Enhancing the dynamic mechanical properties of thermoplastic elastomers: A study on polypropylene /natural rubber blends

Egharevba Owen[a*], Ong Siew Kooi[b], Okieimen Felix Ebhodaghe[c], and Ifijen Ikhazuagbe Hilary[a]

[a]Research Outreach Department, Rubber Research Institute of Nigeria, PMB 1049, Benin City, Edo State, Nigeria
[b]Polymer Engineering Technology Section, Universiti Kuala Lumpur-Malaysian Institute of Chemical and Bioengineering Technology, Alor Gajah, Melaka, Malaysia
[c]Department of Chemistry, University of Benin, P.M.B 1155, Benin City, Edo State, Nigeria

CHRONICLE	ABSTRACT
	The aim of this study was to investigate the modifications of the mechanical properties of polypropylene (PP) by incorporating elastomers, while considering the impact on its stiffness. Specifically, the research focused on determining the optimal loading of elastomer to achieve desirable properties and exploring the influence of these processes on the morphology and mechanical behavior of the prepared blends. Thermoplastic elastomers (TPEs) consisting of polypropylene and natural rubber (PP/NR) were prepared using a melt-mixing process, and the mechanical properties of the blends were evaluated. The stress-strain properties of the blends revealed a successful modification of PP, transforming it from a stiff and strong thermoplastic into a stiff and tough thermoplastic elastomer when 10% NR was included in the PP matrix. As the loading of NR increased, a reduction in tensile strength (TS) and modulus (E) of the blends was observed, while elongation at break (EB) increased. The flexural strength of unmodified PP was 45.9 MPa, which decreased with increasing NR loading. Similarly, the impact strength of unmodified PP was 25.8 KJ/m^2, whereas the values for 10%, 20%, 30%, and 40% NR inclusion were 30.8, 24.3, 20.6, and 15.2 KJ/m^2, respectively. The melt flow index (MFI) of unmodified PP was 14.1 g/10 min, while the values for 10%, 20%, 30%, and 40% NR inclusion were 19.4, 15.7, 11.6, and 10.2 g/10 min, respectively. The best combination of mechanical properties was observed at 10% NR inclusion in the PP matrix. The micrograph of the blends, as observed from SEM micrographs, supported the modification of PP, resulting in the production of TPE with observable adhesion sites, indicating good compatibility between the components. In a nutshell, a significant 47% increase in impact strength was achieved through the modification process.

1. Introduction

Polymers are large molecules consisting of repeating units called monomers[1-2]. They can be found in various natural and synthetic forms, displaying a wide range of properties like flexibility, strength, and resistance to heat and chemicals.[3-4]. These versatile materials are extensively used in industries such as packaging, construction, electronics, and healthcare [5-9].

A significant class of polymers, known as thermoplastic elastomers (TPEs), merges the beneficial properties of thermoplastics and elastomers, offering both flexibility and ease of processing[10]. TPEs have become widely used in various sectors such as automotive and consumer goods due to their distinctive capability to demonstrate elastic behavior while also being malleable and reshaped[11]. Thermoplastic elastomers (TPEs) are a unique class of materials that possess a combination of the desirable processing properties of thermoplastics at elevated temperatures and the physical properties of conventional

elastomers at service temperatures[12]. Polypropylene (PP), due to its advantageous characteristics such as low cost, moldability, and suitability for weight reduction, finds application in a wide range of industries[13]. In fact, PP accounts for over half of all plastic materials utilized in the automotive sector[14-15]. However, the poor impact resistance of PP, particularly at low temperatures, has prompted significant interest in improving its toughness and impact properties[16-17].

One established method for enhancing the toughness of otherwise brittle polymers involves the incorporation of a dispersed rubber phase. This approach relies on the formation of microscale craze zones within the polymer matrix, enabling energy absorption upon impact and leading to improved impact strength[18]. Nevertheless, it has been noted that the modification of thermoplastics with elastomers often results in a decrease in stiffness[19]. Researchers have shown interest in utilizing natural rubber (NR) for the modification of plastic materials[20]. However, NR has predominantly been employed as a compatibilizer in polymer blends.

