MALAYSIAN JOURNAL OF MICROSCOPY
Journal homepage: https://malaysianjournalofmicroscopy.org

RESEARCH ARTICLE

EFFECT OF MICROCRYSTALLINE CELLULOSE LOADING AND TMPTA CROSSLINKING ON THE PROPERTIES OF LOW-DENSITY POLYETHYLENE COMPOSITES

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Abstract. Low-density polyethylene (LDPE) is commonly used in packaging and industrial applications; however, it shows limited stiffness and poor interfacial compatibility with natural fillers. This study explores the integration of microcrystalline cellulose (MCC) sourced from coconut fibre and the use of microwave-assisted crosslinking with 3 wt.% trimethylolpropane triacrylate (TMPTA) to improve the mechanical and structural characteristics of LDPE composites. MCC was incorporated at 0–8 wt.%, and the composites underwent curing for 5 and 15 minutes. Mechanical, morphological, and structural analyses were performed through tensile testing, scanning electron microscopy (SEM), and X-ray diffraction (XRD), with each test repeated three times to ensure statistical reliability. The findings indicated that the composite with 6 wt.% MCC cured for 15 minutes demonstrated the highest tensile strength and elongation at break, due to efficient stress transfer and enhanced matrix–filler adhesion. SEM confirmed a uniform dispersion at this loading, whereas 8 wt.% MCC resulted in fiber agglomeration. The XRD results showed that MCC enhanced crystallinity, while the crosslinking induced by TMPTA resulted in a slight reduction due to restricted chain mobility. The combined effect of MCC reinforcement and controlled microwave-assisted crosslinking resulted in LDPE composites that exhibit enhanced mechanical integrity and structural stability. The optimal formulation for high-performance LDPE-based materials was established at 6 wt.% MCC and 3 wt.% TMPTA with a curing time of 15 minutes.

Keywords: Low-density polyethylene, microcrystalline cellulose, crosslinking, microwave-assisted curing, polymer composites.

Article Info

Received 28 September 2025
Accepted 30 October 2025
Published 4 December 2025

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ISSN: 1823-7010, eISSN: 2600-7444

1. INTRODUCTION

Low-density polyethylene (LDPE) is a commonly employed thermoplastic polymer recognized for its flexibility, durability, and processability. Due to its branching molecular structure formed by high-pressure free-radical polymerization, LDPE provides excellent impact resistance and chemical inertness, rendering it essential in various industrial and packaging applications [1]. Nevertheless, its comparatively poor stiffness and restricted thermal resistance frequently limit its application in structural components, prompting investigations into reinforcing solutions to enhance its mechanical performance [2].

The incorporation of natural fillers like microcrystalline cellulose (MCC) has proven to be an efficient technique for augmenting the stiffness and strength of polymer matrices while minimally adding weight. MCC is a partially depolymerized cellulose derivative derived from lignocellulosic biomass sources, including wood pulp, cotton, and different agricultural by-products. This investigation involved the extraction of MCC from coconut fibre, which is a widespread and often overlooked agricultural by-product found in tropical areas. Coconut fibre stands out as a sustainable and economical alternative to traditional wood- or cotton-derived MCC, boasting a high α-cellulose content, exceptional microfibrillar strength, and advantageous crystallinity. This distinction highlights the originality of the current study, in which MCC derived from coconut fibre is utilized as a sustainable reinforcement in LDPE composites. The extraction of MCC from coconut fiber provides a notable benefit due to its elevated α-cellulose concentration, intrinsic fiber strength, and refined microfibrillar architecture [3]. Prior research indicates that the chemical procedures employed in MCC isolation eliminate hemicellulose and lignin, resulting in a highly crystalline product with enhanced polymer compatibility [4]. The novelty of this study resides in the use of MCC derived from coconut fibre, as opposed to traditional cellulose sources, to reinforce LDPE via microwave-assisted crosslinking, thereby offering performance and sustainability benefits.

The efficacy of cellulose-filled composites is significantly influenced by the interfacial adhesion between hydrophilic microcrystalline cellulose (MCC) and hydrophobic low-density polyethylene (LDPE). To address their incompatibility, crosslinking chemicals have been utilized to establish covalent bonds between polymer chains and filler surfaces. Trimethylolpropane triacrylate (TMPTA) is a versatile acrylate monomer extensively utilized to improve the mechanical strength and dimensional stability of polymer systems [5]. TMPTA can establish robust intermolecular networks that enhance load-bearing capacity and restrict plastic deformation [6]. Upon activation by microwave irradiation, TMPTA facilitates swift and uniform crosslinking, enhancing filler dispersion and stabilizing the polymer matrix [7].

