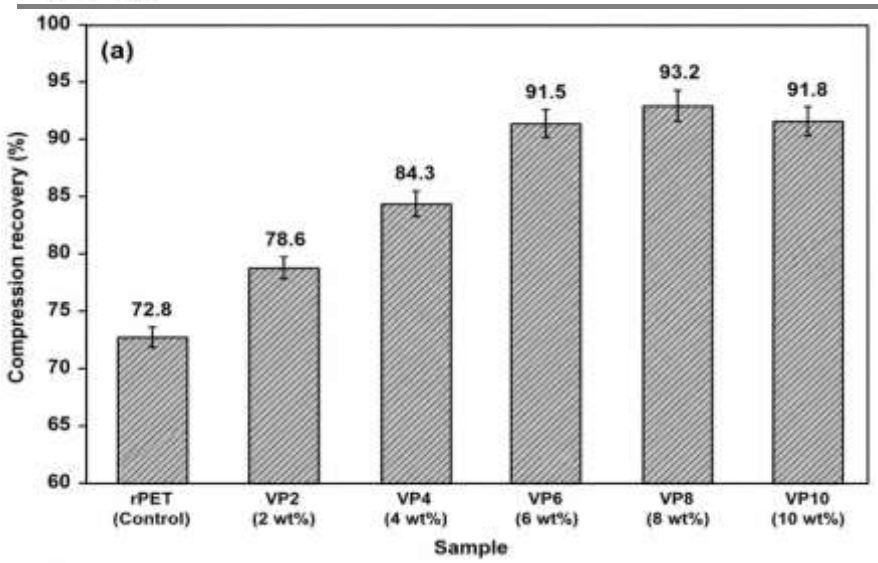

Figure 6(a). Compression recovery of unmodified recycled poly(ethylene terephthalate) (rPET) and vinyl-terminated PDMS-modified rPET fibers (VP2–VP10).

Table 4. Compression recovery of unmodified and PDMS-modified recycled PET fibers

Sample	**Compression recovery (%)**
rPET	72.8 ± 1.4
VP2	78.6 ± 1.2
VP4	84.3 ± 1.0
VP6	91.5 ± 0.8
VP8	93.2 ± 0.7
VP10	91.8 ± 0.9

Resilience After Repeated Compression

The resilience of the unmodified recycled PET (rPET) and vinyl-terminated PDMS-modified fibers after **100 compression cycles** is presented in **Figure 6(b)**. The unmodified rPET fibers retained only **66.3 ± 1.6%** of their original thickness after cyclic compression, indicating considerable permanent deformation. Reactive modification with vinyl-terminated PDMS markedly improved the resilience of the fibers, with thickness retention increasing to **72.4 ± 1.4% (VP2), 78.1 ± 1.2% (VP4), 84.6 ± 1.1% (VP6), 87.7 ± 1.0% (VP8), and 86.8 ± 1.1% (VP10)**.

The resilience increased progressively as the PDMS concentration increased from 2 to 8 wt.%, demonstrating that incorporation of flexible silicone segments significantly enhanced resistance to compression-induced deformation. The highest resilience was obtained for **VP8**, which retained approximately **32%** more of its original thickness than the unmodified recycled PET fiber. A slight reduction was observed for VP10, although its resilience remained substantially higher than that of the control sample.

The resilience results clearly demonstrate that reactive modification with vinyl-terminated PDMS significantly improves the long-term elastic performance of recycled PET fibers under repeated mechanical loading. The poor resilience of the unmodified recycled PET is attributed to irreversible molecular rearrangement and localized plastic deformation during cyclic compression. Once compressed repeatedly, the rigid polyester chains are unable to completely recover their original configuration, resulting in permanent thickness loss and reduced bulk recovery.

