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Enhancing Performance: Cross-Linking Strategies For Improved Mechanical And Rheogical Properties In LDPE/iPP Polymer Blends

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The field of polymer blend design is rapidly evolving, offering opportunities to create materials with precisely tailored properties. Understanding the morphology resulting from blends of immiscible polymers is essential for achieving desired mechanical attributes. This study delves into the intricate interplay between structure and properties in both unmodified blends of LDPE and iPP and cross-linking-modified blends. Through a combination of rheological assessments and mechanical testing, our investigation reveals significant insights. We find that cross-linked materials exhibit notably higher viscosity, indicative of a three-dimensional network formation and enhanced stability. Additionally, LDPE demonstrates improved behavior post- crosslinking. The decrease in melt flow index post-crosslinking suggests heightened resistance to flow and deformation, owing to the formation of additional interchain bonds. Unmodified blends display increased flexibility and ductility with higher PP content, while cross-linking induces rigidity. However, cross-linking mitigates rigidity in blends with lower iPP content, resulting in enhanced flexibility. Moreover, mechanical strength significantly improves with iPP incorporation and cross-linking agents, reinforcing structural integrity. Furthermore, impact resistance benefits from iPP incorporation and cross-linking, rendering the materials suitable for applications requiring robust performance under challenging conditions.

Keywords: Crosslinking, Blends, LDPE, iPP.

1. Introduction

Polymer blend design has emerged as an interest area of research for materials with the main properties for diverse applications. Among the numerous polymer blends, those comprising low-density polyethylene (LDPE) and polypropylene (PP) stand out due to their prevalence in plastic solid waste and industrial sectors. However, the immiscibility and incompatibility of LDPE and PP present challenges for recycling and limit their potential applications. Traditional recycling methods often fail to harness the full potential of these blends due to difficulties in achieving homogeneous morphology and desired mechanical properties.

In recent years, the concept of the circular economy has gained traction, advocating for sustainable approaches to material use and waste management. In this context, transforming waste polymers into high-value materials with minimal energy input has become imperative. Microfibrillar composites have emerged as a promising solution to enhance the performance of immiscible polymer blends, leveraging their inherent immiscibility to create a hierarchical structure with improved mechanical properties.

The present study aims to explore the potential of cross-linking strategies in enhancing the mechanical and morphological properties of LDPE/PP polymer blends. Cross-linking a widely studied technique in polymer science involves the formation of covalent bonds between polymer chains leading to improved mechanical strength, thermal stability as well as resistance to environmental factors. By selectively cross-linking LDPE/PP blends, it is possible to modify their morphology and enhance their overall performance.

Previous research has laid the groundwork for understanding the behavior of LDPE/PP blends and the effects of cross-linking on their properties. Zhang et al. [1] investigated the effect of cross-linking on the mechanical properties of LDPE/PP blends and reported significant enhancements in tensile strength and impact resistance. In the other hand, Wang and Li [2] studied the rheological behavior of cross-linked LDPE/PP blends and observed improvements in melt viscosity and processability. Smith et al. [3] investigated the rheological behavior of LDPE/PP blends and observed significant improvements in melt viscosity with the addition of a compatibilizer. Similarly, Jones and Brown [4] explored the impact of cross-linking on the mechanical properties of LDPE/PP blends and reported enhanced tensile strength and impact resistance.

Many studies contribute to the understanding of cross-linking strategies for improving the mechanical and morphological properties of LDPE/PP polymer blends: Thato et al. [5], explored the effect of cross-linking on the thermal properties of LDPE/wax blends. It was observed that the thermal stability of the blends decreased with an increase in wax content, and no direct correlation between thermal stability and cross- link density. Jun et al. [6], investigated Triple Shape Memory Effects (SME) of Cross- Linked Polyethylene/Polypropylene Blends with Cocontinuous Architecture. Results showed that the chemical cross-linking causes lower melting temperature (Tm) and smaller melting enthalpy (ΔHm). This new strategy of chemically cross-linked immiscible blends with cocontinuous architecture can be used to design and prepare new SMPs with triple SMEs. Yoko et al. [7], Studied the modification of the rheological properties of polypropylene under elongational flow by adding polyethylene. Results showed that

adding LDPE to PP increased the strain hardening in the transient elongational viscosity and the nucleating ability of deformed HDPE to PP may also contribute to the drawdown force.