Microwave-assisted crosslinking has garnered interest as a time-efficient and energy-conserving method in contrast to traditional thermal curing. The electromagnetic field produced during irradiation causes localized dipole movement and volumetric heating, facilitating uniform reaction kinetics and enhanced crosslinking efficiency [8]. Optimizing the interplay between MCC loading and TMPTA-induced crosslinking is crucial for attaining an optimal equilibrium of strength, flexibility, and structural integrity. The extent of crosslinking measured by gel content significantly influences the overall characteristics of polymer composites. An increased gel fraction signifies a more compact three-dimensional network, hence improving mechanical and thermal resistance [9]. Conversely, excessive crosslinking may impede chain mobility, resulting in brittleness. Therefore, meticulous regulation of filler composition and curing parameters is essential to customize the ultimate characteristics of the composite material [10,11].

The objective of this study was to examine the enhancement of low-density polyethylene (LDPE) composites by integrating microcrystalline cellulose (MCC) derived from coconut fiber and utilizing TMPTA-induced microwave crosslinking. The study concentrated on analyzing the effects of different MCC loadings and microwave curing parameters on the mechanical, structural, and morphological characteristics of the composites, offering valuable insights for the advancement of high-performance materials applicable in engineering and packaging sectors.

2. MATERIALS AND METHODS

2.1 Materials

Low-density polyethylene (LDPE) pellets with grade LDF200YZ were obtained from Lotte Chemical Titan, Malaysia. The precise grade was unavailable; nonetheless, its melt-flow and density specifications aligned with those of commercial LDPE utilized for injection molding. Coconut peat was sourced from a plantation located in Simpang Empat, Perlis, Malaysia. Sodium hydroxide pellets and sodium chlorite were obtained from Acros Organics (Geel, Belgium), and glacial acetic acid was provided by Merck (Darmstadt, Germany). Trimethylolpropane triacrylate (TMPTA, 3 wt.%) was sourced from UCB Asia Pacific (Malaysia) as the crosslinking agent.

2.2 MCC Preparations

Coconut peat required immersion in water for 24 hours to remove soluble impurities, subsequently being dried in a convection oven at 80 °C for 48 hours. The dried material was subsequently sectioned into smaller pieces to enhance further processing. Following this, the material was processed using a small-scale laboratory grinder (SF-10B model, Zhejiang Tianhe Machinery Co., Ltd., China) and passed through a 150 µm stainless-steel mesh for sieving. The resulting powder was stored in sealed polyethylene bags to prevent moisture absorption.

2.3 Isolation of MCC

The isolation procedure adhered to the alkaline–bleaching method established by Sergi et al. [12], incorporating minor modifications. The ground fiber underwent treatment with 5% w/v NaOH at 80 °C for 2 hours utilizing a magnetic-stirring hot plate. The mixture underwent repeated washing with distilled water until a neutral pH (approximately 7) was attained, followed by drying at 80 °C. Delignification was performed using a 1.7% w/v $NaClO_2$ solution, buffered with CH_3COOH/NaOH (2.7 g NaOH and 7.5 mL acetic acid per 100 mL H_2O), at a temperature of 80 °C for a duration of 4 hours. The treatment was conducted three times until the fibers exhibited a pale yellow to white coloration, signifying the removal of lignin. The purified MCC was subjected to sun-drying and subsequently stored for compounding purposes.

2.4 LDPE/MCC Composite Preparation

A twin-screw extruder (Thermo Haake Rheomex OS) was employed for the compounding of LDPE, MCC, and TMPTA, with barrel temperatures maintained between 175 °C and 180 °C. The extrudates were pelletized and formed into standard tensile bars (ASTM D638 Type V) utilizing an injection-molding machine. Table 1 presents the composite designations.

Table 1: Designation of LDPE/MCC composites

Code	Description
L0	neat LDPE
M0	Pure MCC powder
LM2	LDPE + 2 wt.% MCC (non-crosslinked)
LM6	LDPE + 6 wt.% MCC (non-crosslinked)
LM8	LDPE + 8 wt.% MCC (non-crosslinked)
XLM6/5	LDPE + 6 wt.% MCC + 3 wt.% TMPTA, microwave-cured 5 min (240 W)
XLM6/15	LDPE + 6 wt.% MCC + 3 wt.% TMPTA, microwave-cured 15 min (240 W)

"LM" refers to non-crosslinked LDPE/MCC composites, "XLM" indicates microwave-cured composites, and the numerical values (0–8) signify the weight percentage of MCC within the matrix. TMPTA was utilized at a constant loading of 3 wt.% for the crosslinked samples.

