Mechanical and Rheological Behavior of High-Density Polyethylene under the Reversible Crosslinking Method and Comparison to a Conventional Method

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ABSTRACT

This study investigates a Reversible Crosslinking (RXR) technique that enables the melt-state crosslinking of High-Density Polyethylene (HDPE). Reactive extrusion was carried out using a Brabender mixer, after which the RXR-crosslinked HDPE was ground in the solid state and subsequently reprocessed by compression molding. Dynamic Rheological Analysis (DRA) was employed to evaluate the crosslinking efficiency and viscosity variation through torque time curves. The Melt Flow Index (MFI) and gel content

measurements were correlated with the DRA results after the second processing cycle. Differential Scanning Calorimetry (DSC), Wide-Angle X-ray Scattering (WAXS), Fourier-Transform Infrared Spectroscopy (FTIR), and mechanical measurements were conducted after the second compression molding. The RXR methodology provided a tunable rheological response, offering clear advantages in processing versatility and industrial optimization. FTIR confirmed the formation of new interchain C–S bonds, demonstrating effective bridging between HDPE main chains. WAXS and DSC revealed a slight reduction in crystallinity, whereas Thermogravimetric Analysis (TGA) indicated enhanced thermal stability in the early stages of decomposition. Mechanical tests showed a strong correlation between the crosslinking degree and crystallinity with both Young's modulus and impact strength, suggesting that higher structural organization improves stiffness and impact resistance.

Keywords-reactive extrusion; reversible crosslinking; TGA; microstructure; HDPE

I. INTRODUCTION

Crosslinking is a widely employed strategy for the chemical modification of polymer structures and is used to enhance the thermal, mechanical, and rheological properties of polymers [1-4]. HDPE stands out due to its exceptional chemical resistance, mechanical strength, and cost-effectiveness, making it widely used in water distribution networks [5]. However, the inherent thermoplastic nature of HDPE limits its structural stability at elevated temperatures and in demanding applications, while it also reduces its recyclability. These shortcomings have motivated extensive research into crosslinking methodologies for improving the thermal and mechanical performance of HDPE [6].

A variety of crosslinking techniques have been investigated, including peroxide-induced crosslinking [7, 8], Diels–Alder reactions [9, 10], silane crosslinking [11], and irradiation methods [9]. While these approaches successfully enhance material performance, they generally result in irreversible covalent networks, thereby restricting recyclability and sustainability [12, 13]. For example, conventional peroxide crosslinking relies on free-radical mechanisms to form permanent covalent bonds [14], making it a popular choice for automotive and structural applications [15]. Silane crosslinking, which involves hydrolysis and condensation reactions, is widely employed in piping and insulation, whereas irradiation crosslinking, which uses high-energy radiation, is particularly valued in medical device manufacturing because of its precision and reliability [16, 17]. Despite these advantages, such methods typically lead to permanent structural modifications that hinder recycling. In response, RXR strategies have emerged as a promising alternative, offering the ability to form dynamic or reversible bonds that enable the reconfiguration of the polymer network under external stimuli such as heat, mechanical stress, or chemical agents [12]. Of particular interest is the incorporation of sulfur-based reversible bonds within the HDPE matrix. This innovative approach not only preserves critical performance characteristics but also enables the repeated modification, repair, and recycling of the polymer. Research has highlighted its potential for applications in high-performance piping and flexible electronics, underscoring both its versatility and practical relevance.

The RXR method is a promising strategy for HDPE modification, aiming to enhance its thermal and mechanical properties while allowing control over its rheological behavior. This technique relies on the dynamic nature of disulfide (S–S) bonds, which can undergo reversible cleavage and reformation (disulfide exchange) at elevated temperatures [18]. The RXR approach offers a sustainable alternative to conventional crosslinking techniques. Its effectiveness has been demonstrated in isotactic polypropylene, traditionally considered a non-crosslinkable material [19]. Moreover, RXR has been utilized as a chemical tool to control both scission and crosslinking reactions, enabling the exfoliation of raw montmorillonite clay into nanocomposite structures [20]. Furthermore, this method has been successfully extended to other members of the polyolefin family, such as polybutene-1 [21] and Low-Density Polyethylene (LDPE) [22]. Building on this foundation, the present work applies the RXR technique to HDPE, one of the most widely used and industrially significant polyolefins.

