Effects of Aging in IRM 902 on the Mechanical Behavior of Injection- and Extrusion-Processed Thermoplastic Polymers

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Keywords: *in situ behavior; punch-shear testing; thermoplastic polymers; fabrication processes; environmental aging*

1. Abstract

Qualifying the reliability of thermoplastic polymers in harsh environments representative of oil and gas operations is critical for ensuring long-term structural performance. A recently developed in situ punch-shear testing device has been successfully applied to establish experimental correlations between tensile properties (ASTM D638) and shear properties (ASTM D732) for polyethylene of raised temperature (PERT) under dry conditions, and has subsequently been extended to thermoplastic polymers aged in deionized (DI) water. Previous studies have shown that selected thermoplastics, including polyphenylene sulfide, PERT, and polypropylene, exhibit either mild or negligible fluid uptake when exposed to DI water.

The present work extends this investigation to the same three thermoplastic polymers fabricated by injection molding and extrusion, with aging conducted in IRM 902 standardized mineral reference oil. The objectives of this study are threefold: (i) to assess the influence of fabrication method on measured mechanical properties, (ii) to compare experimentally established correlations between tensile and shear responses across different polymer grades prior to fluid exposure at evaluated temperatures, and (iii) to evaluate the reversibility of tensile and shear properties following saturation aging in IRM 902 at different temperatures.

Specimens were manufactured using injection molding and extrusion and subjected to controlled aging protocols until saturation was achieved. Fluid uptake and saturation behavior were monitored gravimetrically at regular intervals. Tensile testing was performed using a universal testing machine. Strain was measured using an optical extensometer. Tests were conducted at room temperature and elevated temperatures of 60 and 95 °C using an environmental oven mounted on the testing frame. Shear behavior was evaluated using a modular in situ punch-shear device. For in situ shear testing, specimens were immersed in the aging fluid at 95 °C and 1.1 bar.

The ongoing experimental program is expected to provide insight into the coupled effects of fabrication route, fluid aging, and testing methodology on the mechanical performance of thermoplastic polymers and their composites, supporting improved durability assessment for service in hydrocarbon-rich environments.

2. Introduction

Thermoplastic polymers are widely used in engineering applications because they offer ease of processing, durability, corrosion resistance, design flexibility, cost-effectiveness, recyclability, and broad material availability [1,2]. Common fabrication routes include extrusion, injection molding, compression molding, thermoforming, and blow molding. The selected processing route can influence crystallinity, morphology, dimensional stability, and mechanical or thermal performance [1,2]. Beyond general applications in packaging, automotive components, medical products, aerospace-related structures, and lightweight functional materials [1–4], thermoplastic-based pipe systems are increasingly used or evaluated for oil, gas, and water transportation because of their corrosion resistance, flexibility, and suitability for spoolable installation in both onshore and offshore environments [5,6]. In these applications, material selection depends strongly on the transported fluid, operating temperature, pressure, and long-term environmental exposure, making fluid-aging behavior and mechanical property retention important considerations [5,6]. Processing and manufacturing conditions, including heating, consolidation pressure, consolidation speed, cooling history, and bonding quality, can also influence the final structure and performance of thermoplastic pipe materials and related composite systems [6].

Among thermoplastic materials, polypropylene (PP), polyethylene of raised temperature resistance (PERT), and polyphenylene sulfide (PPS) represent different performance levels relevant to pipe-related and oilfield environments. PP and PERT are polyolefin-based polymers, while PPS is a high-performance engineering aromatic sulfide polymer selected for improved thermal and chemical resistance. Previous studies have shown that thermoplastic properties are strongly affected by processing route, crystallinity, pressure, molding time, cooling rate, and environmental history [7–9]. Fluid exposure is particularly important because polymer properties can change after aging through mechanisms such as plasticization, leaching, and chain scission [10]. In addition, conventional ex situ mechanical testing after aging may underestimate fluid-induced property changes when absorbed fluid desorbs during sample handling or testing in a dry environment [10]. IRM 902 oil is a standardized reference immersion oil defined in ASTM D5964 as a replacement for ASTM No.2 oil used in ASTM D471 liquid-immersion testing [11]. It has been used to evaluate heat and oil resistance of thermoplastic vulcanizates by measuring retained tensile properties and elongation at break after elevated-temperature immersion [12]. However, limited information is available on the comparative IRM 902 aging response of representative thermoplastics such as PP, PERT, and PPS, particularly when both fabrication route and mechanical testing method are considered.