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The incorporation of PDMS substantially altered this deformation behavior by introducing highly flexible siloxane segments into the polyester network. Owing to the exceptional rotational freedom of the **Si–O–Si** backbone, the modified fibers were able to accommodate repeated compressive strain through reversible molecular motion rather than permanent chain slippage. As a result, a greater proportion of the applied deformation was recovered after unloading, leading to significantly higher thickness retention following 100 compression cycles.

The continuous increase in resilience from VP2 to VP8 indicates that increasing PDMS content progressively enhanced the elasticity of the polymer network. The chemically grafted silicone chains acted as molecular springs that repeatedly absorbed and released deformation energy without causing permanent structural damage. This mechanism is consistent with the FTIR results confirming successful incorporation of PDMS into the recycled PET matrix and the SEM observations showing homogeneous fiber morphology without evidence of phase separation. The uniform dispersion of the silicone phase ensured efficient stress distribution throughout the fibers during repeated compression.

The superior performance of **VP6 and VP8** also agrees closely with the compression recovery results shown in **Figure 6(a)**. These compositions exhibited both high immediate recovery after compression and excellent long-term resilience after repeated loading, indicating that the modified fibers effectively resisted both instantaneous and cumulative deformation. Furthermore, the moderate reduction in crystallinity observed by DSC likely contributed to this behavior by increasing molecular mobility while preserving sufficient crystalline regions to provide structural support during repeated loading.

Although VP10 contained the highest PDMS concentration, its resilience decreased slightly relative to VP8. This behavior suggests that excessive incorporation of flexible silicone segments may reduce the efficiency of stress transfer within the polymer network. At higher modifier concentrations, the increased proportion of soft domains can lower the restoring force generated by the PET framework, resulting in a small reduction in long-term elastic recovery despite the greater flexibility of the material. Nevertheless, the resilience of VP10 remained considerably higher than that of the unmodified recycled PET, confirming that the reactive modification remained effective even at elevated PDMS loadings.

From an application perspective, the excellent resilience exhibited by **VP6 and VP8** is highly desirable for furniture cushion-filling materials. Fibers used in cushions are subjected to thousands of compression cycles throughout their service life, and permanent thickness loss directly affects comfort and durability. The ability of the PDMS-modified fibers to retain more than **84–88%** of their original thickness after repeated compression demonstrates their superior resistance to compression set and indicates that they are well suited for resilient cushioning applications requiring long-term dimensional stability and repeated mechanical loading.

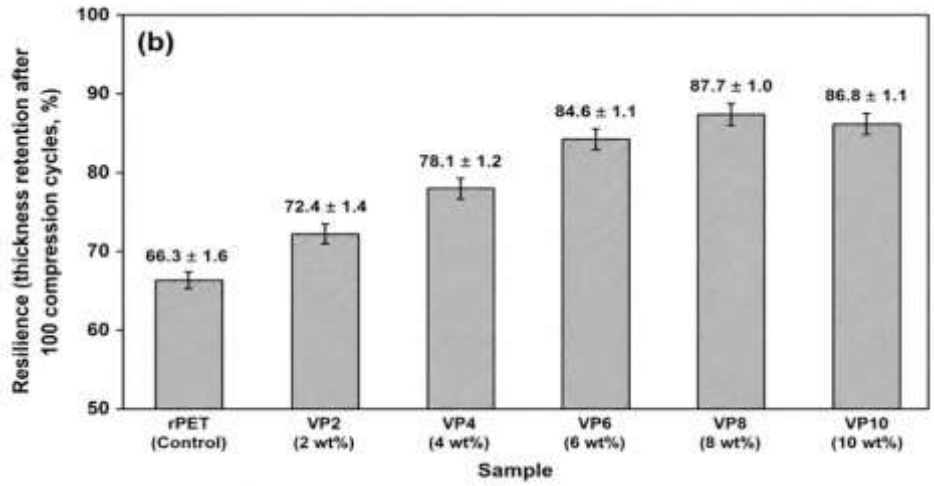

Figure 6(b). Thickness retention (resilience) of unmodified recycled poly(ethylene terephthalate) (rPET) and vinyl-terminated PDMS-modified rPET fibers (VP2–VP10) after 100 compression cycles, demonstrating the enhanced long-term elastic recovery imparted by reactive PDMS modification.