II. EXPERIMENTAL

A. Materials

HDPE (HYA 600) with a density of 0.954 g/cm^3 and a Melt Flow Index ($MFI_{2.16}$) of 0.37 g/10 min was supplied by Exxon Mobil Chemical. The peroxide used, di(tert-butyl peroxy isopropyl) benzene (DTBPIB), commercially known as Perkadox 14-40 B-gr, was manufactured by Akzo Nobel Polymer Chemicals (Amersfoort, Netherlands). Tetramethyl Thiuram Disulfide (TMTDS) was provided by Rhodia, France. Sulfur was supplied by Wuxi Huasheng, China.

B. Processing

The RXR crosslinking of HDPE was carried out in two steps: initial mixing in a Brabender plastograph, followed by reactive extrusion and then compression molding to obtain isotropic samples for tensile and impact testing. Crosslinking was initiated by the rapid shearing of HDPE with peroxide, an activator, and the accelerator TMTDS, which modulated the peroxide activity to control the macroradical generation. The polyethylene was mixed with different ratios of Crosslinking Agents (CAs) in the plastograph, which allows real-time monitoring of torque and rheological behavior during processing in the melt phase. A total of 40 g of raw material was thoroughly mixed with CA at 170 °C and a rotation speed of 30 rpm for 30 min. Two different RXR modes were applied: the first, designated as Method 1, used only peroxide and accelerator, and the second, Method 2, included sulfur in addition to the previous components. Both methods were compared to a conventional crosslinking method (C). The specific ratios of each component are detailed in Table I and are given in parts per hundred resin (phr).

TABLE I. SAMPLE COMPOSITION

Sample/ composition	Peroxide (phr)	Sulfur (phr)	Accelerator (phr)	HDPE (phr)	Code
Virgin material	-	-	-	100	PE0
Conventional crosslinking method: C	0.8	-	-	100	PEXC
RXR crosslinking method	0.8	0.4	0.2	100	PEX1
	1.2	0.6	0.3	100	PEX2

CA = 1 part peroxide + ½ part sulfur + ¼ part accelerator

C. *Characterization Techniques*

DRA was carried out using an internal mixer (Brabender Plasti-Corder, Germany) to evaluate the compounding performance. Torque-time curves were recorded for each formulation during mixing. The compounded materials were then ground into granules and used to determine the MFI. MFI measurements were performed with a CONTROLAB melt flow rate apparatus in accordance with ASTM D1238, using two different loads of 2.16 kg and 5 kg at 230 °C for 10 min. The melt flow ratio was calculated as the ratio of MFI at 5 kg to that at 2.16 kg. The gel content of the crosslinked samples was determined according to ASTM D2765 (test Method A). Thin films prepared by compression molding were further characterized by Fourier Transform Infrared Spectroscopy (FTIR) using a Perkin Elmer Spectrum spectrometer. The spectra were recorded at room temperature in the range of 4000-400 cm^{-1} with 32 scans. WAXS measurements were conducted using a Bruker D8 Advance X-ray diffractometer. The crystallinity and crystallite size of the samples were analyzed in the 2θ range of 5–50°. The thermal properties were analyzed using a Setaram Differential Scanning Calorimeter (DSC-141) by heating from 20 °C to 180 °C at a rate of 10 °C/min, holding at 180 °C for 5 min, cooling to 40 °C at 5 °C/min, and holding at 40 °C for 5 min. A second heating cycle was performed under the same conditions to eliminate the thermal history. TGA was performed on samples of approximately 7 mg, heated from 20 °C to 700 °C at a rate of 20 °C/min. The mechanical properties were evaluated using an Instron 3344 tensile testing machine with a crosshead speed of 50 mm/min. Impact strength tests were conducted according to ASTM D256–04 (Method A), using a Zwick/Roell IZOD pendulum impact tester with a 4 Joules capacity. All samples were die-cut according to ASTM D638–03 (Type V). The specimens for the mechanical tests were prepared by compression molding at 190 °C and 30 kN for 3 min using a Paul-Otto Weber press.