Many research works [8-12], focused on the reactive extrusion process to cross-link polymer blends as well as polymer nanocomposites. Authors investigated the combined effects of compatibilization and cross-linking on the properties of the different compositions.

Other research works contribute to the growing body of knowledge on cross-linking strategies for improving the mechanical and morphological properties of LDPE/PP polymer blends, offering insights into novel approaches, material enhancements, and characterization techniques : Dhoble et al. [13], investigated the effects of reactive extrusion cross-linking on the morphology and properties of LDPE/PP blends, highlighting the potential for tailoring material properties through controlled processing conditions. Dorigato and Fredi [14], explored the incorporation of nanofillers into LDPE/PP blends followed by cross-linking, aiming to synergistically enhance mechanical and thermal properties while maintaining processability.

Some research works [15,16], contributed to expanding the knowledge base on cross-linking strategies for enhancing the mechanical and morphological properties of LDPE/PP polymer blends, addressing various aspects such as thermal stability, adhesion, characterization techniques, and specialized applications.

Other ones [17-19] contributed to the advancement of cross-linking strategies for LDPE/PP blends, addressing a wide range of topics including biodegradability, 3D printing, flexible packaging, rheological behavior, and toughness enhancement.

Many studies [20-23] further expand the knowledge base on cross-linking strategies for LDPE/PP blends, covering aspects such as chemical resistance, outdoor durability, fiber reinforcement, nanoparticle incorporation, and sustainability in agricultural applications.

The current research aims to delve deeper into the intricate interplay between structure and properties in LDPE/PP blends modified by cross-linking. By systematically varying blend compositions and cross-linking agents, we seek to elucidate the underlying mechanisms governing the performance enhancement observed in these materials. Comprehensive characterization through rheological assessments and structural analyses will provide valuable insights into the effect of cross-linking on the morphological evolution and mechanical behavior of LDPE/PP blends.

The cross-linked LDPE/PP composites explored in this study demonstrate remarkable potential for groundbreaking applications in sustainable packaging and high-performance automotive components. Using their enhanced mechanical and morphological properties, these materials offer innovative solutions that prioritize both performance and environmental sustainability.

2. Experiment

2.1. Materials

The LDPE (B21 sak) used in this study is provided from National Petrochemical Company Industrial Zone P.O. Box 210 Skikda 21000, Algeria in granulated form, with a density of 0.92 g/cm³. iPP Sabic-Vestolen 9000-67404 is supplied by Chemische Werke Hu¨ls, Postfach 1320, D-4370 Marl 1, Germany in granulated form, with a density of 0.90 g/cm³. The dicumyl peroxide (DCP) used in this study is of the dialkyl peroxide type, its physical form a white powder. Its decomposition temperature is 140°C. It is provided from Acros Organics, BV. Janssen Pharmaceuticalaan 3A 2440; Belgium. Sulfur (S) (vulcanizing agent for rubber) is supplied byWuxi Huasbeng Chemical Additives Factory, No. 18, Weiye Road, Qianqiao, Wuxi, Jiangsu, China. The used accelerator is ‘‘Super accelerator 501. (TMTD); supplied by Rhodia 25 Clichy Street, Paris 75 75009, France.

2.2. Polymers blend Elaboration

To prepare the blends, the mixtures were subjected to melt-processing using a Brabender internal plastograph at 190°C and 50 rpm for 15 minutes. The sulfur concentration equaled that of the peroxide consistently. Sulfur and peroxide were included at concentrations of either 0.2 or 0.4 wt %. In each case, the accelerator comprised one-fourth of the combined concentration of sulfur and peroxide. The composition of the samples examined in this study is outlined in Tables I.

