Malaysian Journal of Chemistry, 2026, Vol. 28(2), 329-338
(Special Issue: *1st International Conference on Chemical and Energy Engineering 2025, ICChEE2025*)

Sustainable Vulcanization of Natural Rubber Latex using Organic Peroxides as Accelerator-Free Alternatives for Improved Crosslinking and Mechanical Performance

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This study presents a sustainable approach to the vulcanization of natural rubber latex (NRL) films by eliminating conventional accelerators, which are known to produce nitrosamine by-products associated with health and environmental hazards. Instead, long-chain organic peroxides—dicumyl peroxide (DCP) and 2,5-dimethyl-2,5-di(tert-butylperoxy) hexane (DHBP)—were utilized as accelerator-free curing agents. Latex compounds were prepared and subjected to detailed physicochemical and mechanical characterization, including swelling index, total solid content, pH, specific gravity, tensile strength, elongation at break, and modulus, along with FTIR analysis to assess the crosslinking behavior. The results indicate that DHBP outperformed DCP in promoting crosslink density and mechanical performance. At a concentration of 2 phr, DHBP showed a swelling index of 80%, total solid content of 29.03%, pH of 9.15, and specific gravity of 0.98. It also achieved superior mechanical performance, with a tensile strength of 14.26 MPa, elongation at break of 863.81%, and a modulus of 0.74 MPa. In comparison, DCP at the same concentration resulted in a swelling index of 82%, total solid content of 31.35%, pH of 8.95, specific gravity of 0.97, tensile strength of 12.37 MPa, elongation of 873.98%, and modulus of 0.86 MPa. These findings support the viability of organic peroxide-based vulcanization systems as environmentally friendly alternatives to traditional accelerators. By eliminating accelerator-derived nitrosamines, this approach contributes to safer industrial processing and aligns with the global shift toward green and sustainable rubber manufacturing. The study provides valuable insights for the development of next-generation NRL films tailored for applications requiring reduced toxicological impact without compromising material performance.

Keywords: Natural rubber latex, organic peroxides, accelerator-free, vulcanization, sustainability

Received: July 2025; Accepted: February 2026

Natural rubber latex (NRL) is a renewable and biodegradable biopolymer derived from the latex of Hevea brasiliensis, a milky exudate tapped from the tree bark [1]. This latex is composed of rubber hydrocarbon particles suspended in an aqueous medium enriched with proteins, carbohydrates, lipids, and other organic and inorganic compounds. The rubber particles, which typically constitute 25% to 45% of the latex, contribute significantly to the elasticity and mechanical resilience of NRL-based products [2]. Geographically, more than 80% of global natural rubber production is concentrated in Southeast Asia, notably Malaysia, Indonesia, and Thailand, where favorable agroclimatic conditions and established plantation practices support the supply chain [3].

NRL films are widely used in medical gloves, balloons, and industrial coatings due to their excellent mechanical performance. Conventionally, these films are produced through sulfur vulcanization in the presence of chemical accelerators. Accelerators such as zinc dibutyldithiocarbamate (ZDBC) play a crucial role in promoting crosslinking between polymer chains during curing, significantly improving the tensile strength, elasticity, and durability of the final product [4,5]. However, this conventional system has raised critical sustainability and safety issues. The thermal decomposition of accelerators during vulcanization leads to the formation of nitrosamines and other potentially hazardous by-products, many of which are classified as carcinogenic and sensitizing agents [6,7]. This presents significant

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†Paper presented at the 1st International Conference on Chemical and Energy Engineering 2025 (ICChEE2025)

risks to workers and users and contributesto the environmental burden of rubber product manufacturing.

In light of growing global demands for cleaner production systems and safer occupational practices, peroxide-based vulcanization has emerged as a viable accelerator-free alternative. Organic peroxides, including dicumyl peroxide (DCP) and 2,5-dimethyl-2,5-di(tert-butylperoxy) hexane (DHBP), act as free-radical initiators, enabling efficient crosslinking without the use of accelerators [8,9]. These compounds decompose at controlled temperatures to generate radicals that initiate crosslinking reactions, reducing nitrosamine exposure and improving environmental safety. The successful application of such peroxides depends on precise thermal control to ensure complete water removal and to avoid degradation of the NRL matrix [10].