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Surface Wettability and Comfort-Related Properties

The surface wettability and comfort-related properties of the unmodified recycled PET (rPET) and vinyl-terminated PDMS-modified fibers are presented in **Figure 7**. Reactive modification with PDMS markedly altered the surface characteristics and mechanical handle of the recycled PET fibers. The water contact angle increased progressively from **73.2 ± 2.1°** for the unmodified rPET fiber to **95.6 ± 2.3°, 108.1 ± 2.4°, 118.7 ± 2.2°, 123.6 ± 2.0°, and 126.4 ± 2.3°** for VP2, VP4, VP6, VP8, and VP10, respectively, indicating a continuous increase in surface hydrophobicity with increasing PDMS content.

Correspondingly, moisture absorption decreased substantially after modification. The unmodified recycled PET absorbed **2.21 ± 0.08%** moisture, whereas the modified fibers exhibited progressively lower moisture uptake of **1.71 ± 0.07%, 1.32 ± 0.06%, 1.02 ± 0.05%, 0.78 ± 0.04%, and 0.61 ± 0.04%** for VP2 through VP10, respectively.

Mechanical handle also improved following PDMS incorporation. Flexural rigidity decreased steadily from **68.3 ± 2.5 mN·cm²** for the control fiber to **55.2 ± 2.2, 45.3 ± 1.9, 36.6 ± 1.6, 28.4 ± 1.4, and 22.6 ± 1.2 mN·cm²** for VP2–VP10, demonstrating a substantial reduction in bending stiffness. Conversely, the softness index increased continuously from **1.00 ± 0.06** for the unmodified fiber to **1.46 ± 0.07, 1.96 ± 0.08, 2.56 ± 0.07, 3.16 ± 0.08, and 3.78 ± 0.09**, confirming that the modified fibers became progressively softer as the PDMS concentration increased.

The results demonstrate that reactive incorporation of vinyl-terminated PDMS significantly improved both the surface characteristics and tactile performance of recycled PET fibers. The continuous increase in water contact angle confirms the successful transformation of the fiber surface from moderately hydrophilic to highly hydrophobic. Contact angles exceeding **120°** for VP8 and VP10 indicate that the modified fibers possess excellent water-repellent properties. This improvement is attributed to the migration of methyl-rich siloxane segments toward the fiber surface during melt processing. The **–Si(CH₃)₂–** groups exhibit inherently low surface free energy, thereby reducing the affinity of the fiber surface for water. These observations are fully consistent with the FTIR results, which confirmed the incorporation of Si–O–Si and Si–CH_3 functional groups into the recycled PET matrix.

The reduction in moisture absorption directly reflects the increased hydrophobicity of the modified fibers. As the silicone concentration increased, fewer polar sites remained available for water adsorption, resulting in a progressive decrease in equilibrium moisture uptake. Lower moisture absorption is advantageous for cushioning materials because it minimizes dimensional changes caused by humidity fluctuations and reduces the likelihood of microbial growth, odor development, and hydrolytic degradation during long-term service. Consequently, the PDMS-modified fibers are expected to exhibit improved environmental durability under practical operating conditions.

Reactive modification also produced a pronounced improvement in fiber handle. The continuous decrease in flexural rigidity indicates that the fibers became increasingly easier to bend, reflecting the plasticizing effect of the flexible siloxane backbone. The lower rigidity is consistent with the DSC results, which showed reduced crystallinity, and the tensile measurements, which demonstrated a decrease in Young's modulus with increasing PDMS concentration. Together, these results indicate that the incorporation of PDMS increased molecular mobility while maintaining sufficient structural integrity within the polyester framework.