Therefore, this study investigates the IRM 902 aging behavior of PP, PERT, and PPS fabricated by injection molding and extrusion. The effects of fabrication method, testing temperature, and IRM 902 aging on mechanical properties were evaluated using mass uptake measurements, chemical structure analysis, tensile testing, and punch-shear testing. The results are intended to clarify how material class and processing route influence mechanical property retention and durability of thermoplastic polymers under hydrocarbon-rich service conditions.

3. Materials and methods

3.1. Materials and specimen fabrication

For the three thermoplastic polymers, PP, PERT, and PPS, specimens were prepared using both injection molding and extrusion. Injection-molded specimens were produced using an Arburg injection molding machine (Lossburg, Germany), with tensile coupons molded directly and punch-shear coupons cut from injection-molded disks. Extruded specimens were prepared from fabricated plates, from which both tensile and punch-shear coupons were water-jet cut.

3.2. Aging and characterization methods

IRM 902 aging was conducted to evaluate fluid uptake and mechanical property retention of the thermoplastic polymers under service-relevant oil exposure. Specimens were immersed in IRM 902 at room temperature (RT) and 95 °C. For each material and specimen type, ten coupons were immersed in the same glass jar, including five injection-molded coupons and five extruded coupons. Mass uptake was monitored gravimetrically at regular intervals. Before each measurement, specimens were removed from the fluid and first wiped with a dry cloth to remove surface oil. A small amount of alcohol was then sprayed onto the cloth, and each coupon was wiped twice more to remove residual surface oil. The specimens were then weighed using a balance (Cgoldenwall Model HZ 5002, Hangzhou, China) and returned to the aging environment. The percentage mass uptake was calculated from the initial and aged specimen masses using Equation (1):

$$M_t = \frac{m_t - m_0}{m_0} \times 100 \quad (1)$$

where M_t is the percentage mass uptake, m_0 is the initial dry mass, and m_t is the mass after aging time *t*. At each measurement time, the reported mass uptake was averaged from five coupons with the same material, fabrication method, and specimen type.

Tensile testing was conducted based on ASTM D638 [13] using a universal testing machine (Instron, Model 5966, Norwood, MA, USA) equipped with an optical extensometer (Epsilon ONE, Jackson, WY, USA). Type I tensile specimens were tested at a constant crosshead speed of 50 mm/min, with strain measured over a 50 mm gauge length. For pristine specimens, tests were performed at room temperature (RT), 60 °C, and 95 °C, while aged specimens were tested at RT and 95 °C. Elevated-temperature tests were conducted using an environmental chamber (Instron A351-36, Norwood, MA, USA) mounted on the testing frame, with specimens allowed to equilibrate at the target temperature before testing.

Punch-shear measurements were conducted based on ASTM D732 [14] using the Instron Model 5966 machine fitted with compression platens at a constant crosshead speed of 1.25 mm/min. Circular punch-shear coupons had an outside diameter of 50.8 ± 1 mm and a concentric hole diameter of 11 ± 0.5 mm. Detailed information on the in situ punch-shear device design and coupon installation procedure has been reported previously [10]. For in situ testing, the coupon was immersed in IRM 902 inside the punch-shear device, and an estimated pressure of 5 psi was applied to maintain fluid contact around the coupon during testing. Figure 1 shows the

experimental setup used for in situ punch-shear testing at 95 °C. Due to the high viscosity of IRM 902 at RT, in situ punch-shear testing at RT was not feasible.