The PEX1 and PEX2 specimens were conditioned at 25 °C and 50 ± 5% relative humidity for 48 h prior testing. For each formulation, at least five specimens were tested. However, the PEXC specimens could not be reprocessed in the molten state for repeated measurements because of the formation of a non-reversible network after the first cycle in the Brabender mixer.

III. RESULTS AND DISCUSSION

A. *Rheological Properties*

The rheological behavior of the raw and crosslinked HDPE is shown in Figure 1. The torque–time curves, gel content, and MFI results are shown in Figures 1(a-c), respectively.

The torque–time curves show an initial minimum at point A, indicating the transition of the material into a molten state. Then, as the crosslinking reaction begins, the torque increases, reaching a peak at B, which represents the maximum reactivity of the crosslinking process for both the RXR and conventional methods. A slight decrease follows due to partial network degradation, stabilizing into a plateau at point C. This trend can also be inferred from the peak torque height, which corresponds to the maximum torque recorded.

The gel content results reveal that higher gel fractions indicate stronger crosslinking. The conventional method (PEXC) exhibits the highest gel content when using 0.8 phr of peroxide. In comparison, the RXR-crosslinked materials (PEX1 and PEX2) show moderate gel levels despite PEX2 containing 1.2 phr peroxide, reflecting the reversible nature of the RXR process.

The MFI results obtained under 2.16 kg and 5 kg loads indicate higher viscosity as a result of enhanced crosslinking. This behavior is consistent with Poiseuille flow in capillary rheometry and drag flow observed during Brabender mixing. The drop in MFI after intense shearing confirms effective network formation, demonstrating the complementary value of MFI in evaluating newly developed HDPE structures. Moreover, the gel content correlates well with the torque measurements, highlighting the influence of crosslinking on melt viscosity.

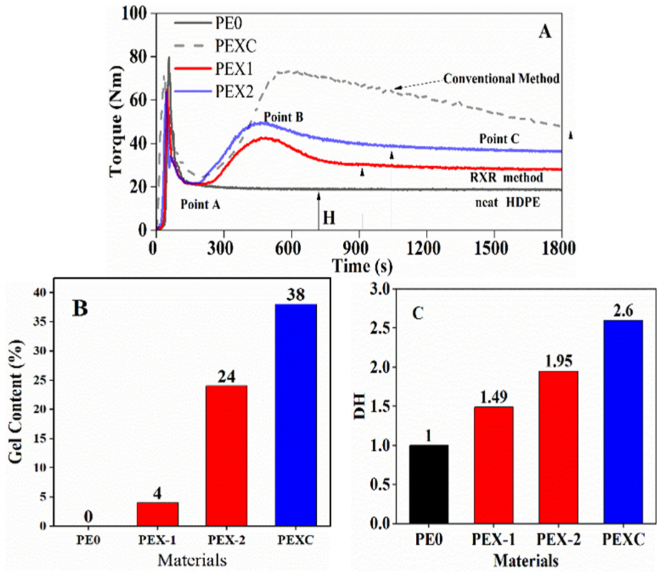

Fig. 1. Rheological behavior: (a) torque-time, (b) gel content, and (c) DH for raw HDPE and different crosslinked.