Despite these advantages, challenges remain in achieving mechanical performance on par with accelerator-based systems. According to ASTM D3577-01a, natural rubber latex cured solely by peroxide vulcanization often exhibits reduced tensile strength [11,12]. Therefore, the present study focuses on optimizing peroxide vulcanization in accelerator-free NRL films. The primary objective is to formulate and characterize latex films using DCP and DHBP at varying concentrations, and to evaluate their physicochemical and mechanical properties—specifically swelling index, total solid content, pH, and tensile strength. By identifying effective peroxide formulations, this research supports the development of high-quality, environmentally friendly rubber materials that align with sustainable production goals and promote safer manufacturing practices in the latex industry.

EXPERIMENTAL

Chemicals and Materials

High-ammonia natural rubber latex (HA-NRL, 60% total solid content) was obtained from Sri Trang, Thailand. The organic peroxides used as vulcanizing agents were dicumyl peroxide (DCP, $C_{18}H_{22}O_2$) and 2,5-dimethyl-2,5-di(tert-butylperoxy) hexane (DHBP, $C_{16}H_{34}O_4$), both procured from Sigma-Aldrich with ≥97% purity. Other analytical-grade reagents included zinc oxide (ZnO), zinc dibutyldithiocarbamate (ZDBC), sulphur, potassium hydroxide (KOH), titanium dioxide (TiO_2), calcium carbonate, calcium nitrate, and wetting agents. All chemicals used were analytical grade to ensure experimental consistency.

Sustainable Formulation of Latex Compounds

Formulation of the accelerator-free latex films, six sets of compounds were prepared (Table 1). The control sample (Set 0) included 2 phr of ZDBC. Set 1 contained no accelerator, while Sets 2 and 3 were formulated with 1.5 and 2.0 phr of DHBP. Sets 4 and 5 used 1.5 and 2.0 phr of DCP, respectively. All latex mixtures were stirred at ambient temperature for 36 hours to ensure homogeneity. The chemical dosages were calculated based on the phr (parts per hundred rubber) method using Equation (1) to ensure minimal excess and reduce chemical waste:

$$\text{Weight of chemicals, g} = \frac{W_L \times TSC_L \times phr_C}{TSC_C/100} \qquad (1)$$

where W_L is the weight of latex (g), TSC_L is the Total Solid Content of latex (%), phr_C is the part per rubber of chemical used and TSC_C is the Total Solid Content of chemical (%).

Table 1. Formulation of natural rubber accelerator free latex compound.

Chemical	Function	Set					
		Control (g) (2 phr ZDBC)	Set 1 (g) (no accelerator)	Set 2 (g) (1.5 phr DHBP)	Set 3 (g) (2 phr DHBP)	Set 4 (g) (1.5 phr DCP)	Set 5 (g) (2 phr DCP)
HA NRL	Raw material	500	500	500	500	500	500
KOH	Stabilizer	1.8	1.8	1.8	1.8	1.8	1.8
Sulphur	Vulcanizing agent	4.5	4.5	4.5	4.5	4.5	4.5
ZnO	Activator	3	3	3	3	3	3
DHBP	Organic peroxide	-	-	18	24	-	-
DCP	Organic peroxide	-	-	-	-	18	24
ZDBC	Accelerator	24	-	-	-	-	-
TiO2	Pigment	0.43	0.43	0.43	0.43	0.43	0.43
Antioxidant	-	6	6	6	6	6	6

Characterization of Latex Compound

To evaluate the efficiency of peroxide-induced crosslinking and its implications on material performance and environmental sustainability, a comprehensive set of physicochemical and structural analyses were performed. The physicochemical properties (TSC, pH, and specific gravity) were measured on compounded latex prior to curing, whereas swelling index, FTIR, and mechanical testing were performed on fully cured latex films [14].

Swelling Index (SI)

The degree of crosslinking within the latex compound was assessed via a toluene swelling test, which indirectly quantifies the crosslink density of vulcanized films. Circular specimens (3.0 cm diameter) were precisely cut using a metal die to ensure uniform surface area and reproducibility. Each sample was immersed in reagent-grade toluene at room temperature (25 ± 2 °C).

Swelling was allowed to occur for 30 minutes in a static condition without agitation to simulate passive solvent uptake. After immersion, final diameters of the samples were measured using graph paper. The swelling index was calculated using the following equation 2:

$$\mathrm{SI\%} = \frac{\mathrm{D_F - D_I}}{\mathrm{D_F}} \times 100\ \% \qquad (2)$$

where D_I is the initial diameter (cm) and D_F is the final diameter post-swelling. A lower SI value indicates a higher crosslink density, which is desirable for improved solvent resistance and material durability.