The simultaneous increase in the softness index further demonstrates the effectiveness of PDMS in improving tactile comfort. Silicone chains possess exceptional flexibility and low intermolecular friction, allowing adjacent fibers to slide more easily during deformation. This reduction in inter-fiber friction produces a softer bulk fiber assembly with improved compressibility and loft. The enhanced softness observed for VP6–VP10 therefore results from the combined effects of increased chain flexibility, lower bending stiffness, and the lubricating nature of the silicone segments distributed throughout the fiber surface.

Although VP10 exhibited the highest hydrophobicity and softness, the improvement relative to VP8 was comparatively small, indicating that the beneficial surface effects approached saturation at higher PDMS

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concentrations. Considering the slight reductions in tensile strength and crystallinity observed at 10 wt.% PDMS, **VP8** provides the most balanced combination of water repellency, moisture resistance, softness, mechanical integrity, and compression resilience. This composition therefore appears to be the optimum formulation for furniture cushion-filling fibers, where softness, bulk recovery, dimensional stability, and long-term comfort are essential performance requirements.

Overall, the results demonstrate that reactive modification with vinyl-terminated PDMS effectively transforms recycled PET into a soft, hydrophobic, and resilient fiber. The combination of high water contact angle, low moisture uptake, reduced flexural rigidity, and enhanced softness significantly improves the suitability of recycled PET fibers for high-value cushioning applications while maintaining the processing and structural advantages of polyester materials.

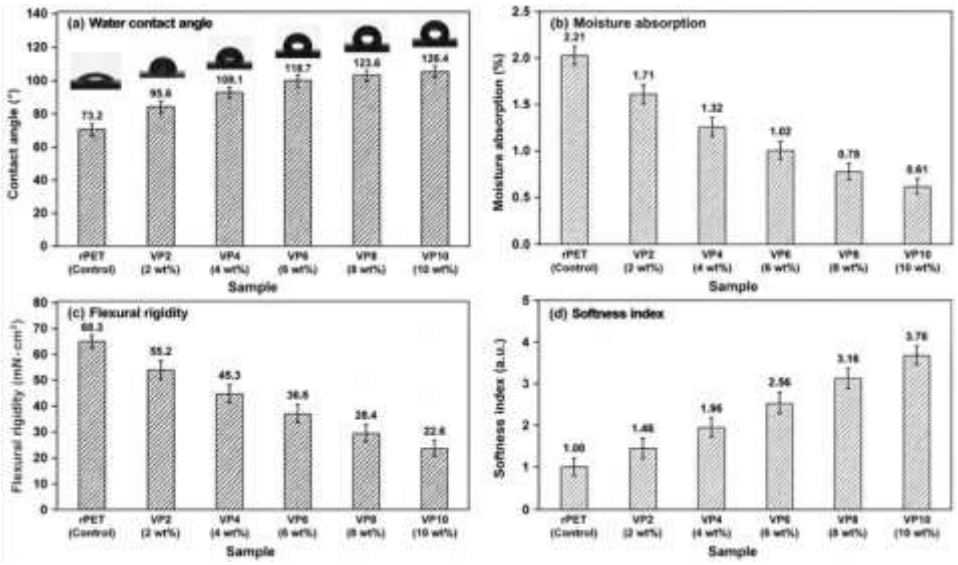

Figure 7. Surface and comfort-related properties of unmodified recycled poly(ethylene terephthalate) (rPET) and vinyl-terminated PDMS-modified rPET fibers (VP2–VP10): (a) water contact angle, (b) moisture absorption, (c) flexural rigidity, and (d) softness index.

Structure–Property Relationship

The collective characterization results demonstrate that the enhanced performance of the modified recycled PET fibers arises from the synergistic effects of reactive compatibilization and silicone modification. During reactive extrusion, the peroxide initiator generated free radicals that promoted grafting reactions between the vinyl groups of PDMS and the recycled PET chains, while the epoxy-functional chain extender repaired chain scission defects produced during PET recycling. These simultaneous reactions produced a chemically integrated polymer network in which flexible siloxane segments were incorporated into the polyester matrix without disrupting the fundamental PET backbone. The FTIR spectra confirmed the formation of silicone-containing structures through the appearance of characteristic Si–O–Si and Si–CH_3 absorption bands, while the persistence of the PET carbonyl band demonstrated that the polyester framework remained intact after modification.

