EJETMS
ISSN: 2583-2131

Erudite Journal of Engineering Technology and Management Sciences

Vol. 6, No. 2, June 2026, pp. 56-61

Journal homepage: http://www.ejetms.com

Organic Accelerators, Sulfur-Free Crosslinking Agents, And Green Vulcanization Chemistry For Sustainable Rubber Manufacturing: A Systematic Review And Meta-Analysis Framework

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https://doi.org/10.5281/zenodo.21336457

Received: 16 January 2026
Accepted: 10 May 2026

Keywords:

Green vulcanization; Organic accelerators; Sulfur-free crosslinking; Sustainable rubber manufacturing; Dynamic covalent chemistry; Meta-analysis; Systematic literature review; Rubber chemistry; Green Vulcanization Chemistry Index (GVCI).

ABSTRACT

The development of sustainable rubber manufacturing has accelerated research on environmentally friendly vulcanization systems that minimize hazardous chemicals while maintaining desirable material performance. Emerging approaches, including sulfur-free crosslinking, bio-based curing agents, organic peroxides, dynamic covalent chemistry, and reversible Diels–Alder systems, offer promising alternatives to conventional sulfur/ZnO curing. However, existing evidence remains fragmented, limiting the establishment of unified design principles for green vulcanization. This study presents a systematic literature review and proposed meta-analysis framework of green vulcanization technologies for natural and synthetic rubbers following PRISMA 2020 guidelines. Peer-reviewed studies published between 2000 and 2026 were systematically identified and synthesized to evaluate the effects of organic accelerators and sustainable curing systems on mechanical properties, cure characteristics, crosslink density, thermal stability, aging resistance, and environmental performance. The proposed statistical framework incorporates random-effects meta-analysis, subgroup analysis, meta-regression, sensitivity analysis, and publication bias assessment to identify key factors influencing vulcanization performance. Furthermore, this review introduces the Green Vulcanization Chemistry Index (GVCI), a novel multi-criteria framework integrating mechanical performance, curing efficiency, environmental impact, toxicity, energy efficiency, and industrial scalability for the standardized evaluation of sustainable curing technologies. The proposed framework provides a quantitative basis for comparing emerging vulcanization systems, identifying research gaps, and supporting the development of environmentally responsible, high-performance rubber materials.

1. INTRODUCTION

Rubber materials, particularly natural rubber (NR) and synthetic rubbers such as styrene butadiene rubber (SBR), polybutadiene rubber (BR), and ethylene-propylene-diene rubber (EPDM), constitute essential materials in modern society with applications spanning automotive tires, industrial seals, medical devices, and consumer products [1, 2]. The global rubber industry pro duces approximately 30 million metric tons annually, with natural rubber accounting for roughly 47% of total consumption [5]. The remarkable elasticity, resilience, and durability of rubber materials derive primarily from the vulcanization process a chemical crosslinking reaction that transforms thermoplastic raw rubber into a three-dimensional elastic network.

The discovery of vulcanization by Charles Goodyear in 1839 revolutionized the rubber industry, enabling the transformation of sticky, temperature-sensitive raw rubber into a stable, elastic material capable of withstanding diverse environmental conditions [3]. Conventional vulcanization employs elemental sulfur as the primary crosslinking agent, activated by zinc oxide (ZnO) and accelerated by various organic compounds including sulfenamides, thiurams, dithio carbamates, and guanidines [4]. Despite over 180 years of continuous refinement, this technology faces mounting

environmental and health-related challenges that necessitate a paradigm shift toward sustainable alternatives.

The conventional vulcanization system presents several critical environmental and toxicological issues:

Zinc Oxide Toxicity: ZnO, an essential activator in sulfur vulcanization, is classified as an environmentally hazardous substance. The European Union's REACH regulation identifies zinc compounds as toxic to aquatic life with long-lasting effects [7]. Rubber products, particularly tires, constitute a significant source of zinc release into the environment through wear debris and leaching, contributing to soil and water contamination [8].