In this study, the objective is to incorporate natural rubber into a polypropylene matrix through melt-mixing processes. The investigation will focus on determining the optimal loading of NR to achieve desirable properties and assessing how these processing techniques influence the morphology and mechanical behavior of the resulting blends. By carefully studying these aspects, valuable insights can be gained into the potential of NR as a modifier for improving the performance of PP-based materials.

2. Results and discussion

2.1 Thermoplastic elastomer properties of PP/NR blends

Processing characteristics

To gain a comprehensive understanding of the processing behavior during the melt-mixing process of Polypropylene (PP) and natural rubber (NR), the monitored stabilized torque values provide valuable insights into the viscosity characteristics of the melt system. The obtained results reveal that the stabilized torque values exhibit an increase with higher loading of NR, as presented in **Table 1**. **Fig. 1** illustrates the typical torque curves for PP/NR blends at various NR loadings.

Table 1. Stabilization torque values for PP/NR blends at various NR loading

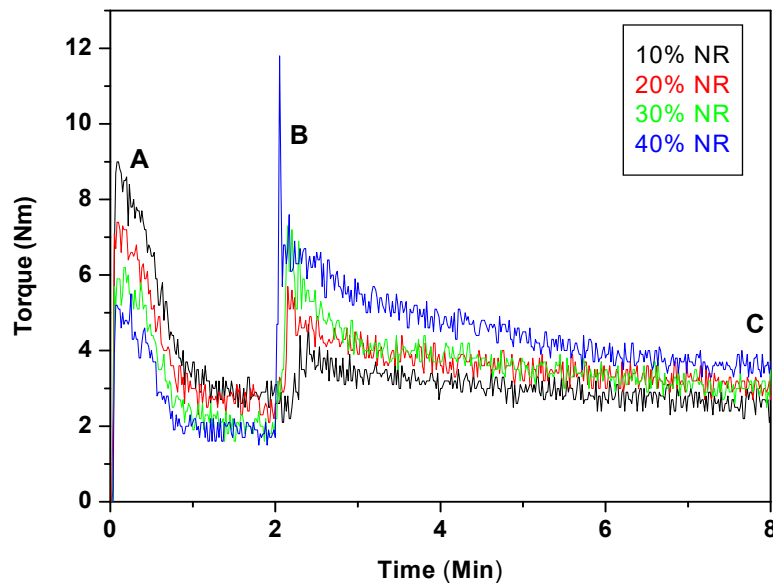| PP/NR Blends (% NR)	Maximum torque value after adding NR (N/m)	Torque value at the end of the mixing cycle (N/m)
10	4.7	2.3
20	5.7	2.7
30	7.3	3.5
40	11.8	3.7

Fig. 1. Mixing torque-time for PP/NR blends at various NR loading Points: (A) polypropylene addition, (B) natural rubber addition, (C) stabilization torque

The torque curves in **Fig. 1** exhibit distinct peaks at points (A) and (B), which correspond to specific stages in the melt-mixing process. The initial peak observed at point (A) is attributed to the presence of polypropylene in the mixing chamber. During the first 2 minutes, as PP is charged into the mixing chamber, the torque increases due to the resistance imposed on the rotors by the un-melted PP. As the PP gradually melts and experiences mechanical shearing, the temperature inside the chamber rises, leading to a reduction in torque over time.

At approximately 2 minutes into the melt-mixing process, the addition of NR occurs, denoted by point (B) on the torque curve. This introduction of NR causes a sharp increase in torque. As the mixing progresses and becomes more homogeneous, the torque steadily decreases until it reaches a relatively constant value around the 8th minute (point C). This stabilization torque at point C indicates the completion of the mixing process, where a consistent and uniform blend of PP and NR has been achieved. The monitoring of torque values throughout the melt-mixing process provides valuable information about the progression and completion of mixing, aiding in the characterization of the melt behavior and the achievement of an optimized blend of PP and NR.