The improved compatibility achieved through reactive processing was reflected in the fiber morphology. SEM observations showed smooth fiber surfaces, homogeneous cross-sections, and the absence of observable phase separation, indicating efficient dispersion of the silicone phase throughout the polyester matrix. The restoration of molecular weight by the chain extender enhanced melt strength and stabilized polymer flow during melt spinning, thereby reducing melt fracture and producing fibers with uniform diameters and fewer structural defects. This homogeneous morphology minimizes stress concentration sites and provides a stable microstructural foundation for improved mechanical performance.

Thermal analysis further demonstrated that PDMS modified the internal structure of recycled PET without compromising its thermal stability. DSC measurements revealed only minor changes in glass transition and

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melting temperatures but a gradual reduction in crystallinity as PDMS concentration increased. The flexible siloxane chains partially restricted the regular packing of PET molecules during crystallization, producing a less rigid polymer structure with greater molecular mobility. At the same time, TGA showed progressive increases in both the onset degradation temperature and maximum decomposition temperature together with higher residual char yields. These improvements are attributed to the thermally stable Si–O–Si backbone and the formation of silica-rich residues during thermal decomposition, which delayed degradation and enhanced resistance to thermo-oxidative attack.

The structural changes produced by reactive silicone modification translated directly into improved mechanical performance. The moderate reduction in crystallinity and stiffness increased chain mobility, allowing the fibers to deform more readily under applied stress while maintaining adequate tensile strength. Consequently, elongation at break increased substantially, whereas Young's modulus and flexural rigidity decreased progressively with increasing PDMS content. Simultaneously, the chemically anchored siloxane segments acted as reversible elastic components that absorbed and released deformation energy during loading and unloading. This mechanism accounts for the marked improvements in compression recovery and resilience after repeated compression cycles, demonstrating the ability of the modified fibers to resist permanent deformation under cyclic loading.

The incorporation of PDMS also significantly altered the surface characteristics of the recycled PET fibers. The migration of methyl-rich siloxane segments to the fiber surface reduced surface free energy, resulting in higher water contact angles and lower moisture absorption. The improved hydrophobicity enhances dimensional stability under humid conditions and reduces susceptibility to moisture-induced deterioration during service. In addition, the lubricating nature of the silicone segments reduced inter-fiber friction, producing lower flexural rigidity and higher softness indices. These characteristics contribute to improved bulk recovery, easier fiber opening during processing, and enhanced tactile comfort in cushioning applications.

Although increasing PDMS concentration generally improved flexibility, resilience, and hydrophobicity, the results indicate that these benefits reached an optimum at 6–8 wt.% PDMS. At these concentrations, the fibers exhibited excellent compression recovery, high resilience, low flexural rigidity, and superior softness while maintaining relatively high tensile strength and satisfactory thermal stability. Increasing the PDMS content to 10 wt.% produced only marginal improvements in surface properties but resulted in a more noticeable reduction in crystallinity and tensile strength. These findings suggest that excessive incorporation of flexible silicone segments begins to reduce stress transfer efficiency within the polyester matrix, limiting the overall performance gains.

Overall, the structure–property relationship established in this study demonstrates that reactive silicone modification is an effective strategy for upgrading post-consumer recycled PET into high-value functional fibers. The combination of improved melt processability, enhanced flexibility, excellent elastic recovery, superior moisture resistance, and adequate thermal and mechanical stability provides a comprehensive performance profile that is highly suitable for sustainable furniture cushion-filling materials. The optimum PDMS concentration of 6–8 wt.% offers the best balance between structural integrity and functional performance, highlighting the potential of reactive polysiloxane modification for the development of advanced recycled polyester fibers with enhanced commercial value.