Total Solid Content (TSC)

TSC is a critical indicator of latex compound formulation consistency and determines the actual rubber content present during the compounding phase. A 0.5 g sample of each latex compound was dispensed onto pre-weighed aluminum foil dishes (3 replicates per formulation), which had been pre-dried and cooled to room temperature in a desiccator. Samples were oven-dried at 150 °C for 20 minutes to remove moisture and volatiles. The dried samples were cooled again in a desiccator for 15 minutes before final weighing.

$$\mathrm{TSC\ \%} = \frac{\mathrm{c-a}}{\mathrm{b}} \times 100\ \% \qquad (3)$$

where a = weight of foil (g), b = weight of wet latex (g), and c = final weight of foil plus dry residue (g). All weights were measured using an analytical balance with 0.0001 g sensitivity.

pH Measurement

To assess the colloidal stability of the latex compounds, the pH was measured directly using a benchtop digital pH meter (Mettler Toledo, FiveEasy Plus) calibrated with standard buffer solutions (pH 4.00, 7.00, and 10.00). Measurements were conducted at ambient temperature in triplicate for each formulation to ensure statistical accuracy. Maintaining an optimal pH is crucial for preserving latex dispersion, reducing coagulant waste, and promoting uniform film formation.

Specific Gravity

Specific gravity was determined using a stainless-steel pycnometer (capacity: 100 mL), filled with each latex compound at 25 °C. The method followed ASTM D891, with density calculated by dividing the net mass of latex by the volume of the pycnometer. Results were corrected for temperature using water as a calibration reference. Specific gravity data helps predict sedimentation tendencies and optimize transportation and storage efficiency of latex formulations.

Fourier Transform Infrared Spectroscopy (FTIR)

To evaluate chemical structure changes due to peroxide-induced crosslinking, FTIR spectra were acquired using a PerkinElmer Paragon 1000 spectrophotometer operating in the 4000–500 cm^{-1} range. Each film was scanned using Attenuated Total Reflectance (ATR) mode to avoid additional sample preparation and solvent usage. Spectra were used to identify characteristic bands for –OH, –CH, –C=O, and –C–O–C– groups, which provide insight into the success of vulcanization and any potential degradation pathways. Only representative films from each formulation group were analyzed to minimize resource consumption.

Dipping Process

Mold preparation and film dipping followed an environmentally conscious, small-scale batch process. Molds were first cleaned, dried at 70 °C, and cooled to 55 °C before immersion into a preheated coagulant solution containing calcium nitrate for 30 seconds. After pre-curing at 120 °C for 1–2 minutes, the molds were cooled and dipped into the latex compound for 20 seconds. Film thickness was maintained between 0.3 and 0.5 mm.

The dipped molds were then dried at 110 °C for 1–2 minutes, followed by pre-leaching in 60 °C water for 1–2 minutes to remove unreacted materials. Final curing was conducted at 110 °C for 25 minutes. Once cooled, the formed films were stripped and lightly powdered with corn starch to prevent sticking.

Mechanical Testing of Latex Film

Mechanical performance of the latex films was assessed using an Instron Universal Testing Machine according to ISO 11193. Samples were cut into dumbbell shapes and tested at 25 °C with a crosshead speed of 500 mm/min. The tensile strength, elongation at break, and modulus at 300% elongation were recorded to evaluate the influence of peroxide type and concentration on the film's mechanical integrity.

RESULTS AND DISCUSSION

Physicochemical Properties of Compounded Latex

In order to understand the effect of peroxide-based formulations on natural rubber latex, it is important to evaluate the physicochemical properties of the compounded latex. These properties, including total solid content, pH, and specific gravity, provide insights into latex stability, processability, and suitability for film formation, which are critical for ensuring consistent dipping and curing performance as per stated by Vaysse et. al. [2].

Swelling Index (SI) and Crosslink Density

The swelling index (SI) results are presented in Table 3. The control set (2 phr ZDBC) exhibited a swelling index of 86%, serving as a baseline indicative of moderate crosslink density. Sets incorporating peroxide-based vulcanizing agents displayed notable variations: Set 3 (2 phr DHBP) recorded the lowest SI (80%), indicating the highest crosslink density and optimal solvent resistance. Similarly, Set 5 (2 phr DCP) showed improved crosslink density (82%) compared to lower peroxide concentrations (Set 2: 91%, Set 4: 94%). These findings emphasize that higher peroxide concentrations, particularly DHBP, significantly enhance crosslinking efficiency and durability, supporting sustainable latex film production by reducing reliance on conventional accelerators and mitigating harmful by-product formation.