The analysis of stabilization torque values presented in **Table 1** indicates a notable increase in torque with higher elastomer content in the blends. This observation can be attributed to the addition of natural rubber, which possesses a higher viscosity compared to the polypropylene matrix. As a result, the presence of natural rubber introduces greater resistance to the rotation of the rotors, leading to an increase in torque during the melt-mixing process.

The study's results are in line with earlier research[24], indicating that blends with higher elastomer content require increased energy input for successful melt processing. Previous studies reported stabilization torque values of up to 5 Nm and 10 Nm during melting mixing of un-vulcanized PP/EPDM/NR ternary blends and polypropylene/acrylic rubber blends, respectively [25-26]. It is essential to note that the rubber phase's crosslinking can further contribute to higher torque values. This phenomenon is observable in dynamically vulcanized blends, such as those consisting of poly(Epichlorohydrin-co-ethylene oxide) and polypropylene[26]. Evidence from both the present study and earlier research underscores the impact of elastomer content on the viscosity and processing characteristics of blends. The rise in torque with elevated elastomer concentrations emphasizes the necessity for meticulous attention and precise adjustment of processing parameters to guarantee efficient and consistent melt mixing of PP and NR blends.

2.2 Mechanical properties: Modulus, TS, EB and flexural strength

During the investigation of the mechanical properties of the blends, tensile data was obtained from stress-strain curves of PP/NR blends with varying NR loadings, as illustrated in **Fig. 2**. It was observed that increasing the NR loading in the PP matrix resulted in a decrease in tensile strength (TS) and modulus (E) of the blends, while the elongation at break (EB) increased (**Table 2**). This trend aligns with findings from previous studies[27], which support the notion that increasing elastomer content in polymer blends leads to a decrease in stiffness and strength properties, accompanied by an improvement in ductility.

Table 2. Tensile properties of PP/NR thermoplastic elastomers

PP/NR Blends (% NR)	Young's modulus (MPa)	Tensile strength (MPa)	EB (%)	Flexural strength (MPa)
0	740.76	31.76	14.71	45.90
10	561.73	23.14	18.80	33.77
20	421.53	16.30	21.85	27.39
30	359.68	12.23	22.26	21.74
40	243.58	7.82	28.07	18.95

To gain further insights into the nature of the stress-strain curves obtained for the different blends, a careful assessment was conducted. Stress-strain curves of polymer materials have been categorized based on established templates[28]. In the case of the respective PP/NR blends, the stress-strain curves were categorized accordingly. The nature of PP-based thermoplastic elastomers was classified as follows: unmodified PP - a stiff and strong material; 10% NR loading - a stiff and tough material; 20% NR loading - a flexible and tough material; 30% NR loading - a flexible and weak material; and 40% NR loading - a flexible and weak material.

Additionally, the flexural properties of the blends were evaluated using three-point bending tests, and the results are also presented in **Table 2**. The flexural strength of unmodified PP was determined to be 45.9 MPa. It was observed that the addition of NR resulted in a reduction in flexural strength, with values decreasing as the NR loading increased.