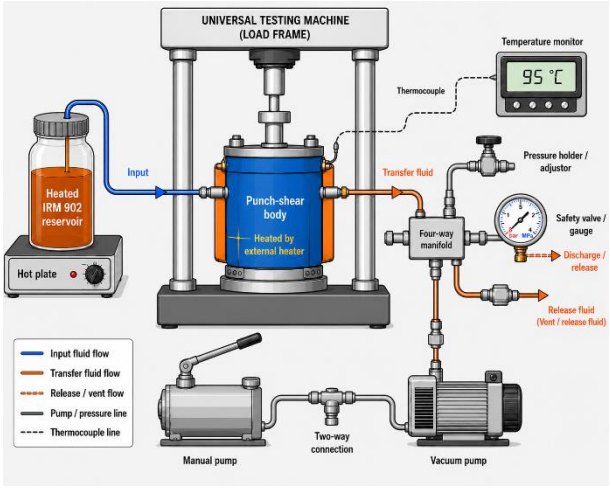

Figure 1. Schematic of the in situ punch-shear testing setup for thermoplastic coupons immersed in IRM 902 at 95 °C, including the heated fluid reservoir, pressure-control system, and temperature monitoring system.

For punch-shear experiments, stress and apparent shear strain were calculated from the raw load-displacement data using Equations (2) and (3):

$$\tau = \frac{F}{\pi D a} \tag{2}$$

where τ is the punch-shear stress, F is the applied load, D is the punch diameter, 25.4 mm, and a is the specimen thickness.

$$\gamma_{app} = \frac{d}{a} \tag{3}$$

where γ_{app} is the apparent shear strain and d is the punch displacement.

Fourier-transform infrared spectroscopy (FTIR) was conducted using an IRSpirit FTIR spectrophotometer with attenuated total reflectance (ATR) capability (Shimadzu, Kyoto, Japan). Spectra were collected from pristine specimens and specimens aged in IRM 902 at 95 °C to assess possible aging-induced chemical changes. Differential scanning calorimetry (DSC, DSC Model 1, Mettler Toledo, Columbus, OH, USA) was performed under nitrogen flow of 50 mL/min. Samples were held at 25 °C for 3 min, heated at 10 °C/min to 230 °C for PP and PERT, or 330 °C for PPS, held for 3 min, cooled to 25 °C at 10 °C/min, and reheated to the same target temperature at 10 °C/min. DSC was used to compare melting, crystallization, and thermal-transition behavior before and after aging.

4. Results and discussion

4.1. Mass uptake at room temperature and 95 °C

Figure 2 shows the mass uptake of PP, PERT, and PPS coupons aged in IRM 902 at RT and 95 °C. At RT, all materials showed low uptake, generally below 1%, indicating limited oil absorption under ambient conditions. The overall uptake followed the trend PERT > PP > PPS. PPS showed values close to zero, with small fluctuations and occasional negative values attributed to experimental scatter associated with surface cleaning and gravimetric measurement. At 95 °C, the uptake response became more pronounced and followed the trend PP > PERT > PPS. PP exhibited the highest uptake and continued to increase over the tested aging period, suggesting slower diffusion toward saturation, a higher equilibrium uptake, or possible surface/morphology-related effects. In contrast, PERT showed faster initial uptake and reached an apparent saturation level earlier than PP, at approximately 11%. PPS remained close to zero at both RT and 95 °C, indicating strong resistance to IRM 902 absorption. The curves that stopped before the final aging time indicate coupon groups that had reached saturation and were subsequently removed for testing, whereas the curves continuing to the final time indicate groups that had not yet fully saturated and remained under aging.