Total Solid Content (TSC)

Table 3 illustrates the total solid content (TSC) across formulations, reflecting compound stability and processing efficiency. The highest TSC (36.27%) was recorded for Set 4 (1.5 phr DCP), suggesting optimal compounding at lower peroxide concentrations. The control set demonstrated a moderately high TSC (35.49%), while Sets 3 (2 phr DHBP) and 2 (1.5 phr DHBP) exhibited lower TSC values (29.03% and 30.75%, respectively). This trend indicates that increased peroxide concentrations may slightly reduce TSC, likely due to peroxide interaction and minor volatility during mixing. Optimizing TSC contributes to sustainable practices by enhancing material utilization and reducing wastage.

pH and Colloidal Stability

The pH values (Table 3) provide insights into colloidal stability critical for sustainable latex processing. The control set maintained a slightly alkaline pH of 8.88, while accelerator-free formulations exhibited increased alkalinity (Set 1: 9.65, Set 2: 9.32, Set 4: 9.58). Notably, increasing peroxide concentrations lowered pH values moderately (Set 3: 9.15, Set 5: 8.95), indicating mild chemical interactions due to peroxide decomposition without negatively impacting latex stability. Stable and predictable pH conditions align with sustainable manufacturing goals by minimizing chemical additives required to adjust colloidal properties during processing.

Specific Gravity

Specific gravity results (Table 2) were consistent across all formulations, ranging narrowly between 0.97–0.98. Such uniformity confirms effective compound dispersion, reproducibility, and material consistency. Stable specific gravity values support sustainable manufacturing by ensuring predictable handling, storage, and processing conditions, minimizing material losses, and enhancing process control.

Table 2. Physicochemical properties of compounded latex.

Properties	Control set (2 phr ZDBC)	Set 1 (no accelerator)	Set 2 (1.5 phr DHBP)	Set 3 (2 phr DHBP)	Set 4 (1.5 phr DCP)	Set 5 (2 phr DCP)
Swelling Index % (before maturation)	186	157	157	146	129	137
Swelling Index % (after maturation of 36 hours)	86	94	91	80	94	82
Total solid content (%)	35.49	33.61	30.75	29.03	36.27	31.35
pH	8.88	9.65	9.32	9.15	9.58	8.95
Specific gravity	0.97	0.98	0.98	0.98	0.98	0.97

Structural Analysis of Latex Compounds

FTIR Analysis of Latex Compounds

The FTIR spectra (Figure 1) clearly illustrate the differences in chemical structures resulting from various curing systems, particularly between accelerator-based (ZDBC control) and accelerator-free (DHBP and DCP) formulations. The findings were compared directly with previous research by Yahya et al. [13] to affirm peak identifications and functional group transformations.

The control sample (ZDBC) exhibited characteristic absorption bands consistent with the natural rubber backbone, including prominent peaks around 3015–2744 cm⁻¹ corresponding to asymmetric and symmetric C–H stretching vibrations (–CH_2– and –CH_3). Additionally, the peaks within 1488–1398 cm⁻¹ corresponding to C–H bending vibrations were present. Peaks representing cis-1,4-polyisoprene, the primary constituent of natural rubber, appeared distinctly within 870–780 cm⁻¹. Furthermore, the broad absorption band observed between 3567–3157 cm⁻¹ represented hydroxyl (–OH) groups inherent in natural rubber latex, possibly related to moisture or residual stabilizers [13]. Although these peaks indicate baseline functionality, the presence of ZDBC as an accelerator is linked to potential nitrosamine generation, presenting significant environmental and occupational health concerns [1,14].

In contrast, notable spectral variations were observed for peroxide-cured formulations, particularly for Set 3 (2 phr DHBP) and Set 5 (2 phr DCP). A sharper, more pronounced absorption around 1600 cm⁻¹ corresponding to C=C stretching vibrations emerged, signaling effective peroxide-induced crosslinking and polymer network formation. Specifically, Set 3 (DHBP-based) demonstrated significantly intensified peaks, suggesting enhanced crosslink density due to efficient radical generation by DHBP. Similarly, Set 5 (DCP-based) exhibited intensified C=C stretching vibrations, though slightly less pronounced compared to DHBP, suggesting effective but comparatively lower radical reactivity [15, 16].

Furthermore, peroxide-treated samples exhibited clearer peaks in the range of 1488–1398 cm⁻¹ (C–H bending vibrations), indicating structural changes related to enhanced crosslink density and chain scission. Such changes reflect peroxide-driven radical reactions, which effectively modified polymer networks, enhancing mechanical integrity while significantly reducing harmful by-product formation common in accelerator-based systems.