Considering the overall desirable properties, the PP/NR blend containing 10% NR loading demonstrated a favorable combination of tensile properties, exhibiting a balance between elongation and modulus responses. This suggests that this particular blend achieved an optimum balance between toughness and stiffness, making it a promising choice in terms of desired material characteristics.
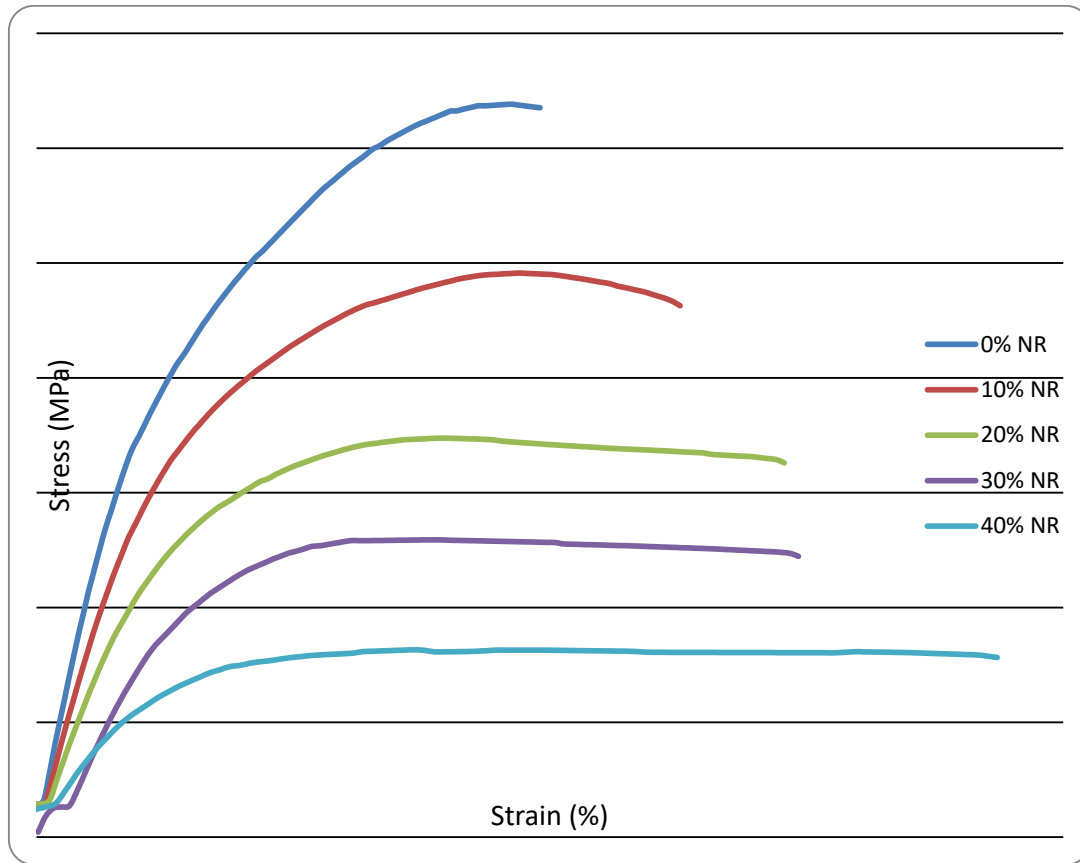
Fig. 2. Stress-strain curve for PP/NR thermoplastic elastomers at various NR loading

2.3 Melt flow index (MFI) and impact strength

Table 3 provides information on the melt flow index (MFI) and Charpy impact properties of the thermoplastic elastomers (TPEs) composed of polypropylene and natural rubber. The MFI of a polymer refers to the rate of flow, measured in grams per 10 minutes, through a standard die under specific test conditions of temperature and applied pressure[26]. A higher MFI value indicates that the polymer has a greater flow rate.