2.5 Crosslink of LDPE/MCC Composite

Crosslinking was performed in a Sharp R-202 commercial microwave oven (2450 MHz, 240 W) equipped with a rotating turntable to ensure uniform heating. Each composite was irradiated for either 5 min (XLM6/5) or 15 min (XLM6/15). After curing, specimens were cooled to ambient temperature and conditioned at 23 °C and 50 % RH for 24 h prior to testing.

2.6 Characterisation of LDPE/MCC Composite

2.6.1 Determining of Degree of Crosslinking

The solvent extraction method is commonly used to evaluate the degree of crosslinking in LDPE/MCC composites containing TMPTA. The cured composite sample is cut into five small pieces of a specified weight and subjected to reflux in boiling xylene at approximately 130–140 °C for a period of 24 hours. The solvent efficiently dissolves the non-crosslinked (soluble) fraction, whereas the crosslinked (insoluble) network is maintained. After extraction, the residue is filtered, dried in a vacuum oven until a constant weight is reached, and then weighed again [13]. The degree of crosslinking is quantified as gel content, determined by the following formula:

$$\text{Gel content (\%)} = (W_1 / W_0) \times 100, \tag{1}$$

Where, W_0 represents the initial dry weight of the sample prior to solvent extraction, while W_1 indicate the dry weight of the insoluble residue subsequent to extraction and drying.

2.6.2 Tensile Test

Tensile properties were measured using an Instron 5567 Universal Testing Machine fitted with a 1 kN load cell at a crosshead speed of 50 mm min^{-1}, following ASTM D638. Five replicate specimens per formulation were tested, and average results are reported. The tensile strength and elongation at break were obtained through the test.

2.6.3 Scanning Electron Microscope(SEM)

Fractured surfaces of tensile specimens were sputter-coated with gold utilizing an Autofine coater to reduce charging effects. Micrographs were acquired using a JEOL JSM-6390LV scanning electron microscope at 10 kV with a magnification of 500x.

2.6.4 X-Ray Diffraction (XRD)

Crystalline phases were analysed using a Bruker D2 Phaser diffractometer (Cu Kα, λ = 1.5406 Å, 40 kV, 30 mA) with 2θ ranging from 10° to 40° at 2° min^{-1}. The crystallinity index (CI) was determined using the Segal Equation [14].

$$CI\ (\%) = \left[\frac{I_{002} - I_{AM}}{I_{002}}\right] x100 \tag{2}$$

where I_{002} is the highest peak intensity for the crystalline region, and I_{AM} is the minimum peak intensity for the amorphous region. I_{002} represents both crystalline and amorphous regions, while I_{AM} represents the amorphous regions only.

3. RESULTS AND DISCUSSION

3.1 Degree of Crosslinking

Table 2 presents the gel content of the LDPE/MCC composites. The increase in gel content for microwave-cured samples (XLM6/5 and XLM6/15) suggests crosslinking promoted by TMPTA. The extension of irradiation time from 5 minutes to 15 minutes led to a slight increase in gel fraction, indicating a higher crosslink density due to the prolonged exposure duration. The observed enhancement is attributable to the increased formation of free-radical sites on the LDPE chains due to microwave curing [15]. The non-crosslinked composites (LM series) exhibited minimal gel content, suggesting the absence of network formation. The lack of gel-content testing for XLM6/5 and XLM6/15 in the initial submission represents a significant limitation, which has been acknowledged and discussed in later communications. Minor crosslinking may occur in LDPE due to chain scission and recombination under microwave exposure; nevertheless, significant network development was seen exclusively in the presence of TMPTA, hence affirming its essential function as the principal crosslinking agent. The observed increase in gel fraction is associated with the improved tensile strength and crystallinity addressed later in this section [16,17].