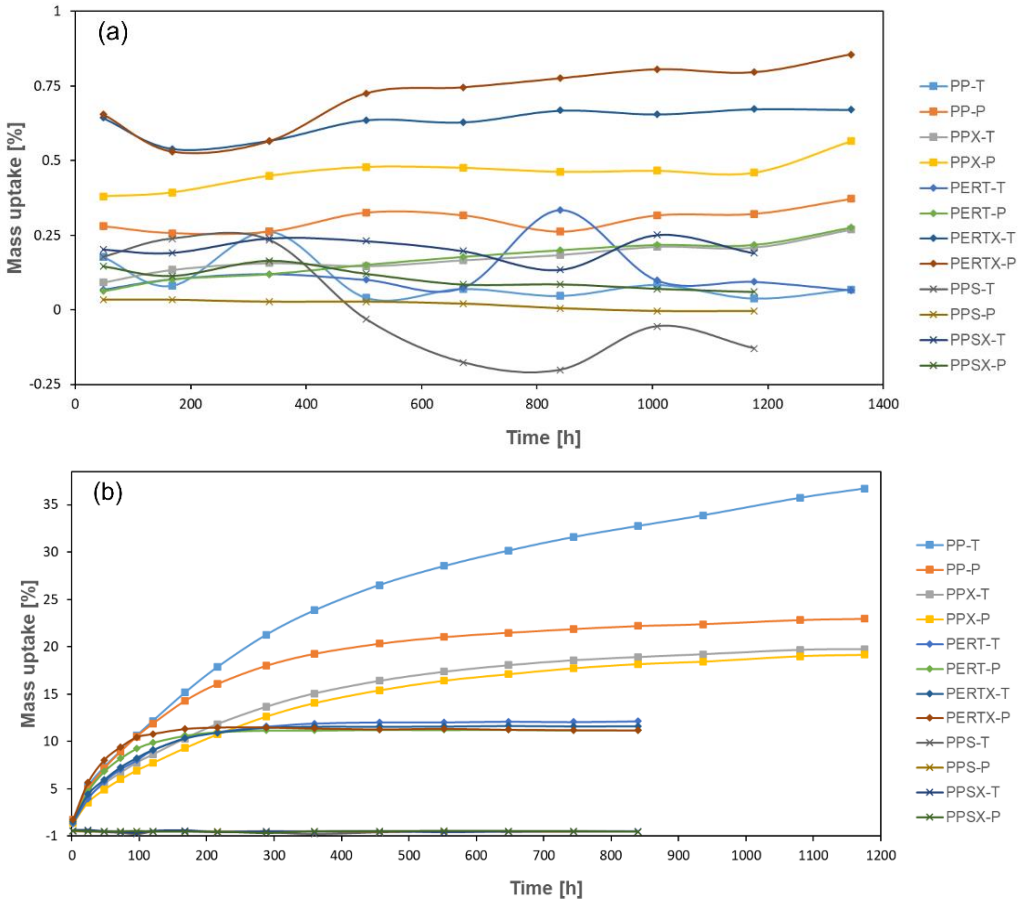

Figure 2. Mass uptake for PP, PERT, and PPS coupons aged in IRM 902 at (a) RT and (b) 95 °C. Labels with "X" denote extruded specimens (as opposed to injection-molded), and "T" and "P" denote tensile and punch-shear coupon geometries, respectively.

Differences between fabrication methods and coupon geometries were also observed, suggesting that crystallinity, morphology, surface condition, and exposed edge area may influence fluid uptake and should be considered when interpreting the subsequent mechanical-aging results. For example, at RT, extruded PERT punch-shear coupons showed higher uptake than the corresponding tensile coupons, while for PP punch-shear coupons, extruded specimens showed higher absorption than injection-molded specimens. In addition, localized blistering was observed on injection-molded PERT coupons after aging at 95 °C, as shown in Figure 3. Although this observation was made for PERT rather than PP, it indicates that local surface damage or fluid-rich regions can occur during elevated-temperature oil aging. Similar localized swelling, blistering, or surface-retention effects may partly explain the unusually high uptake measured for selected PP coupon groups, particularly the injection-molded tensile specimens, which reached approximately 36% after 1,176 h of aging at 95 °C.

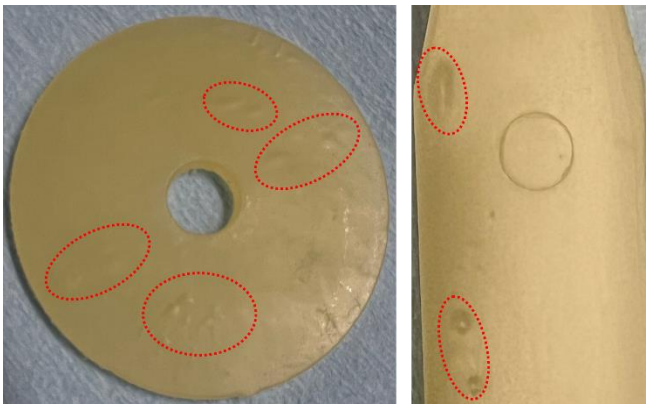

Figure 3. Representative surface blistering observed on injection-molded PERT coupons after IRM 902 aging at 95 °C (left: punch-shear coupon, right: tensile coupon). Red dashed circles indicate localized blistered regions.