Table 3. % loss or gain in melt flow index and impact strength of PP/NR blends

PP/NR Blends (%NR)	MFI (g/10 min)	% MFI (Gain or loss)	Impact strength (KJ/m²)	% Impact strength (Gain or loss)
0	14.10	-	25.81	-
10	19.40	37.6↑	38.04	47.3↑
20	15.74	11.6↑	24.33	5.7 ↓
30	11.63	17.5 ↓	20.56	20.3 ↓
40	10.24	27.4 ↓	15.18	41.2 ↓

↑denotes increase, while ↓denotes decrease

The data obtained from this study reveals that the TPE containing 10% NR inclusion in the polypropylene matrix exhibited the highest MFI value among all the prepared blends. This suggests that this blend is most suitable for injection molding processes, as it demonstrates a greater flowability under the specified test conditions. The MFI value is an important factor to consider in selecting materials for injection molding applications, as it influences the ease and efficiency of the molding process. Regarding the impact properties, the impact strength of unmodified PP was measured to be 25.8 KJ/m². However, upon incorporating NR at a loading of 10%, a significant increase of 47% in impact strength was observed. The impact properties of a polymer are crucial parameters when considering materials for applications requiring impact resistance. The results from this study indicate that the incorporation of NR resulted in a considerable modification of the impact properties of the PP matrix, leading to improved impact strength.

Impact data, such as the Charpy impact test, are widely utilized in material selection and quality control processes. These properties provide valuable insights into the material's ability to withstand impact forces and its suitability for impact-related applications. The findings from this study highlight the significant enhancement of impact properties achieved

through the incorporation of NR into the PP matrix, reinforcing the potential benefits of this TPE blend in impact-sensitive applications.

3. Morphology

Fig. 3a displays a scanning electron micrograph of unmodified PP, revealing a brittle domain in the microstructure. However, upon investigating the PP/NR blend with a 10% NR loading, the study found that it exhibited the best combination of mechanical properties among all the prepared TPEs. Based on this observation, further analysis was conducted to examine the compatibility of the PP/NR (90/10) blend using scanning electron microscopy. The corresponding micrograph is presented in **Fig. 3b**.
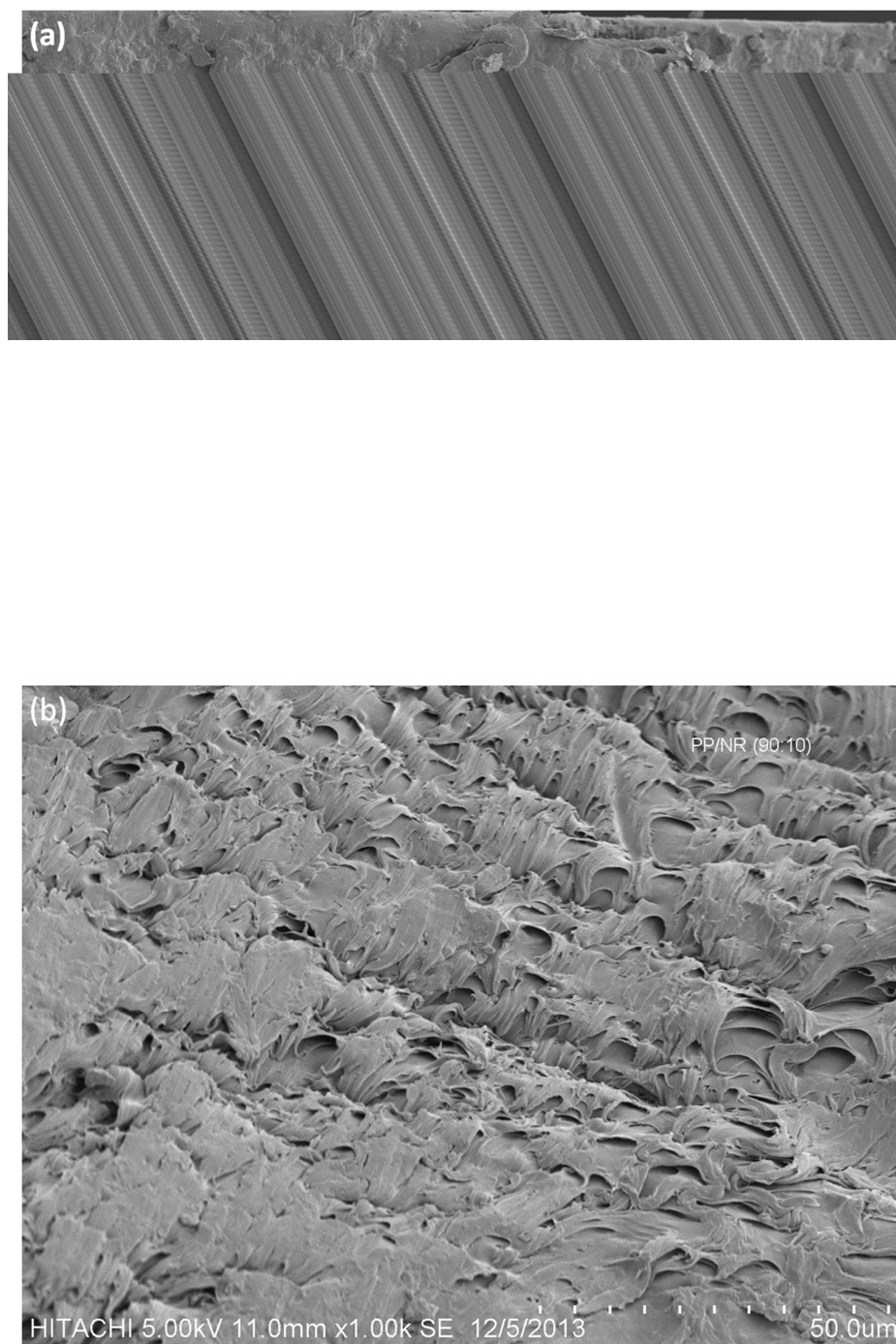