Long-Term Durability under Accelerated Aging

The long-term durability of the fibers was evaluated by cyclic compression and accelerated humidity aging to simulate the service conditions encountered in furniture cushioning applications. **Figure 8a** presents the compression recovery after repeated loading, while **Figure 8b** summarizes the retention of mechanical properties following humidity exposure.

The PDMS-modified fibers exhibited significantly higher compression recovery than the unmodified recycled PET fibers throughout the fatigue test. After 10,000 compression cycles, samples containing intermediate PDMS contents (VP4–VP8) retained the highest thickness recovery and exhibited only minimal permanent deformation. In contrast, the unmodified recycled PET fibers showed a progressive reduction in recovery with

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increasing compression cycles, indicating greater structural fatigue and irreversible deformation. The superior cyclic performance of the modified fibers demonstrates that reactive incorporation of PDMS effectively enhances the elastic stability of recycled PET under repeated mechanical loading.

Accelerated humidity aging produced only a slight reduction in the tensile strength and compression recovery of the PDMS-modified fibers, whereas the recycled PET fibers exhibited noticeably greater moisture absorption and a corresponding decline in mechanical performance. The contact angle of the modified fibers remained relatively high after humidity exposure, indicating that the hydrophobic surface characteristics introduced by PDMS were largely retained throughout the aging period. Consequently, the modified fibers maintained their bulk structure and cushioning efficiency more effectively than the untreated recycled PET fibers.

The improved durability of the PDMS-modified fibers can be attributed to the unique molecular characteristics of the siloxane backbone. The highly flexible Si–O–Si chains undergo reversible deformation during repeated compression, allowing mechanical stresses to be distributed more uniformly throughout the polymer network. This stress redistribution reduces localized stress concentration and suppresses the irreversible molecular rearrangement that normally occurs in recycled PET during cyclic loading. Furthermore, the reactive grafting of PDMS onto the PET backbone provides stronger interfacial interactions than those obtained through physical blending alone, thereby improving the structural integrity of the modified fibers during prolonged mechanical deformation.

The superior humidity resistance of the modified fibers is primarily associated with the intrinsically low surface energy of PDMS. The outward orientation of methyl groups on the fiber surface increases hydrophobicity and reduces moisture penetration into the polyester matrix. As a result, hydrolytic degradation of the polymer chains is minimized, enabling the fibers to retain their mechanical integrity and elastic recovery after prolonged exposure to humid environments. The lower moisture uptake also contributes to improved dimensional stability and reduced susceptibility to environmental aging.

The combination of enhanced fatigue resistance, improved compression recovery, and superior humidity stability demonstrates that reactive modification with vinyl-terminated PDMS significantly improves the long-term durability of recycled PET fibers. These results suggest that the modified fibers possess the resilience and environmental stability required for repeated loading conditions encountered in furniture cushions, pillows, mattresses, and other fiber-filled products. Consequently, the accelerated aging evaluation provides strong evidence that reactive silicone modification not only enhances the initial mechanical performance of recycled PET fibers but also substantially extends their functional service life under simulated end-use conditions.