4.2. Mechanical behavior at different testing temperatures

Figure 4 compares the tensile and punch-shear properties of pristine PP, PERT, and PPS specimens tested at RT, 60 °C, and 95 °C. For both testing methods, all materials showed a clear reduction in modulus and yield strength with increasing temperature, reflecting thermally induced softening of the thermoplastic matrices. PPS exhibited the highest modulus and yield strength over the full temperature range, followed by PP and PERT. This trend was observed in both tensile and punch-shear measurements, indicating that the punch-shear method can capture the relative temperature-dependent mechanical response among the investigated materials.

A positive correlation was observed between tensile modulus and apparent shear modulus, as shown in Figure 4(a). The overall linear fit gave an R^2 value of 0.860, indicating a reasonable correlation across different materials, fabrication routes, and testing temperatures. However, the modulus correlation showed greater scatter than the strength correlation. This is expected because the apparent shear modulus obtained from punch-shear testing is more sensitive to local deformation constraint, fixture compliance, specimen geometry, and temperature-dependent viscoelastic effects. Therefore, the punch-shear modulus should be interpreted as an apparent modulus rather than a direct replacement for tensile modulus.

In contrast, the yield-strength correlation was much stronger, as shown in Figure 4(b), with an R^2 value of 0.989. This indicates that punch-shear yield strength follows tensile yield strength closely across the investigated materials and temperatures. The stronger correlation suggests that the yield point is less affected by compliance and local constraint than the initial stiffness response. Therefore, punch-shear testing provides a reliable comparative method for evaluating temperature-dependent yield behavior, particularly when conventional tensile testing is difficult under in situ fluid-aging conditions.

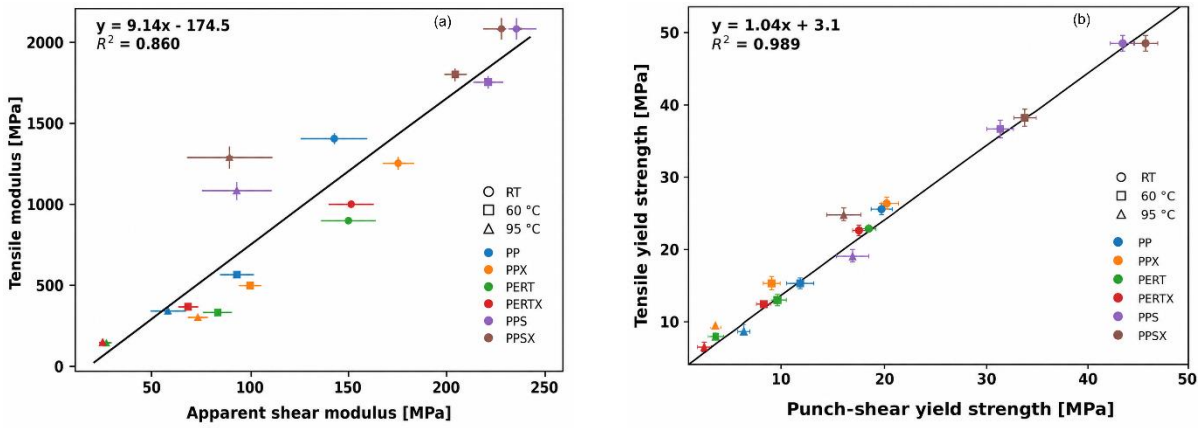

Figure 4. Correlation between tensile and punch-shear properties of pristine thermoplastic polymers tested at RT, 60 °C, and 95 °C: (a) modulus and (b) yield strength. Colors indicate material/fabrication route, symbols indicate testing temperature, and error bars represent standard deviation.