Set 1 (no accelerator) served effectively as a baseline, illustrating minimal crosslinking and confirming the critical role of curing agents. In comparison, peroxide-based systems clearly demonstrated superior crosslinking efficiency. Notably, DHBP's unique molecular structure allowed more controlled radical initiation, resulting in optimal crosslink networks as reflected in spectral intensities. These observations align closely with previous literature emphasizing DHBP's efficacy and sustainability benefits as a vulcanizing agent in natural rubber systems [17, 18].

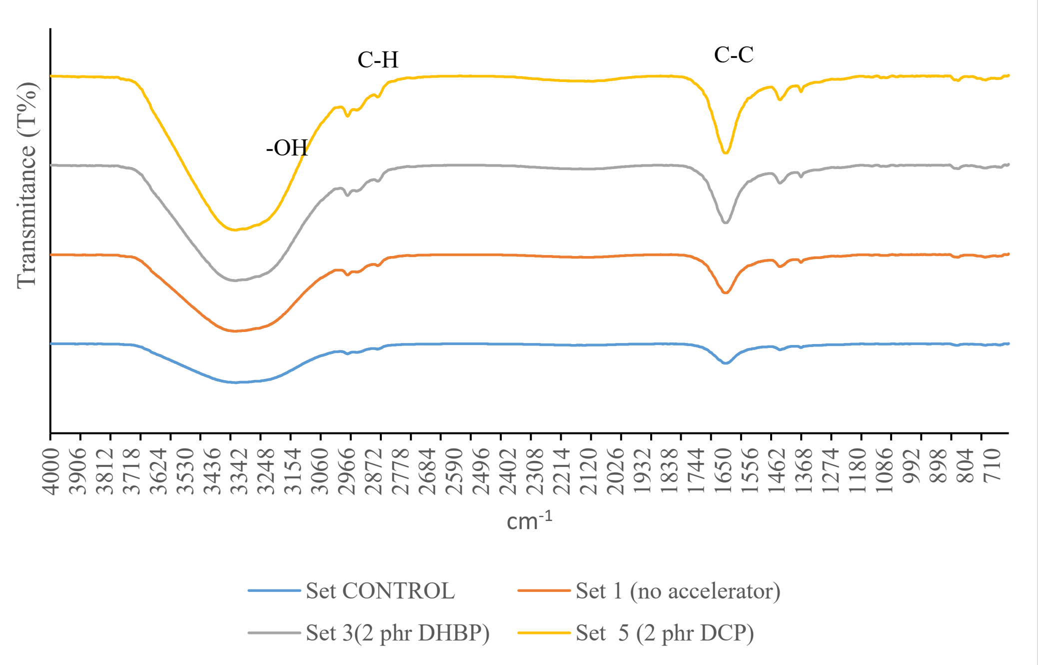

Figure 1. FTIR analysis measurements of natural rubber liquid with control (ZDBC), DHBP and DCP curing systems.

In summary, the FTIR analysis concretely demonstrated that DHBP and DCP induce effective crosslinking while significantly reducing environmental and occupational hazards associated with ZDBC accelerators. DHBP emerged as the most effective sustainable alternative, delivering enhanced polymer integrity through controlled radical initiation and reduced harmful emissions. This reinforces the viability of organic peroxides, particularly DHBP, as critical components for sustainable and safe natural rubber latex film production.

Mechanical Properties of Latex Films

The mechanical performance of cured natural rubber latex films is directly affected by the crosslink density and network structure established during peroxide type vulcanization. Key properties such as tensile strength, elongation at break, and modulus provide critical information on film durability, flexibility, and suitability for practical applications. These properties are widely used to evaluate the efficiency of curing systems and to correlate crosslink density with film performance as also supported by Kruželák et al. [10] and Coran [15].

Tensile Strength

Tensile strength is a crucial indicator of a polymer film's structural integrity and durability, directly influencing the product's lifecycle and usability in real-world applications. The tensile strength analysis presented in Figure 2 demonstrates substantial variations between different curing systems, reflecting the efficiency and sustainability potential of peroxide-based curing agents versus conventional accelerators [19].

The DHBP formulation at 2 phr (Set 3) exhibited the highest tensile strength of 14.26 MPa, significantly surpassing the conventional ZDBC-based control set (11.48 MPa). This notable improvement can be attributed to DHBP's highly efficient radical initiation, facilitating more uniform and effective crosslink formation. Such robust crosslinking not only enhances the mechanical performance of natural rubber latex films but also contributes directly to product durability, reducing the frequency of replacements and, consequently, material waste-an essential factor in sustainability-focused manufacturing.