Fig. 3.   SEM micrographs of (a) unmodified PP, (b) thermoplastic elastomer of PP/NR (90/10) blend

Upon examining the micrograph, the presence of adhesion sites between the PP and NR phases was observed, indicating good compatibility between the two components. This observation provides a plausible explanation for the enhanced mechanical properties observed in the prepared thermoplastic elastomer with a 10% NR loading, as compared to unmodified PP. The presence of adhesion sites suggests effective interfacial bonding and interaction between the PP matrix and NR phase, resulting in improved mechanical performance. This compatibility and strong interfacial adhesion contribute to the overall enhanced properties, such as improved toughness, impact resistance, and elongation at break, observed in the PP/NR blend with 10% NR loading.

The scanning electron micrograph provides visual evidence supporting the hypothesis that the adhesion and compatibility between the PP and NR phases are crucial factors contributing to the improved mechanical properties of the TPE blend. These findings further support the potential of utilizing NR as a modifier for enhancing the properties of PP, particularly in the development of thermoplastic elastomers with improved performance characteristics.

4. Conclusion

The melt-mixing process of natural rubber (NR) in the polypropylene (PP) matrix successfully resulted in the preparation of a thermoplastic elastomer (TPE) with enhanced mechanical properties. The stress-strain curve analysis indicated a desirable modification of the PP matrix, transforming it from a stiff and strong thermoplastic into a stiff and tough thermoplastic elastomer at a 10% NR inclusion. This modification was further supported by the morphology of the blends observed in scanning electron microscopy (SEM) micrographs, which showed the presence of adhesion sites indicating good compatibility between the PP and NR phases. The incorporation of NR at 10% loading led to a significant improvement in impact strength, with a 47% increase compared to unmodified PP. This enhancement in impact strength is of great importance for applications requiring impact resistance. The successful development of the TPE with improved mechanical properties demonstrates the potential of using NR as a modifier to enhance the performance characteristics of PP. Overall, this study highlights the effectiveness of the melt-mixing process in producing thermoplastic elastomers with desirable properties. The combination of mechanical property improvement, as evidenced by the stress-strain curves and impact strength results, along with the observed compatibility in the SEM micrographs, supports the potential application of PP/NR blends as thermoplastic elastomers in various industries where improved toughness and impact resistance are required.