4.3. Effects of IRM 902 aging on mechanical, chemical and thermal properties

Figure 5 shows the mechanical property retention of PPS/PPSX and PERT/PERTX after aging in IRM 902 at 95 °C. The aging response was strongly material dependent. PPS-based specimens showed excellent mechanical stability, with punch-shear modulus and yield-strength retention close to or above 100%. The tensile modulus and tensile yield strength of PPS and PPSX also increased after aging, reaching approximately 123–139% retention. This increase may be attributed to physical annealing, structural relaxation, or further crystallization during prolonged exposure at 95 °C. This interpretation is consistent with the very low mass uptake of PPS in Figure 2, suggesting limited oil absorption and strong resistance to IRM 902 aging. In contrast, PERT-based specimens showed clear degradation in tensile stiffness after aging. The tensile modulus retention decreased to approximately 74% for PERT and 60% for PERTX, while tensile yield-strength retention remained higher, at approximately 84% and 81%, respectively. This indicates that stiffness was more sensitive to IRM 902 aging than yield strength. The reduced tensile modulus is consistent with the higher fluid uptake of PERT-based specimens, where absorbed oil may cause swelling, plasticization, or changes in crystalline morphology. The punch-shear results showed smaller reductions than tensile testing, especially for yield strength, suggesting that localized punch-shear deformation is less sensitive to bulk stiffness loss than uniaxial tensile loading.

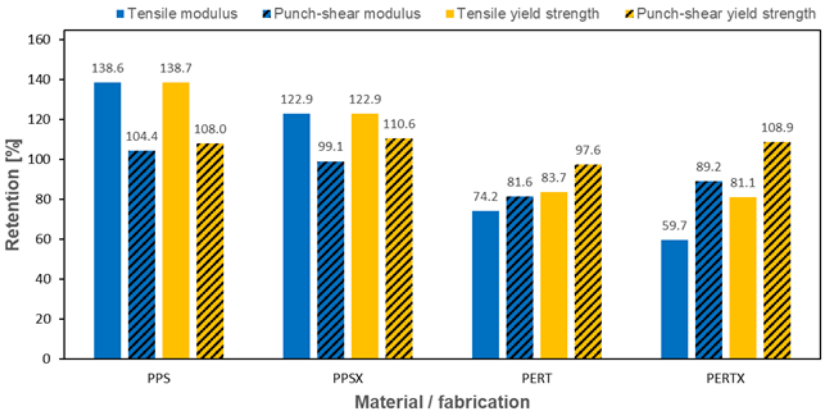

Figure 5. Mechanical property retention of PPS, PPSX, PERT, and PERTX after IRM 902 aging at 95 °C. Retention was calculated relative to the corresponding unaged baseline tested at 95 °C.

Figure 6 compares the FTIR spectra of PERT/PERTX and PPS/PPSX before and after IRM 902 aging at 95 °C. For PERT/PERTX, the main absorption bands associated with CH_2 stretching, bending, and rocking remained at similar positions after aging. Similarly, PPS/PPSX retained the characteristic aromatic-ring and C–S-related absorption bands. No obvious new absorption bands were detected after aging, suggesting that IRM 902 exposure at 95 °C did not cause measurable chemical degradation under the present conditions.

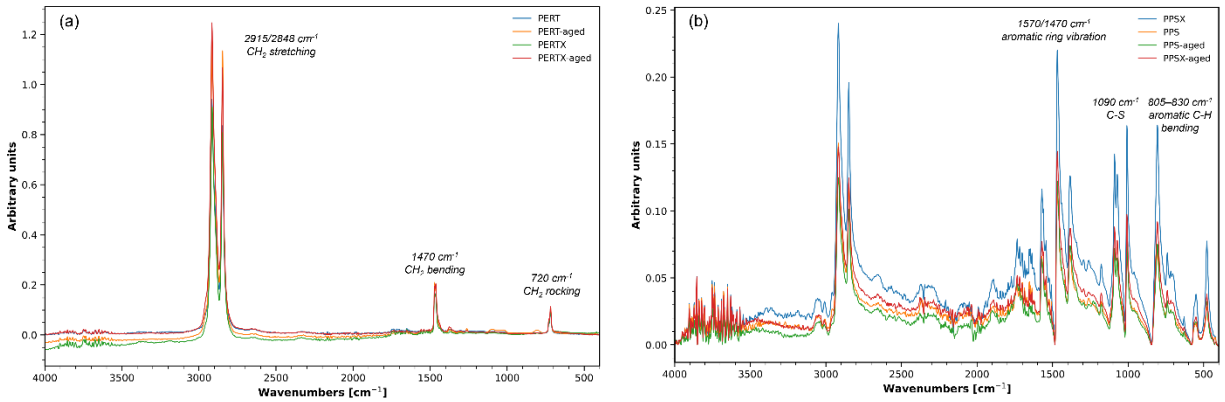

Figure 6. FTIR spectra of PERT/PERTX and PPS/PPSX before and after IRM 902 aging at 95 °C: (a) PERT and PERTX, and (b) PPS and PPSX.