Table 2: Crosslinking degree of LDPE/MCC composites

Sample Code	Gel Content (%)
L0	61.5
LM2	62.4
LM4	62.6
LM6	63.5
LM8	60.7
XLM6/5	64.4
XLM6/15	65.3

3.2 Tensile and Morphological Properties

Figure 1 illustrates the tensile strength of LDPE/MCC composites in relation to MCC content. The tensile strength increased with the incorporation of MCC, peaking at 6 wt.% (LM6), before declining at 8 wt.% (LM8). This trend results from the combined influence of filler–matrix interactions and the dispersion of fillers. At a moderate loading of 6 wt.%, the well-dispersed MCC particles served as effective stress-transfer sites, attributed to enhanced interfacial adhesion with the LDPE matrix. The hydroxyl groups on MCC surfaces may interact with the polar acrylate groups of TMPTA or with LDPE chain ends, facilitating mechanical interlocking and partial hydrogen bonding. This led to an effective distribution of stress when subjected to load. At elevated filler content (8 wt.%), agglomeration of MCC particles was observed, as evidenced by the SEM micrographs. The aggregation of rigid particles created microvoids and stress concentrations, which compromised the continuity of the matrix, resulting in early failure and diminished tensile performance. Comparable agglomeration-induced weakening has been documented in other cellulose-filled thermoplastics [18,19].

The mechanical performance was further enhanced by microwave-assisted crosslinking with TMPTA. The XLM6/5 and XLM6/15 composites demonstrated superior tensile strength compared to their non-crosslinked counterparts, indicating that TMPTA facilitated covalent bond formation among

LDPE chains. The extended curing duration of 15 minutes resulted in increased crosslink density, which restricted chain slippage during deformation and led to improved tensile strength. The observed enhancement indicates that the synergistic effects of physical reinforcement from MCC and chemical crosslinking (TMPTA) play a significant role in fortifying the polymer network.

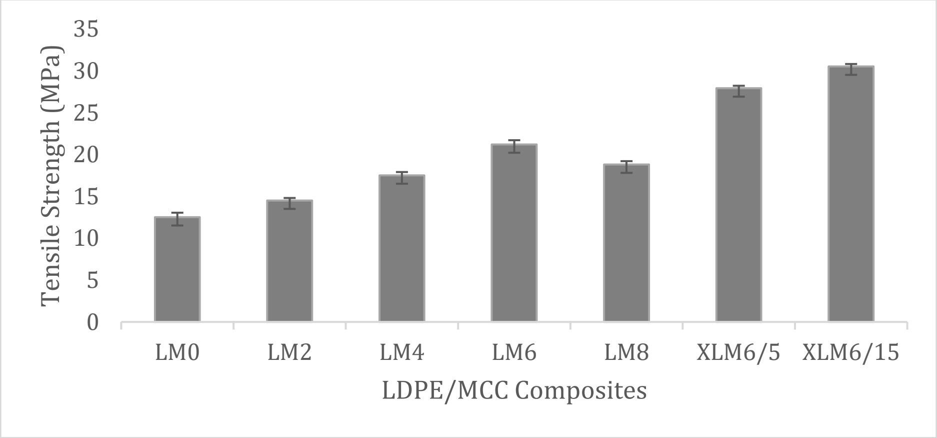

Figure 1: The tensile strength of MCC/LDPE composites with varying TMPTA weight percentage

The elongation at break diminished with the addition of MCC, indicating limited chain mobility as a result of the rigid filler (Figure 2). However, the reduction was less significant in the crosslinked samples, indicating that networks generated by TMPTA facilitated elastic recovery and mitigated brittle fracture. The equilibrium between stiffness and ductility underscores the necessity of optimizing filler content and crosslinking duration.

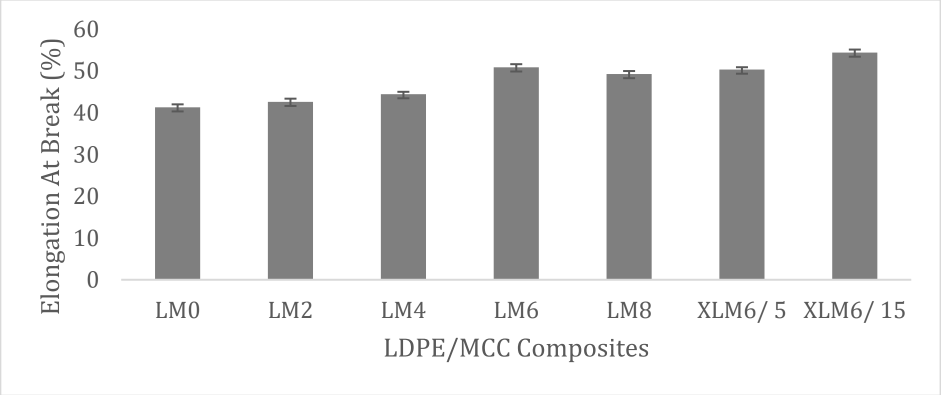

Figure 2: The elongation at break of MCC/LDPE composites with varying TMPTA weight percentage