Table 1. LDPE/iPP Blend Composition

Formulation	Formulation 1	Formulation 2	Formulation 3	Formulation 4	Formulation 5
LDPE/iPP Ratio	100/0	97/3	95/5	90/10	85/15
Crosslinking agent concentrations (%)	0.4	0.4	0.4	0.4	0.4

The previously prepared mixtures were ground and then transformed into samples with dimensions of 4 mm thickness, 10 mm width, and 78 mm length for Charpy impact strength tests, and those with dimensions of 2 mm thickness, 5 mm width, and 40 mm length for tensile strength tests. A "POLYLAB" hydraulic press was used for shaping the various samples at 190°C, with a preheating time of 10 minutes.

2.3. Characterization

The Dynamic Rheological Behavior (DRA) study, pioneered by Harpell and Walrod [24], assesses crosslinking reactions to understand reaction kinetics and confirm rheological behavior changes due to structural modifications. The plastograph, with its thermo- regulated mixing chamber and two rotors connected to a motor, is ideal for this study. It records torque over time as the polymer melts, sharply increases torque to a maximum, then gradually decreases until stabilizing at a lower level. This torque-time

plot provides valuable insights, especially for polyolefins.

Measuring the melt flow index is a commonly employed method that offers insights into the rheological characteristics of polymers. The assessments of various blends were conducted using a Melt-Indexer, model 5, equipped with a vertically oriented cylinder ending at its lower extremity with a standard die measuring 8 mm in length and 2.09 mm in diameter. Tests were conducted at two temperatures, 190 and 230°C, utilizing two loads of 2.16 and 5 kg.

The tensile test, widely employed in mechanics, assesses a material's resistance to external stresses and identifies its breaking point. Conducted at a speed of 5 mm/min, the test utilized specimens prepared according to ASTM-D-638 standards. The testing machine, a "TMS" type, was computer-assisted.

Impact testing arises from subjecting a material to high-speed high-energy mechanical stress resulting in sample fracture. It serves to evaluate material brittleness under defined experimental conditions. The outcome is influenced by molecular relaxation processes linked to rupture time and temperature as well as by sample geometry (notched and unnotched). This study employed both Charpy and Izod methods, for notched and unnotched samples, in accordance with ISO 179 standard.

3. Results and Discussion

Suggested Mechanism

The fundamental concept behind the method patented by Bouhelal in 2009 [25] revolves around the creation of macroradicals via peroxide decomposition through a homolytic reaction which occurs under specific processing conditions. This reaction leads to the formation of a three-dimensional network as polymer chains become linked by sulfur atoms. Additional information regarding the methods described above can be found in references [8, 9 and 26].

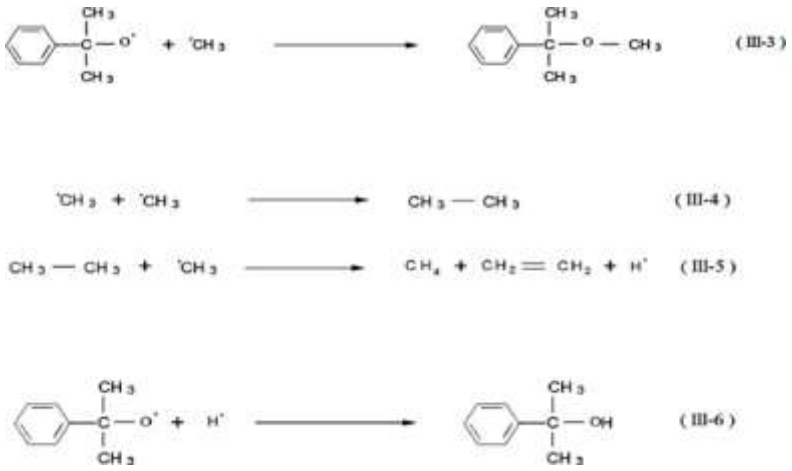
In our study, we utilized DCP as one of the components referred to as "active agents" (explained below). The other "active agents" include sulfur and an activator (TMTD, in our case), while the remaining components of the system studied are LDPE and iPP.