B. Structural Properties

As depicted in Figure 2(a), new peaks appear at 473 and 501 cm^{-1} in the PEX1 and PEX2 spectra, corresponding to the aryl–S–S–aryl and alkyl–S–S–alkyl bonds, respectively. In the RXR samples, PEX1 and PEX2, the coexistence of dual sulfur sources (pure sulfur and TMTDS), together with chain entanglements, facilitates the formation of both alkyl and aryl sulfur bridges. Given the smaller size of aryl groups compared to alkyl groups, aryl radicals are more likely to participate in the formation of interchain –S–S– linkages in HDPE.

A new peak at 875 cm^{-1} is observed for PEX1 and PEX2, whereas it is absent for PEXC. This peak is attributed to the formation of new interchain S–OR bonds, which likely arise from the reaction between the oxyradicals generated by the DTBPIB peroxide and the available sulfur sources. The C=S stretching band, observed at 1110 cm^{-1}, appears in both PEX1 and PEX2. Furthermore, a peak at 1155 cm^{-1}, assigned to C–N stretching, was detected in the crosslinked materials. However, both C=S and C–N bonds are characteristic of the dithiocarbamyl group. These groups may form when grafted onto lateral polymer chains or between distant chains that fail to establish disulfide (–S–S–) or carbon–carbon (C–C) bonds.

The FTIR spectrum of raw HDPE, presented in Figure 2(b), displays two peaks at 990 and 910 cm^{-1}, corresponding to terminal vinyl groups. At higher crosslinking levels, a slight increase in the intensity of the peak at 965 cm^{-1} is observed, which corresponds to trans-unsaturation (RCH=CHR) along the HDPE backbone.

The oxidative degradation of the samples was evaluated by monitoring the changes in the carbonyl absorption band (1770–1690 cm^{-1}), as illustrated in Figure 2(c). The carbonyl functional groups include ketones (1718 cm^{-1}) and esters (1740 cm^{-1}), which serve as indicators of oxidative degradation and provide insights into the extent of chemical modification within the HDPE matrix.

The Carbonyl Index (*CI*) and degree of branching are quantitative measures of the carbonyl band intensity, typically calculated as the ratio of the area under the carbonyl peak to that of a reference peak. In this study, *CI* was determined as the ratio between the area of the carbonyl peak (A_{1720}) and that of the reference peak (A_{1465}), as expressed in:

$$CI = \frac{A_{peak}}{A_{ref}} \quad (1)$$

The *CI* values of all samples, plotted as a function of the crosslinking agent content (Figure 2(c)), reveal a pronounced increase for PEXC, indicating a higher susceptibility to oxidative degradation. In contrast, PEX1 and PEX2 exhibit improved resistance to oxidation. Both the CI and degree of branching increased significantly in the case of conventional crosslinking, thus confirming the lower oxidative stability of this material. These findings highlight the advantages of the RXR approach, in which RXR not only ensures structural integrity but also enhances resistance to degradation compared to the conventional method. The improvements ranged from 35% to 61%.

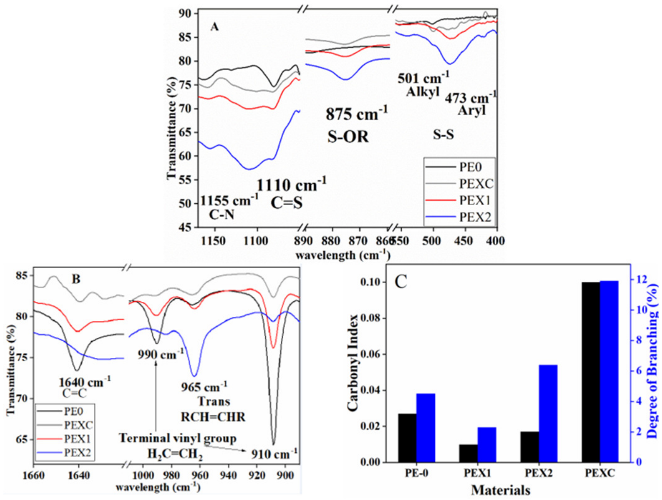

Fig. 2. (a) FTIR spectra of neat HDPE, PEXC, and RXR materials, (b) FTIR spectra of all materials, (c) CI with a degree of branching of PE0, PEXC, PEX1, and PEX2.