SEM observations support these findings (Figure 3). Pure LDPE (L0) displayed a smooth fracture surface characteristic of ductile polymers. Conversely, LM6 exhibited a rougher topography with consistent MCC dispersion, indicating effective stress transfer at the interface. XLM6/15 exhibited a more cohesive surface, suggesting enhanced interfacial bonding and increased crosslink density. In contrast, LM8 exhibited fibre clustering and micro voids, which served as sites for crack initiation under stress. The morphological evolution corroborates the mechanical data, illustrating the influence of microstructure on macroscopic strength and flexibility. The SEM observations align with the mechanical results, indicating that well-dispersed MCC (≤ 6 wt.%) improved interfacial bonding. In contrast, higher loading resulted in filler–filler interactions and the formation of microvoids. Han et al. [18] reported similar trends for LDPE/cellulose composites, as did El-Aal et al. [19] for microwave-cured biopolymer systems.

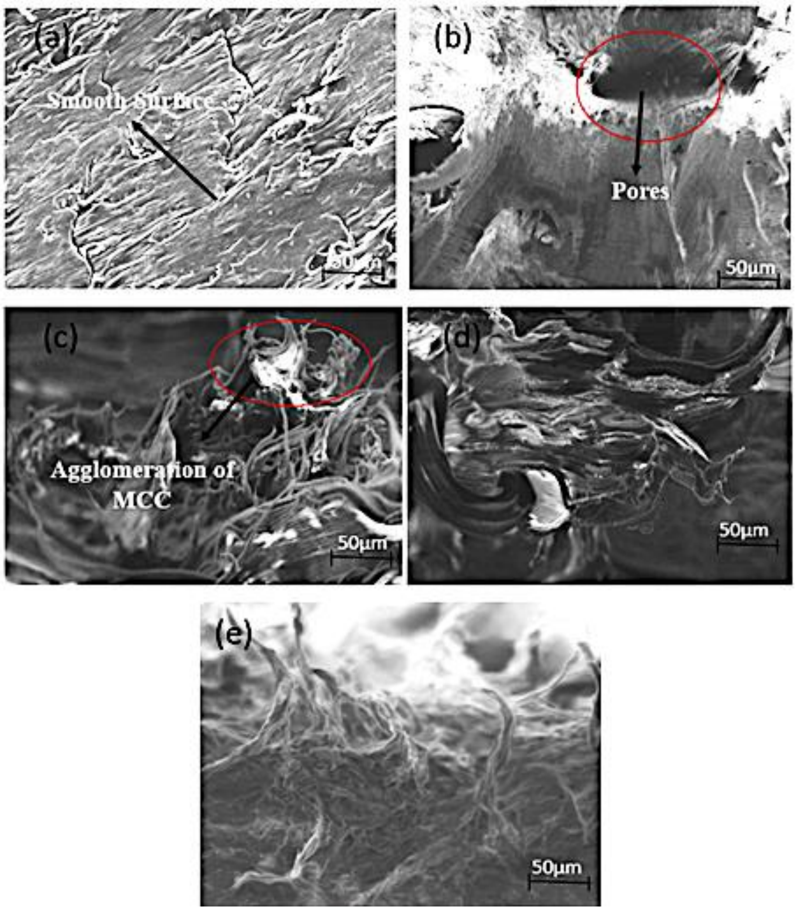

Figure 3: SEM micrographs of (a) L0, (b) LM6, (c) XLM6/5, (d) XLM6/15 and (e) LM8%

The morphological and tensile results align with the crystallinity trends outlined in the following XRD analysis, which indicates improved molecular ordering and more defined diffraction peaks for the optimally crosslinked composite (XLM6/15). The observed correlation indicates that enhanced interfacial adhesion and increased crosslink density contribute to both elevated mechanical strength and improved structural regularity within the LDPE/MCC matrix.