Reactive Mechanism of Dicumyl Peroxide Decomposition

The decomposition of dicumyl peroxide occurs through a radical reaction initiated by heat. Initially, the peroxide breaks down into reactive oxyradicals, as shown in reaction III-1 of Figure III-1. These oxyradicals further decompose, resulting in the formation of acetophenone and a methyl radical, as illustrated in reaction III-2 of Figure III-1.

Following this, the methyl radical combines with the cumyl radical to produce the ether cumyl methyl, depicted in reaction III-3 of Figure III-1. Additionally, there is a possibility of two methyl radicals associating to form ethane, as shown in reaction III-4 of Figure III- 1.

Ethane undergoes attack by methyl radicals, leading to the formation of an ethyl radical. This ethyl radical stabilizes by generating ethylene and an hydrogen radical, as demonstrated in reaction III-5 of Figure III-1. Finally, the hydrogen radical attacks the cumyl radical, resulting in the formation of phenyl dimethyl carbinol, as shown in reaction III-6 of Figure III-1.



This series of reactions illustrates the intricate process of dicumyl peroxide decomposition and the various compounds formed as intermediates.

3.1. Rheological Analysis

3.1.1. Dynamic Rheological Analysis (DRA)

* Dynamic Rheological Analysis (DRA) Results for Unmodified LDPE/iPP Blends:

Analyzing Figure 1 reveals a consistent pattern in the curves across all blends with different iPP ratios. Three key points, marked as A, B, and C, stand out. Initially, there's a sharp increase in torque until reaching point A. This surge is due to the substantial resistance exerted by the solid granules in the blends. Moving past this point, torque gradually decreases until reaching point B, signaling the transition from a solid to a molten state within the blends.

Following point B, torque stabilizes, forming a plateau at point C. This plateau represents the equilibrium torque, offering insights into the final viscosity of the blends. Notably, as the iPP content increases, the final viscosity decreases, indicating a negative correlation between PP concentration and viscosity.

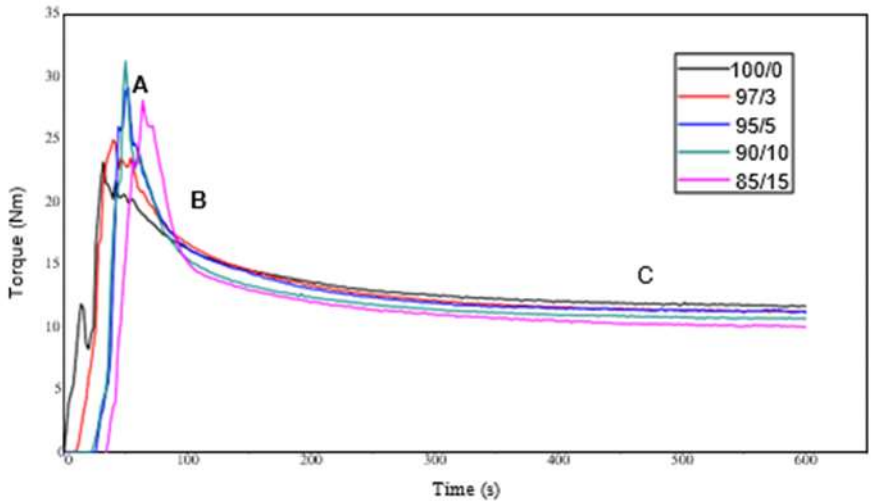


Figure 1: Evolution of Torque-time for LDPE/iPP unmodified blends as a function of Polypropylene content

* Dynamic Rheological Analysis (DRA) Results for LDPE/iPP Blends Modified with Crosslinking Agents:

In Figure 2, which investigates the behavior of LDPE/iPP blends treated with crosslinking agents, it becomes evident that there is a significant increase in torque from point B to point D, indicating the initiation of the crosslinking phase. This rise suggests the formation of a network structure within the blend. Subsequently, a slight decrease in torque occurs, attributed to the partial disruption of this network. Following this, torque stabilizes, forming a plateau at point C, indicating a consistent torque level. Additionally, a decrease in the viscosity of crosslinked blends compared to unmodified blends is observed. Despite this reduction, it's noteworthy that the viscosity of crosslinked blends remains significantly higher than that of unmodified blends.