The DCP formulation at 2 phr (Set 5), while slightly lower at 12.37 MPa compared to DHBP, still demonstrated appreciable tensile performance improvements relative to conventional accelerator systems. Although DCP provided less pronounced radical reactivity and a marginally lower crosslink density compared to DHBP, the resulting strength indicates its suitability as a viable and more sustainable alternative to traditional accelerators. By employing DCP or DHBP, manufacturers can significantly reduce reliance on hazardous compounds such as ZDBC, which are linked to nitrosamine emissions and associated environmental and health risks [20].

Moreover, transitioning toward peroxide-curing systems aligns with circular economy principles by extending product life cycles and reducing environmental impact. Enhanced tensile strength in peroxide-cured films implies reduced susceptibility to tearing and mechanical failure, crucial for applications like medical gloves and industrial films, thereby reducing disposal rates and promoting material efficiency. Furthermore, by mitigating harmful chemical by-products typically associated with accelerator-based curing, these organic peroxides substantially lower occupational health risks and environmental pollutant load, directly contributing to safer, cleaner production practices [21].

Overall, the data affirm that DHBP provides superior performance in balancing mechanical robustness with significant sustainability advantages. Its adoption within latex film manufacturing processes could serve as a strategic move toward achieving higher product quality, extended product lifetimes, safer work environments, and reduced environmental footprint-objectives central to the goals of sustainable production and consumption.

Modulus at 300% Elongation

Modulus results at 300% elongation (Figure 3) illustrated the relationship between crosslinking agents and material stiffness. The ZDBC-based control displayed the highest modulus (1.79 MPa), indicative of a dense, rigid crosslinking network. Conversely, peroxide-based films (DHBP and DCP) exhibited lower modulus values (0.61–0.86 MPa), suggesting a more flexible and less rigid network structure. Such flexibility aligns with sustainable product applications that require moderate stiffness balanced with elasticity. Particularly, DHBP at 1.5 phr (0.82 MPa) provided an optimal balance suitable for sustainable rubber film applications.

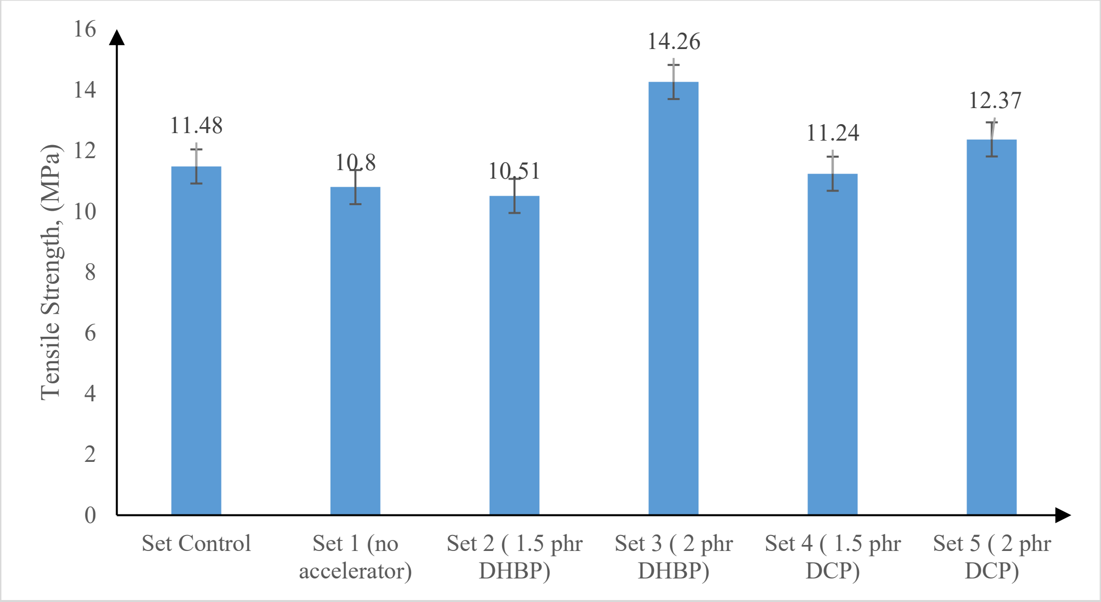

Figure 2. Tensile strength of natural rubber cured with ZDBC, DHBP and DCP.