The DSC results in Table 1 further support that the aging response was mainly associated with physical or morphological changes rather than major chemical changes. For PERT, the first-heating melting temperature and melting enthalpy remained nearly unchanged after aging, while the cooling crystallization enthalpy decreased by approximately 18%. PERTX showed a more pronounced reduction in first-heating melting enthalpy, approximately 14%, together with a similar decrease in crystallization enthalpy. These changes are consistent with the lower tensile modulus retention of PERTX and suggest aging-related changes in crystalline morphology or effective crystallizable fraction. For PPS and PPSX, the melting and crystallization temperatures changed only slightly after aging, with T_m shifts within approximately 4 °C and T_c shifts within approximately 3 °C. The disappearance of the cold crystallization feature observed in baseline PPS further suggests that aging at 95 °C may have promoted structural relaxation or additional ordering

before DSC testing. Together with the FTIR results, these observations indicate that the mechanical-aging responses were mainly related to oil uptake, plasticization, and morphology changes rather than obvious chemical degradation.

Table 1. DSC thermal transition parameters of PERT, PERTX, PPS, and PPSX before and after IRM 902 aging at 95 °C. Values were obtained from the first heating and cooling scans, and enthalpies are reported as absolute values.

Material	Fabrication	Condition	T_m, 1st heating [°C]	ΔH_m, 1st heating [J/g]	T_c, cooling [°C]	ΔH_c, cooling [J/g]
PERT	Injection	Baseline	129.0	146.2	112.3	184.7
PERT	Injection	Aged at 95 °C	130.6	147.4	111.1	151.4
PERTX	Extrusion	Baseline	133.4	172.2	115.7	175.8
PERTX	Extrusion	Aged at 95 °C	127.7	148.5	114.4	143.6
PPS	Injection	Baseline	281.5	22.0	230.4	49.9
PPS	Injection	Aged at 95 °C	277.3	21.7	229.8	36.5
PPSX	Extrusion	Baseline	278.2	27.3	225.8	33.4
PPSX	Extrusion	Aged at 95 °C	280.3	16.2	228.8	35.7

5. Conclusions

This study investigated the effects of fabrication route, testing temperature, and IRM 902 aging on the mechanical, chemical, and thermal behavior of PP, PERT, and PPS thermoplastic polymers. Mass uptake results showed limited absorption at RT, generally below 1%, while aging at 95 °C produced a stronger material-dependent response. At 95 °C, the uptake followed the trend PP > PERT > PPS, with PPS showing negligible mass change and strong resistance to IRM 902 absorption.

For pristine specimens, both tensile and punch-shear properties decreased with increasing testing temperature, reflecting thermal softening of the thermoplastic materials. PPS exhibited the highest modulus and yield strength over the investigated temperature range, followed by PP and PERT. A reasonable correlation was observed between tensile modulus and apparent shear modulus, while a stronger correlation was obtained between tensile and punch-shear yield strength. This indicates that punch-shear testing is particularly effective for comparing yield behavior across different thermoplastic materials and testing temperatures.

After IRM 902 aging at 95 °C, PPS and PPSX retained or increased their mechanical properties, likely due to physical annealing, structural relaxation, or additional ordering during elevated-temperature exposure. In contrast, PERT and PERTX showed reduced tensile stiffness after aging, consistent with their higher oil uptake and possible plasticization or morphology changes. FTIR results showed no obvious new absorption bands after aging, while DSC results indicated mainly physical or morphological changes rather than clear chemical degradation. Overall, PPS showed the greatest resistance to IRM 902 aging, whereas PERT was more sensitive to elevated-

temperature oil exposure. Ongoing tests on PP aged at 95 °C and PP/PERT aged at RT are expected to further complete the dataset and strengthen the comparison of fabrication-route effects, aging temperature, and long-term mechanical property retention.

6. References

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