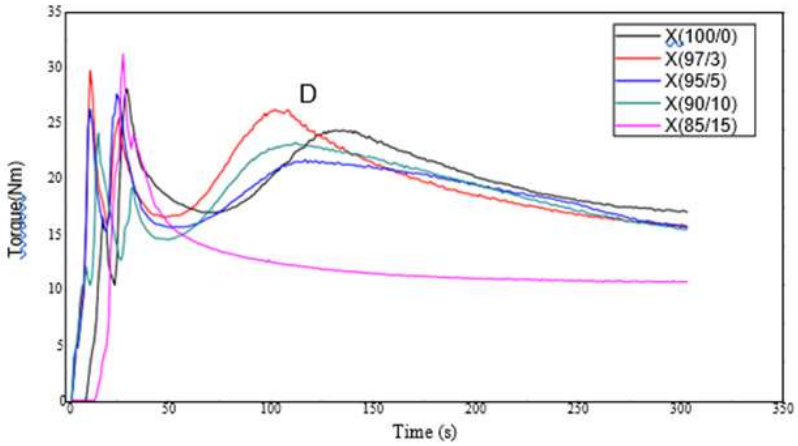


Figure 2: Evolution of Torque-time for modified blends X(LDPE/iPP) as a function

of iso Polypropylene content

3.1.2. Melt Flow Index Analysis

The melt flow index is a parameter which serves as a valuable indicator, providing insights into a polymer's viscosity, branching rate, free volume between chains, and degree of crystallinity.

Examining Figures 3, 4, 5, and 6 reveals an important observation; as the iPP content increases in the LDPE/iPP blends, the melt flow index also rises. This indicates a reduction in viscosity [27-29], attributed to two primary factors. Firstly, the polymer chains exhibit increased movement of polymer chains, resulting in decreased resistance to flow [30]. Secondly, iPP inherently possesses a higher melt flow index compared to LDPE.

Moreover, the influence of temperature and shear rate is notable. At higher temperatures and shear rates, the increase in melt flow index is more pronounced, facilitating easier flow by promoting polymer chain mobility.

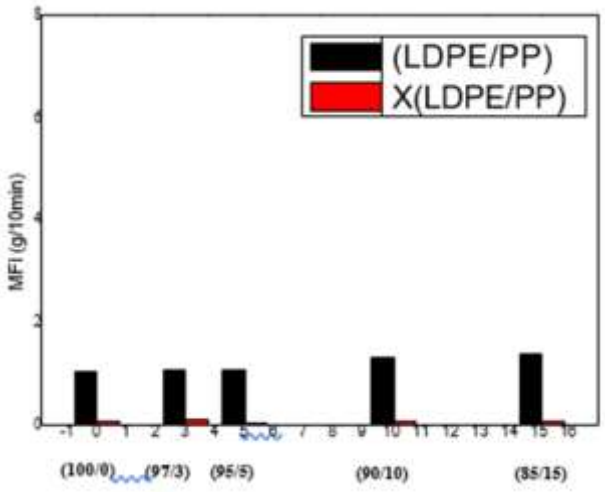


Figure 3: Variation of the melt flow index of blends (LDPE/iPP) and X (LDPE/iPP) at T°=190°C and 2.16Kg

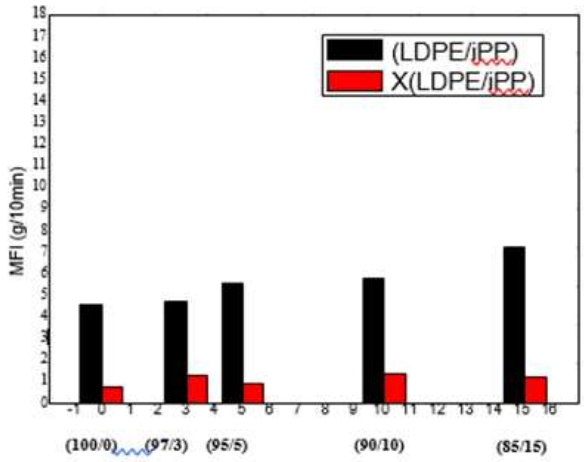


Figure 4: Variation of the melt flow index of blends (LDPE/iPP) and X (LDPE/iPP) at T°=190°C and 5Kg

Conversely, blends modified with crosslinking agents exhibit a decrease in the melt flow index. This decline suggests an increase in viscosity due to crosslinking, which introduces additional bonds between polymer chains, hindering their movement and increasing resistance to flow [31].