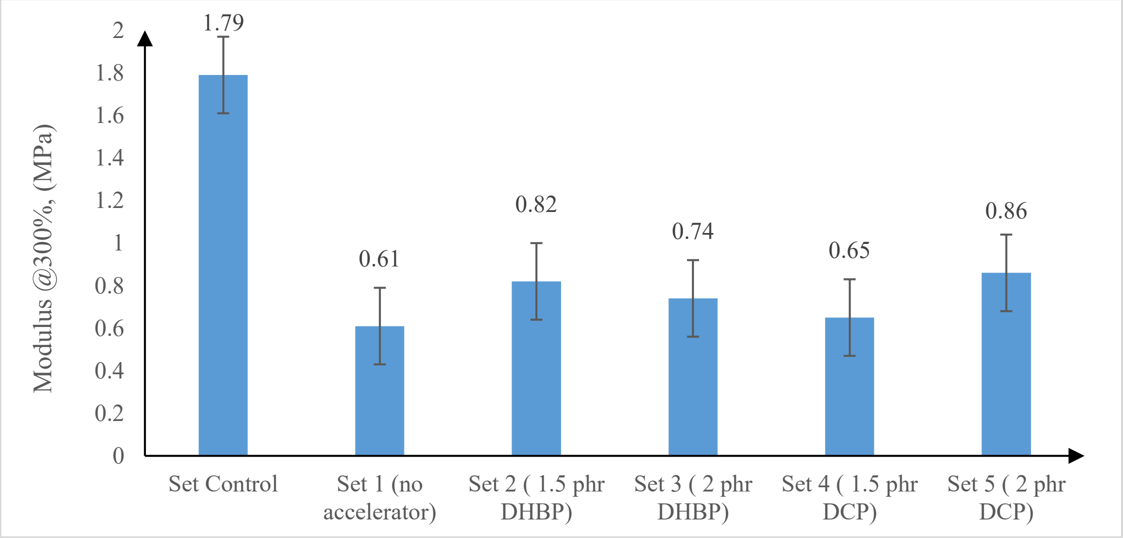

Figure 3. Tensile modulus values of natural rubber cured with ZDBC, DHBP and DCP.

Elongation at Break

Elongation at break (Figure 4) assesses material flexibility, critical for sustainable applications requiring repeated deformation without failure. ZDBC-accelerated samples showed the lowest elongation (673.61%), indicative of limited extensibility due to rigid crosslink networks. Accelerator-free systems significantly enhanced flexibility (DHBP: 863.81–878.98%; DCP: 873.98–892.16%), balancing durability and extensibility effectively. These improved flexibility characteristics contribute directly to sustainability by reducing premature material failures and waste generation, supporting extended product usability and environmental benefits.

Summary and Sustainability Implications

Figure 5 summarizes the mechanical properties clearly, demonstrating the advantages of peroxide-based curing systems in achieving high tensile strength and elongation while moderately reducing modulus. DHBP at 2 phr notably emerged as the most effective curing agent, combining environmental safety with improved mechanical properties, significantly outperforming conventional ZDBC. This highlights the potential for widespread adoption of DHBP-based curing to achieve sustainable and safer production of high-quality natural rubber latex films. By reducing the environmental footprint and health hazards associated with traditional accelerators, these findings strongly support the transition toward greener manufacturing practices.

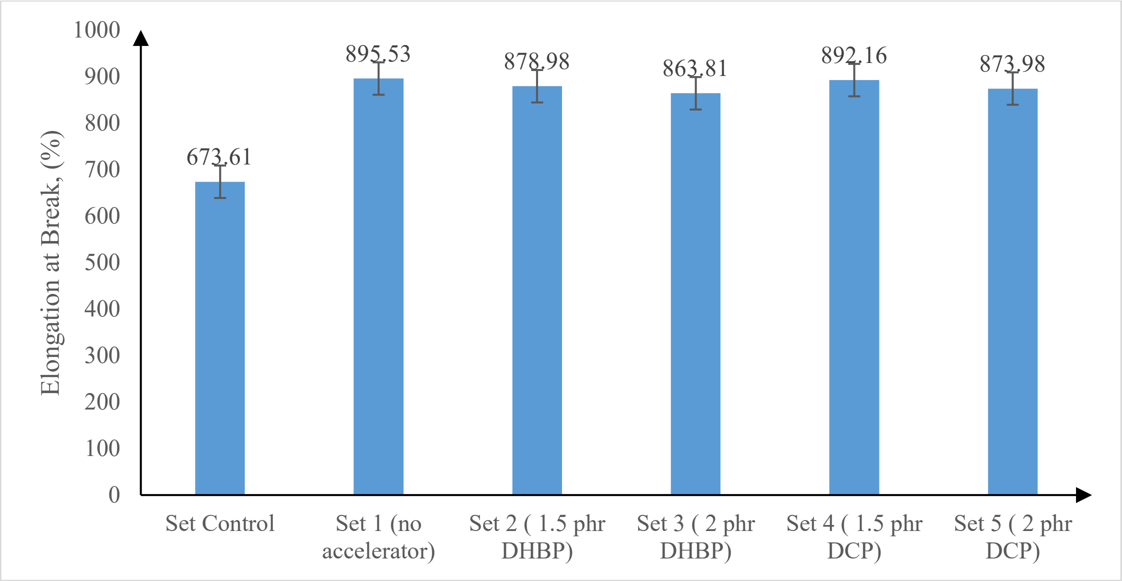

Figure 4. Elongation at break percentages of natural rubber cured with ZDBC, DHBP and DCP.