The WAXS results for the different crosslinked materials are presented in Figure 3. The crystallite size (L_c) of all materials was calculated using the Debye-Scherrer equation:

$$L_C = \frac{k.\lambda}{\beta \cdot \cos\theta} \quad (2)$$

where L_c is the average crystallite size, k is a constant (0.9 for polymers), λ is the X-ray wavelength (1.54 Å), β is the Full Width at Half Maximum (FWHM) of the diffraction peak intensity, and θ is Bragg's angle in radians [21].The degree of crystallinity was determined from the diffraction patterns, which exhibited the characteristic Miller index plane peaks (hkl) at (110) and (200). These peaks are specific to the orthorhombic crystal structure of polyethylene [21].

PEX1 and PEX2 exhibit lower degrees of crystallinity compared with neat HDPE. This indicates an increase in the free volume within the crosslinked polymer chains, disrupting the nucleation and growth rates of crystallization. Consequently, the formation of a 3D network leads to the rearrangement of crystallites, although the crystalline structure remains unchanged.

The conventional crosslinking method resulted in a significant reduction in the crystallite size compared to those of PEX1 and PEX2. This can be attributed to the nature of the crosslinks formed. In the RXR method, the presence of weaker bonds, such as S-S, S-OR, and C-N, allows for greater flexibility in the network compared to the strong C-C bonds formed in the PEXC. It should be noted that the FTIR results and the chemical mechanism observed in the current work are consistent with those reported for LDPE in [22]. However, in the conventional method, the saturation of tertiary carbons with C–C bonds generates rigid bridges that hinder chain reorganization and redistribution during crystallization, leading to lattice distortion.

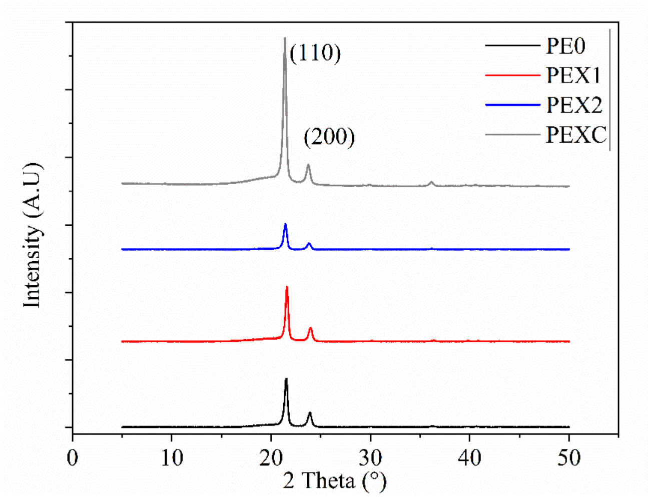

Fig. 3. X-Ray results for all materials.

C. *Thermal Stability*

The melting temperature depression was estimated using the Gibbs–Thomson equation:

$$T_m = T_m^0\left(1 - \frac{2.\sigma_e}{\Delta H_f \cdot L_C}\right) \quad (3)$$

where T_m^0 is the equilibrium melting temperature of an infinitely large crystal, σ_e is the surface free energy of the folding surface, ΔH_f is the enthalpy of fusion, and L_c is the crystallite thickness of the polymer crystal.

The degree of crystallinity (χ_C) was calculated using (4). The theoretical equilibrium melting temperature (T_m^0 = 414.6 K) and the melting enthalpy value for 100% crystalline HDPE (ΔH_m^0 = 293 Jg^{-1}) were used as reference values:

$$\chi_C = \frac{\Delta H_m(T_m)}{\Delta H_m^0(T_m^0)} \times 100\% \quad (4)$$

The crystallite thickness (L_C) was calculated as a function of crystallinity (χ_C) using the Gibbs-Thomson equation:

$$L_C = \frac{2\sigma_e T_m}{\Delta H_m^0(T_m^0 - T_m)} \quad (5)$$

where σ_e = 0.09 Jm^{-2} for polyethylene [7].