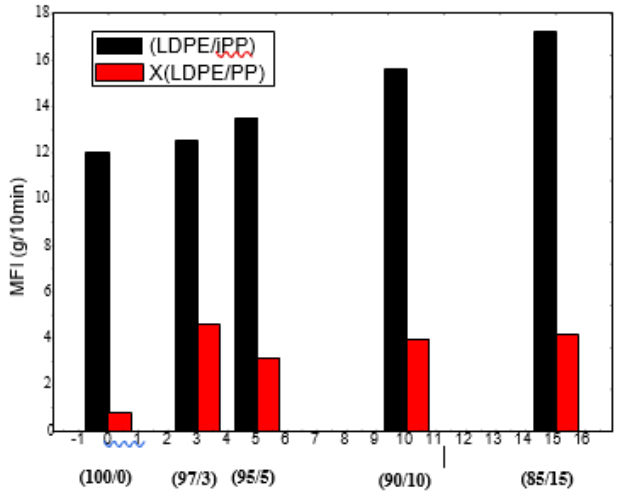


Figure 5: Variation of the melt flow index of blends (LDPE/iPP) and X (LDPE/iPP) at T°=230°C and 5Kg

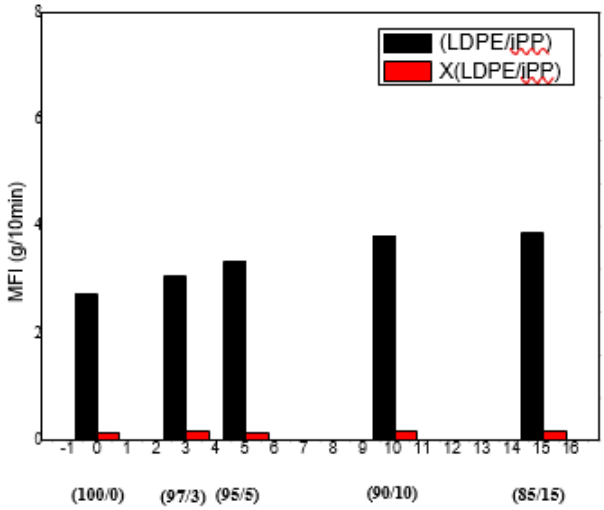


Figure 6: Variation of the melt flow index of blends (LDPE/iPP) and X (LDPE/iPP) at T°=230°C and 2.16Kg

Lastly, it is observed that at elevated temperatures and shear rates, the melt flow index tends to be higher. This may be attributed to chain scission in iPP, as it does not undergo complete crosslinking.

3.2. Tensile Test

- Modulus of Elasticity

According to Figures 7 and 8, in unmodified blends of LDPE/iPP, there is an initial increase in modulus and stress for the lower iPP compositions, followed by a decrease for the higher iPP compositions. This suggests that at lower iPP content, the material exhibits increased rigidity, while at higher iPP content, it becomes more flexible [32-34]. As for blends modified with crosslinking agents, the dynamics are more nuanced. In blends with lower iPP content as 97/3 and 95/5, there is a decrease in modulus, indicating enhanced flexibility due to crosslinking. However, for blends with higher iPP content, there is an increase in modulus, signifying increased rigidity resulting from crosslinking [35, 36].