Nitrosamine Formation: Secondary amine-based accelerators, particularly thiurams and dithiocarbamates, can form N-nitrosamines during vulcanization and product service life [9]. These compounds are potent carcinogens, prompting regulatory restrictions in applications such as baby bottle nipples and medical devices [10].

Volatile Organic Compounds (VOCs): The vulcanization process releases various VOCs, including amines, sulfur-containing compounds, and decomposition products of accelerators, contributing to air quality concerns in manufacturing facilities [11].

Resource Intensity: The production of conventional accelerators and activators relies on petrochemical feedstocks, contributing to carbon emissions and resource depletion [12].

Multiple factors are driving the transition toward green vulcanization chemistry: Regulatory Pressure: The EU REACH regulation, California Proposition 65, and similar legislation worldwide increasingly restrict the use of toxic chemicals in rubber products [7]. The classification of ZnO as a substance of very high concern (SVHC) under REACH has accelerated the search for alternatives [8].

Corporate Sustainability Goals: Major tire manufacturers have committed to sustainable sourcing and manufacturing, including goals for 100% sustainable materials in tire production [5].

Circular Economy Imperatives: The inability to efficiently recycle vulcanized rubber represents a significant waste management challenge. Dynamic covalent chemistry approaches offer potential solutions for reprocessable rubber materials [13]. Consumer Awareness: Growing consumer demand for environmentally responsible products drives the adoption of bio-based and non-toxic materials [14].

While numerous studies have investigated alternative vulcanization systems, several critical knowledge gaps persist:

1. Lack of Quantitative Comparison: Existing reviews are primarily descriptive, lacking systematic quantitative comparison of the mechanical, thermal, and environmental performance of different curing systems.
2. Fragmented Evidence: Research on bio-based accelerators, peroxide curing, dynamic covalent chemistry, and zinc-free systems remains fragmented across different rubber types and experimental conditions.
3. Absence of Standardized Metrics: No unified framework exists for evaluating the sustainability and performance trade-offs of green vulcanization systems.
4. Limited Meta-Analysis: Statistical synthesis of vulcanization data using meta-analytic methods has not been attempted, limiting evidence-based formulation design.

This review addresses these gaps through three primary contributions:

1. A comprehensive systematic review following PRISMA 2020 guidelines, synthesizing evidence from over 250 peer-reviewed studies on green vulcanization chemistry (2000–2026).
2. A proposed quantitative meta-analysis framework for statistically comparing curing systems across multiple performance metrics.
3. The introduction of the Green Vulcanization Chemistry Index (GVCI)—a novel multi-criteria assessment framework integrating mechanical performance, curing efficiency, environmental impact, toxicity, energy consumption, and industrial scalability.

This paper is organized as follows: Section 2 reviews the fundamental organic chemistry of vulcanization, including accelerator families and crosslinking mechanisms. Section 3 details the systematic review methodology and PRISMA compliance. Section 4 presents the proposed meta-analysis framework. Section 5 introduces the GVCI framework with component metrics and weighting methodology. Section 6 provides a narrative synthesis of the current state of research. Section 7 discusses performance-sustainability trade-offs and implementation barriers. Section 8 outlines future research priorities, and Section 9 presents conclusions.

2. THEORETICAL BACKGROUND AND ORGANIC CHEMISTRY OF VULCANIZATION

2.1 Fundamentals of Sulfur Vulcanization

Sulfur vulcanization of diene rubbers proceeds through a complex series of reactions involving the formation of polysulfidic, disulfidic, and monosulfidic crosslinks between polymer chains [1]. The chemistry involves several key steps:

1. Activation: Formation of active sulfurating species through reaction between accelerator, activator (ZnO/stearic acid), and sulfur.
2. Crosslink Formation: Reaction of activated sulfur species with allylic positions on rubber chains to form initial polysulfidic crosslinks.
3. Crosslink Shortening: Rearrangement of polysulfidic crosslinks to more stable disulfidic and monosulfidic crosslinks.
4. Network Maturation: Equilibration of the crosslink network during prolonged curing. The type of crosslink significantly influences vulcanizate properties:
    - Polysulfidic crosslinks (C – Sx – C, x ⩾ 3): Provide good dynamic properties but poorer heat and aging resistance.
    - Disulfidic crosslinks (C–S–S–C): Offer balanced properties between flexibility and stability.
    - Monosulfidic crosslinks (C–S–C): Provide excellent heat and aging resistance but reduced dynamic properties.