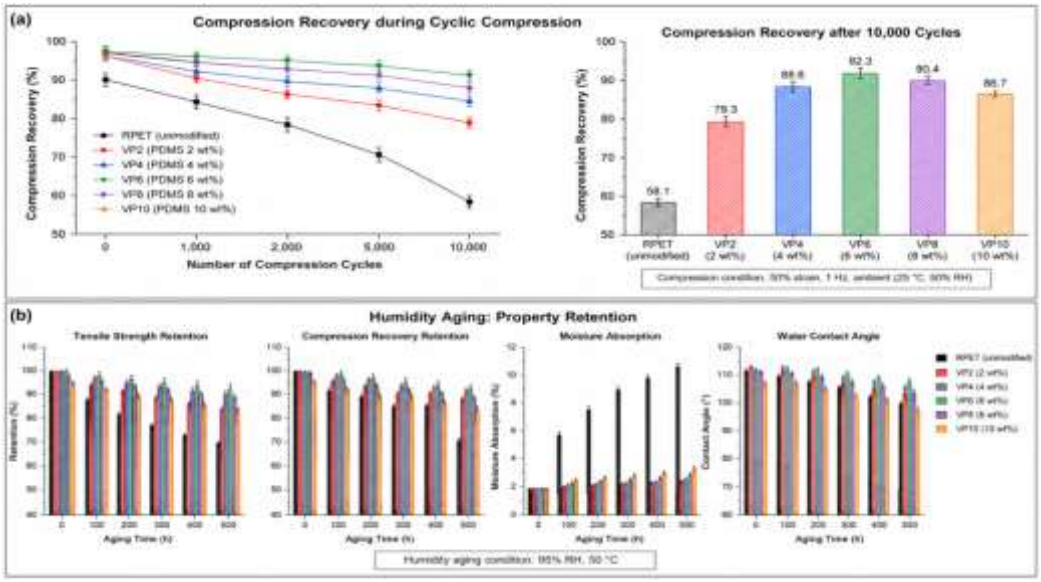

Figure 8. Long-term durability performance of recycled PET and PDMS-modified fibers under accelerated aging: (a) compression recovery during cyclic compression and residual recovery after 10,000 cycles; (b)

retention of tensile strength, compression recovery, moisture absorption, and water contact angle during accelerated humidity aging (95% RH, 50 °C).

CONCLUSION

This study demonstrated that reactive modification of recycled poly(ethylene terephthalate) (rPET) with vinyl-terminated polydimethylsiloxane (PDMS) is an effective approach for producing high-performance silicone-modified fibers suitable for cushion-filling applications. FTIR analysis confirmed the successful incorporation of PDMS into the polyester matrix through the appearance of characteristic Si–O–Si and Si–CH_3 absorption bands while preserving the fundamental PET backbone. SEM observations revealed smooth fiber surfaces, uniform diameters, and homogeneous cross-sections, indicating excellent melt processability and effective compatibility between the recycled PET and silicone modifier.

Thermal characterization showed that PDMS modification had only a minor influence on the glass transition and melting temperatures but moderately reduced crystallinity, thereby increasing molecular flexibility without compromising the crystalline PET framework. Thermogravimetric analysis further demonstrated improved thermal stability through higher degradation temperatures and increased residual char yield, confirming the beneficial contribution of the thermally stable siloxane structure.

Mechanical and functional evaluations revealed that reactive PDMS incorporation significantly enhanced fiber flexibility, elongation at break, compression recovery, resilience, softness, and hydrophobicity while maintaining adequate tensile strength for cushioning applications. The improved elastic recovery and reduced flexural rigidity were attributed to the flexible siloxane backbone, whereas the increased water contact angle and reduced moisture absorption resulted from the low surface energy of the silicone segments. Collectively, these improvements overcome the inherent brittleness and limited resilience of conventional recycled PET fibers.

Among the investigated formulations, 6–8 wt.% vinyl-terminated PDMS provided the optimum balance between tensile strength, flexibility, resilience, thermal stability, and moisture resistance. At this composition, the fibers exhibited excellent recovery after repeated compression, high softness, and superior dimensional stability, making them particularly attractive for furniture cushion filling and other resilient nonwoven applications.

Overall, the findings demonstrate that reactive silicone modification provides a practical and sustainable route for upgrading post-consumer PET waste into value-added functional fibers with enhanced mechanical, thermal, and surface properties. This strategy not only increases the commercial value of recycled polyester but also contributes to circular materials utilization by expanding the application potential of recycled PET in high-performance cushioning products. Future studies should investigate the long-term durability of the modified fibers under accelerated aging conditions and evaluate their performance in industrial-scale melt-spinning and commercial furniture manufacturing.

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