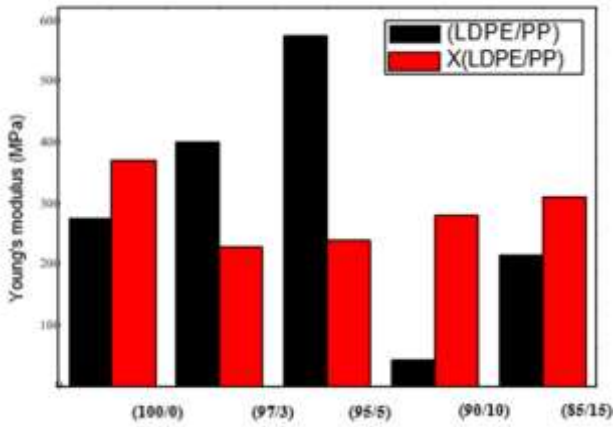


Figure 7: Variation of the Young's modulus of blends (LDPE/iPP) and X (LDPE/iPP)

- Tensile strength

These findings underscore the significant influence of both iPP content and crosslinking on the mechanical properties of LDPE/iPP blends. The effects on rigidity and flexibility vary depending on the blend composition and crosslinking status, highlighting the importance of understanding these factors in materials engineering [37].

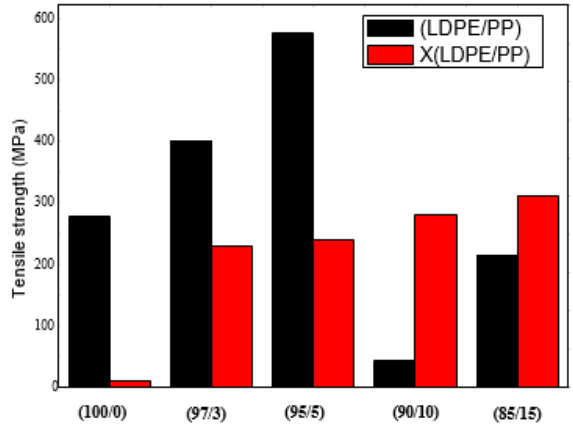


Figure 8: Variation of the tensile strength (σt) of blends (LDPE/iPP) and X (LDPE/iPP)

- **Strain at Break**

Based on the data depicted in Figure 9, significant observations regarding material behavior become evident: at low iPP content, the materials exhibit a consistent level of deformation, reflecting their inherent [38], indicating a transition towards ductile behavior. When crosslinking agents are introduced, the behavior becomes more complex. Initially, for the first three blends, there is a decrease in deformation, suggesting that crosslinking enhances ductility. Conversely, for the last two blends, there is an increase in deformation, implying a shift towards increased brittleness [39].

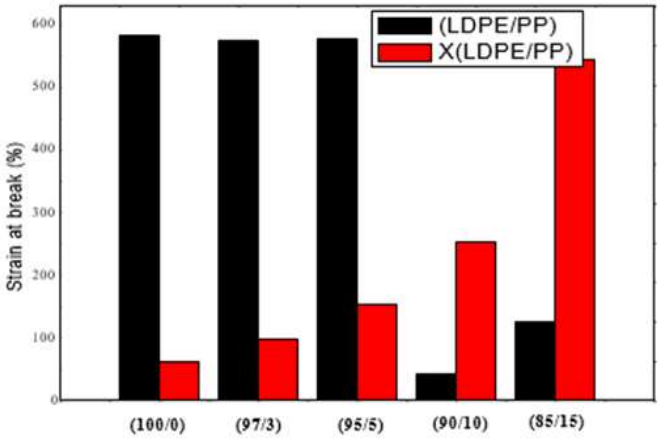


Figure 9: Strain at Break (εb) variation for blends (LDPE/iPP) and X (LDPE/iPP)

These findings highlight the significant impact of both iPP content and crosslinking on the mechanical properties of the materials, influencing their ductility and brittleness [40].

3.3. Impact Strength

Based on the information provided in Figure 10, it's evident that there is an increase in resilience observed, particularly notable in the blend containing 5% iPP. This indicates that the inclusion of Polypropylene contributes to an improvement in the materials' impact resistance [41]. Additionally, it's noteworthy that crosslinking enhances the impact resistance, reaching an optimal value in the blend with 3% iPP.