The enthalpy values of the RXR materials showed a slight decrease, consistent with the trend observed for crystallinity according to both DSC and WAXS measurements (Table II). The crystallinity rate of all materials was determined from the second heating cycle. PEXC exhibited the lowest crystallinity value. This confirms that the RXR method achieves good reversibility of crosslinking.

A slight decrease in the melting temperature (T_m) was observed, whereas the crystallization temperature (T_c) remained relatively constant. Overall, negligible variations were found between the T_m and T_c values of the reversible RXR systems and those of the uncrosslinked HDPE (PE0), indicating that the reversible network formation did not significantly alter the crystalline morphology of the material.

The crystallinity values calculated using DSC were consistent with those obtained from XRD measurements. All results are summarized in Table II.

TABLE II. THE THERMAL BEHAVIOR AND CRYSTALLINITY OF THE RAW AND CROSSLINKED MATERIALS

Thermal behavior			Crystallinity (%)		Crystallite size L_c (nm)	
Sample	T_m (°C)	T_c (°C)	DSC	WAXS	DSC	WAXS
PE0	133	114	61	65	27	21
PEXC	128	112	46	55	16	20
PEX1	131	116	62	62	22	21
PEX2	130	116	62	60	19	21

Figure 4 shows the TGA and DTG curves of virgin and crosslinked HDPE. A small initial mass loss is observed at approximately 275 °C, attributed to the decomposition of the TMTDS accelerator. The weight loss is more pronounced in PEX1 and PEX2, mainly due to volatile by-products generated from peroxide decomposition. As depicted in Figure 4(b), the DTG curves of PEX1 and PEX2 exhibit improved thermal stability compared with the conventional method, indicating that the degradation rate is considerably reduced compared with PE0 and PEXC.

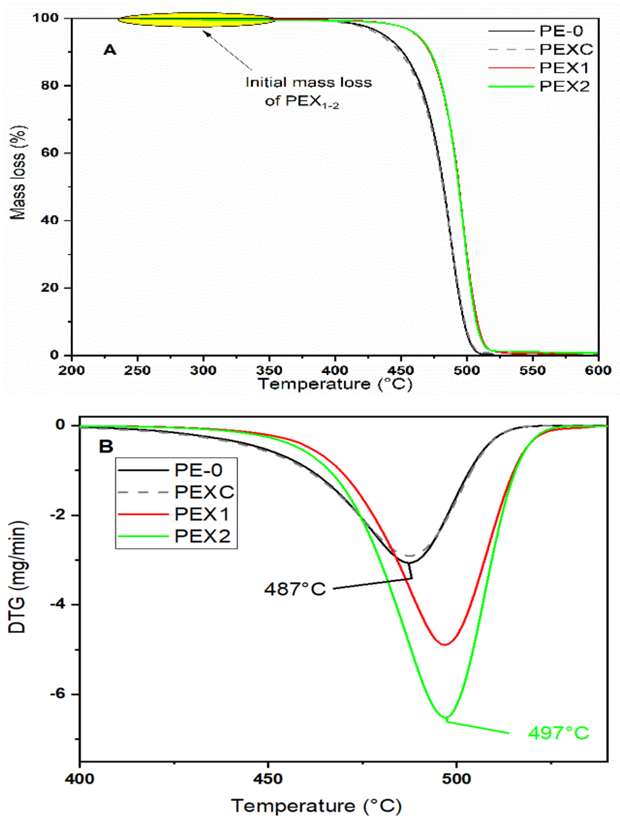

Fig. 4. Thermograms of raw and crosslinked materials: (a) TGA mass loss curves, (b) DTG thermograms.