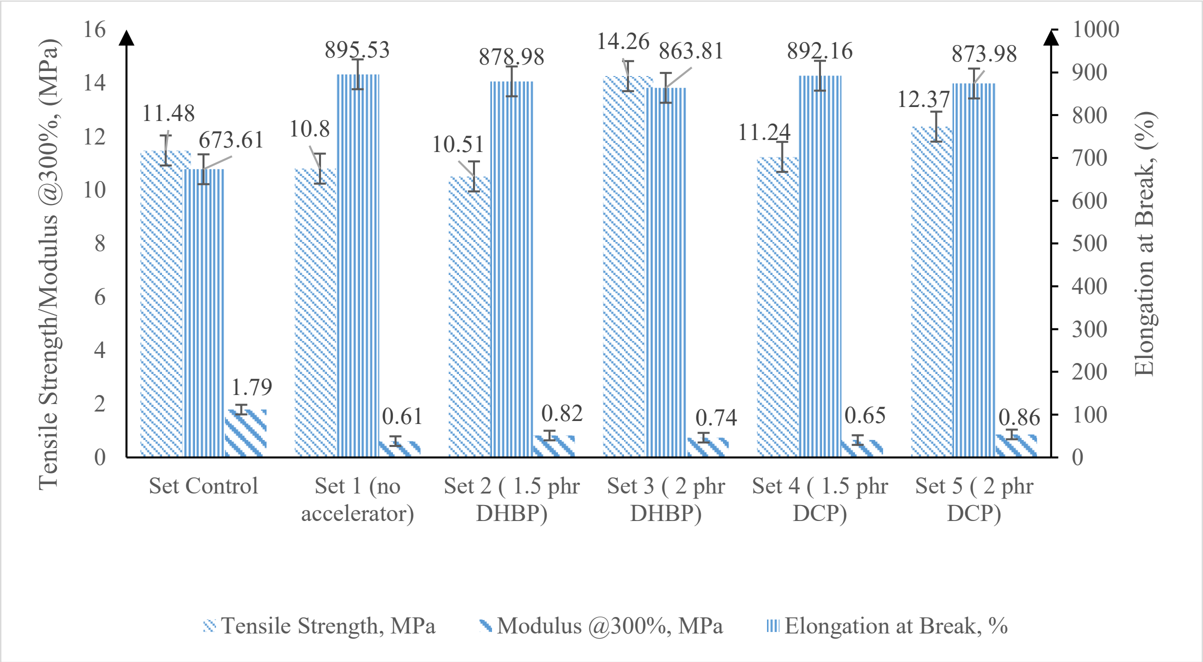

Figure 5. Summarize tensile properties of natural rubber cured with ZDBC, DHBP and DCP.

CONCLUSION

This study demonstrates that organic peroxides, specifically 2,5-dimethyl-2,5-di(tert-butylperoxy) hexane (DHBP) and dicumyl peroxide (DCP), represent effective and sustainable alternatives to traditional accelerators in natural rubber latex (NRL) vulcanization. Among the tested formulations, DHBP at 2 phr notably exhibited the highest tensile strength (14.26 MPa), optimal crosslink density, balanced modulus, and superior flexibility. By significantly reducing hazardous by-products, including nitrosamines, this peroxide-based approach promotes safer working conditions, reduces environmental impacts, and extends product lifetimes. These findings strongly support the shift towards accelerator-free curing methods, aligning closely with sustainable manufacturing principles. Further research is recommended to assess the biodegradability, life-cycle environmental impact, and large-scale industrial feasibility of peroxide-cured NRL films.

ACKNOWLEDGEMENTS

We would like to extend our deepest gratitude to the Ministry of Higher Education for their financial support under the Fundamental Research Grant Scheme (FRGS/1/2023/TK08/UTM/028), Research Contract Grant (PY/2024/01919) and UTM Matching Grant (PY/2024/02437). Furthermore, we sincerely appreciate the generous contribution and collaboration provided by Hartalega Industries Sdn. Bhd., whose support played a pivotal role in the successful completion of this work.

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