Materials and methods

Materials

The Polypropylene utilized in this study was obtained from Basell Technology Company B.V. Germany. It was an injection molding grade with a density of 0.90 g/cm^3 and a melt flow rate of 12 g/10 min at 230°C/2.16 kg. The natural rubber (NR) employed was of SMR-L grade and was sourced from Hokson Rubber Trading Company in Malaysia.

Melt-mixing and blend preparation

To achieve the desired blends, a physical mixing process was employed using the Thermo Haake Polylab System Model Rheomix 600 at a rotor speed of 50 rpm and a temperature of 180°C. All blend ratios were determined based on weights and mixed using the same procedure. The completion of mixing was indicated by stabilization in torque within the specified mixing time. The process began by preheating the Polypropylene (PP) in the Haake mixer for 3 minutes. Subsequently, the PP was rotated in the mixing chamber at 50 rpm for an additional 2 minutes. For the preparation of PP/NR thermoplastic elastomers, strips of NR were introduced into the mixing chamber. Each strip of rubber was initially milled on a two-roll mill to form a sheet, which was then cut into strips.

Measurements

The final blends were molded using the Fried S. Carver Inc. compression molding machine, applying a load of 7,500 metric tons and utilizing a 3" diameter ram hydraulic unit. Sheets with a thickness of 1 mm were molded for tensile tests, while specimens for flexural, impact, and hardness tests were all molded at a temperature of 180°C, following the processing

conditions outlined in **Table 4**. The reported pre-heating time refers to the duration required for the sample to soften and become easily flowable.

Table 4. Processing condition for various test specimens

Processing condition	Tensile test specimen	Impact & flexural tests specimens
Pre-heating time (min)	13	15
Compression time (min)	4	4
Temperature (^{O}C)	180	180
Pumping (no. of times)	2	0
Cooling time (min)	5	5

Compression time refers to the minimum duration before the rubber exhibited signs of degradation. The molded samples were cut using a CR Clarke profile cutter 145. Tensile tests were conducted following the ASTM D3039 standard[21] on a LLOYD instruments model LR30Kplus, with a cross head speed of 10 mm/min at room temperature (approximately 25.0 ± 2.00°C). Seven specimens were tested, and the median value was reported. For the three-point bending (flexural) test, also performed at 25.0 ± 2.00°C, the ASTM D790 standard[21] was followed. The LLOYD instruments model LR30Kplus was used, with a cross head speed of 10 mm/min. The median value of seven specimens was reported.

The un-notched Charpy impact test was conducted at 25.0 ± 2.00°C, in accordance with the ISO 179 standard[22]. The Ray Ran universal pendulum impact system, model RR2500, was employed. The median value of 15 specimens was reported. Further details regarding the Charpy impact test conditions can be found in Table 5. The melt flow index (MFI) was determined in accordance with the MS 1267 standard[23]. The Ray Ran advanced microprocessor systems model was used for this purpose.

Table 5. Charpy impact test condition

Parameters	Values
Temperature (^{0}C)	25.0±2.0
Impact velocity (M/s)	2.9
Hammer weight (Kg)	1.189
Energy (Joules)	5.0
Sample width (mm)	9.87±0.5
Sample thickness (mm)	3.91±0.5

Characterization technique

The Ray Ran advanced microprocessor system model RR 3000 was utilized for the Melt Flow Index (MFI) test. The test was conducted at a temperature of 230°C with a load cell of 2.16 kg. A preheat time of 240 seconds was employed, followed by a 10-second "cut-off" time. To ensure accurate measurements, the barrel was thoroughly cleaned before loading the polymer for each successive MFI determination. Scanning Electron Microscopy (SEM) analysis was performed using the Hitachi model S-3400N instrument. The tensile fractured surface of the specimens was sputtered with gold prior to examination. This gold coating helps enhance the surface conductivity and provides better imaging during SEM analysis.

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