D. Mechanical Properties

Figures 5(a) and 5(b) show the impact strength and the stiffness (modulus and tensile strength), respectively. The samples prepared using the RXR method exhibited higher stiffness than PE0. This increase in stiffness is accompanied by a reduction in elongation at break, which is a natural consequence of crosslinking. However, the elongation values remained acceptable, exceeding 200%, which is considered high [23]. The impact strength of all tested samples reveals that the RXR method yielded significantly higher impact strength values. This enhancement is attributed to the unique inter-chain clusters formed during crosslinking. In the RXR method, the crosslinking bridges are predominantly sulfur-based, which contributes to the improved mechanical performance. However, owing to the irreversible nature of PEXC, the specimen preparation for mechanical characterization was not feasible, preventing a direct comparison in this study.

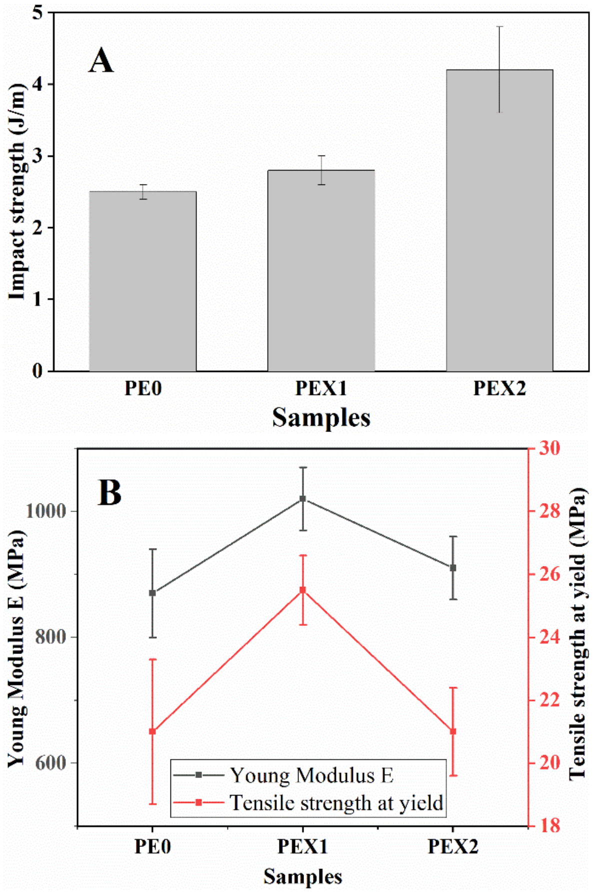

Fig. 5. Mechanical properties of all materials. (a) Impact strength results, and (b) modulus and tensile strength of PE0 and crosslinked materials.

IV. CONCLUSIONS

The results obtained in this study demonstrate that the Reversible Crosslinking (RXR) process successfully induces crosslinking reactions in High-Density Polyethylene (HDPE), while preserving its crystalline morphology and thermal characteristics. The Rheological analysis confirmed the presence of dynamic sulfur-based bonds capable of reversible exchange, leading to a flexible and reprocessable three-dimensional network. Wide-angle X-ray Scattering (WAXS) showed that RXR treatment did not significantly alter the crystallinity, and Differential Scanning Calorimetry (DSC) confirmed that the melting and crystallization behaviors remained similar to those of neat HDPE. Furthermore, the improved thermal stability of PEX1 and PEX2 compared to that of PEXC highlights the stabilizing effect of the dynamically crosslinked structure. Overall, these findings confirm that the RXR approach provides an efficient route for producing crosslinked polyethylene materials that combine reversibility, processability, and enhanced thermal resistance. It should be noted that, for the first time, a simple reactive extrusion process generates a reversible crosslinked network within HDPE, effectively bridging the gap between permanently crosslinked and recyclable thermoplastics. In line with our previous studies, the RXR, method shows great potential for industrial applications where both recyclability and sustainability are required.

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