3.3 XRD Analysis

Figure 4 illustrates the XRD patterns corresponding to LDPE, MCC, and their composite materials. Pure LDPE displayed distinct peaks at 2θ = 21.6° and 23.8°, which are associated with the (110) and (200) planes of orthorhombic polyethylene. The incorporation of MCC resulted in the emergence of an additional broad peak at approximately 22.5°, which is associated with the crystalline regions of cellulose I.

The intensity of LDPE peaks exhibited a slight increase with the incorporation of MCC up to 6 wt.%, suggesting a nucleating effect of cellulose on the crystallization of the polymer. At 8 wt.% MCC, the peaks exhibited reduced distinctness, indicating that particle agglomeration impeded the formation of crystallites. The intensity of the crystalline peaks for the microwave-cured composites increased further, especially for XLM6/15, indicating enhanced molecular ordering as a result of TMPTA-induced crosslinking [20].

The crystallinity index (CI) shown in Table 3, calculated using the Segal method, increased from 53.2% for coconut peat powder (M0) to 75.5% for LM8, suggesting that the addition of MCC to the LDPE matrix improves crystalline ordering. The enhancement observed can be linked to the nucleating effect of MCC, which aids in the alignment of LDPE chains throughout the crystallization process. Nonetheless, a significant reduction in CI was noted in the crosslinked composites, with XLM6/5 and XLM6/15 displaying values of 50.7% and 49.4%, respectively. The decrease in crystallinity correlates with the development of crosslinks that limit the mobility of polymer chains and obstruct the expansion of crystalline domains.

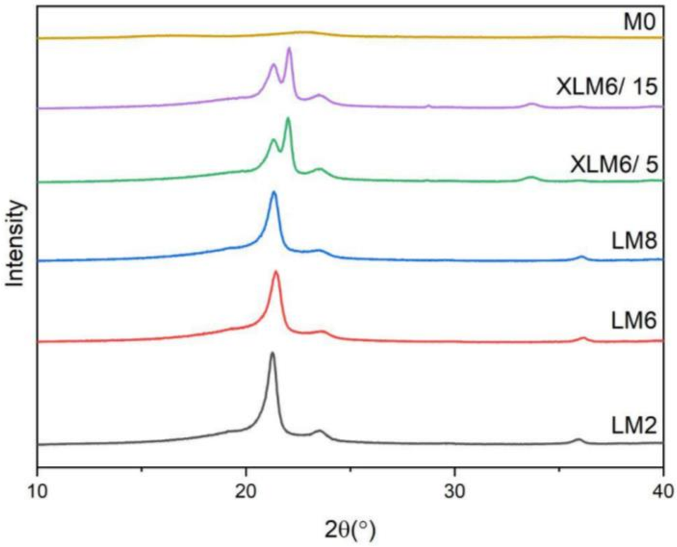

Figure 4: XRD diffractogram of MCC (M0), non-crosslinked (LM series), and crosslinked (XLM series) LDPE/MCC composites

Table 3: Crystallinity percentage of LDPE/MCC and different TMPTA curing time

Sample	Crystallity Index
M0	53.2%
LM2	72.4%
LM6	73.5%
LM8	75.5%
XLM6/ 5	50.7%
XLM6/ 15	49.4%

4. CONCLUSIONS

This study revealed that the integration of microcrystalline cellulose (MCC) sourced from coconut fiber into LDPE, along with 3 wt.% TMPTA during microwave curing, significantly improved the composite's performance. The MCC/LDPE composite film with 6 wt.% MCC cured for 15 minutes had the best tensile strength and elongation, attributed to greater filler dispersion and matrix–filler adhesion. Additionally, MCC increased crystallinity, while TMPTA-induced crosslinking improved structural stability by restricting chain mobility. Nonetheless, as the mechanical tests excluded pristine LDPE (0 wt.% MCC), a direct quantitative comparison with pure LDPE was unfeasible. Consequently, although the findings evidently indicate positive reinforcing and crosslinking effects, the addition of a pure LDPE control is essential to comprehensively clarify the inherent impact of MCC on the mechanical properties of LDPE

Acknowledgements

The authors wish to express their sincere gratitude to the Toyohashi University of Technology for its facilities and testing support.

Author Contributions

All authors made substantial contributions to the conception and design of the study, data acquisition and analysis, and the drafting and critical revision of the manuscript. All authors have read and approved the final version of the manuscript and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors declare that there are no conflicts of interest associated with this publication.

Compliance with Ethical Standards

This study was conducted in accordance with all applicable ethical standards and institutional guidelines.

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