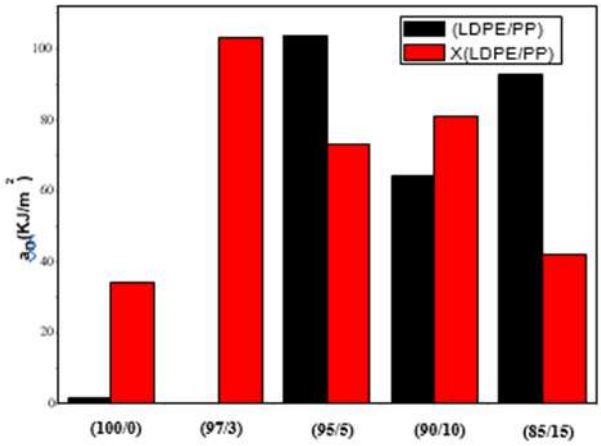


Figure 10: Variation in Charpy resilience of blends (LDPE/iPP) and X (LDPE/iPP)

Figure IV.11 provides compelling evidence that both the inclusion of iPP in LDPE and the addition of crosslinking agents have a substantial positive effect on the impact resistance of the materials. Specifically, results demonstrated a noticeable increase in impact strength as the iPP content increases in the LDPE blend.

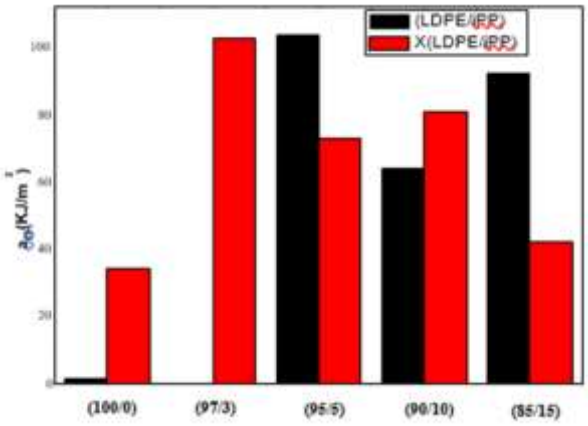


Figure 11: Variation in Izod resilience of blends (LDPE/iPP) and X (LDPE/iPP)

Moreover, the impact resistance is further enhanced by the presence of crosslinking agents. The addition of these agents appears to reinforce the molecular structure of the material, resulting in improved resistance to fracture and deformation under impact loading conditions. This synergistic effect between iPP incorporation and crosslinking is particularly evident, as the impact resistance of the blends with both iPP and crosslinking agents surpass that of the individual components alone.

5. Conclusions

In this study, we delved into the intricate relationship between cross-linking, iPP incorporation, and the mechanical properties of LDPE/iPP blends. Our goal was to unravel the nuanced effects

of these factors on the behavior of the materials. Here's a detailed summary of our key findings:

-Rheological analysis unveiled a notable increase in viscosity upon the addition of crosslinking agents. This elevation in viscosity signifies the successful establishment of a three-dimensional network within the material structure, enhancing its overall integrity and stability. Moreover, LDPE exhibited favorable alterations in its behavior post- crosslinking, indicative of enhanced performance characteristics.

-Melt flow index assessments revealed a discernible decrease post-cross-linking, signifying a reduction in the material's flowability. This reduction in flow index suggests the formation of additional interchain bonds, thereby augmenting the material's resistance to flow and deformation.

-Mechanical testing provided intriguing insights into the structural dynamics of the LDPE/iPP blends. Specifically, unmodified blends showcased enhanced flexibility and ductility at higher PP content, whereas crosslinking induced rigidity. Conversely, blends with lower iPP content exhibited rigidity, which was subsequently mitigated by crosslinking, leading to improved flexibility.

Notably, the mechanical strength of the blends witnessed a significant boost with the incorporation of iPP and cross-linking agents. This augmentation in strength underscores the synergistic effect of iPP incorporation and cross-linking, which reinforces the material's structural integrity and load-bearing capacity.

-Impact resistance emerged as a key beneficiary of iPP incorporation and cross-linking, with a marked improvement observed across all blend compositions. This enhancement in impact resistance signifies the material's increased ability to withstand sudden loads and dynamic forces, making it well-suited for applications requiring robust performance under challenging conditions.

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