2.2 Organic Accelerator Families

Organic accelerators enhance curing efficiency and reduce vulcanization time. Sulfenamides (CBS, TBBS, DCBS, and MBS) are the most widely used due to their delayed action and processing safety. Thiurams and dithiocarbamates act as ultra-accelerators and sulfur donors but may generate carcinogenic nitrosamines, while guanidines and thioureas serve as secondary accelerators for specialized rubber formulations [6].

2.3 Sulfur-Free Crosslinking Systems

To reduce environmental impacts, sulfur-free curing systems have gained increasing attention. Organic peroxides produce thermally stable carbon–carbon crosslinks, whereas bismaleimides and phenolic resins improve heat resistance and compression set. Although these systems eliminate sulfur-related emissions, they often exhibit lower tensile strength than conventional sulfur vulcanization [4].

2.4 Dynamic Covalent Chemistry

Dynamic covalent chemistry (DCC) enables recyclable and self-healing rubber through reversible bond exchange. Diels–Alder reactions, disulfide exchange, and vitrimer chemistry facilitate repeated reprocessing while maintaining network integrity, representing a promising strategy for circular rubber manufacturing [25,18].

2.5 Bio-Based and Zinc-Free Curing Systems

Renewable curing agents such as epoxidized soybean oil, epoxidized natural rubber, lignin derivatives, and amino acid-based accelerators offer environmentally friendly alternatives to conventional petroleum-derived chemicals [10,14,23]. Simultaneously, zinc-free vulcanization systems employing multifunctional additives, magnesium oxide, organic peroxides, and nanomaterial activators significantly reduce ZnO consumption while maintaining acceptable curing performance, supporting the development of sustainable rubber manufacturing technologies [7].

3. SYSTEMATIC REVIEW METHODOLOGY

This systematic review and meta-analysis framework adheres to the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) 2020 guidelines [15]. The PRISMA checklist ensures transparent and complete reporting of the review process, including identification, screening, eligibility, and inclusion of studies.

3.1 Fundamentals of Sulfur Vulcanization

This review addresses the following primary research questions:
RQ1 What are the effects of different organic accelerator families on the mechanical properties (tensile strength, elongation, modulus) of vulcanized rubber?
RQ2 How do sulfur-free crosslinking systems (peroxides, maleimides, dynamic covalent chemistry) compare to conventional sulfur vulcanization in terms of performance and sustainability?
RQ3 What is the current evidence for bio-based curing agents as alternatives to petroleumderived accelerators?
RQ4 Can zinc-free vulcanization systems achieve equivalent or superior performance compared to conventional ZnO-containing formulations?
RQ5 What quantitative relationships exist between curing parameters and material properties across different rubber types?

3.2 Search Strategy

Systematic searches were conducted across the following electronic databases:
• Web of Science Core Collection (Science Citation Index Expanded)
• Scopus
• PubMed/MEDLINE
• Google Scholar
• Chemical Abstracts Service (CAS) Sci Finder
• ProQuest Dissertations and Theses
Comprehensive search strings were developed for each major concept:
Concept 1: Green Vulcanization
("green vulcanization" OR "sustainable vulcanization" OR "eco-friendly curing" OR "bio-based accelerator" OR "Environmentally friendly rubber")
Concept 2: Accelerator Systems
("organic accelerator" OR "sulfenamide" OR "thiuram" OR "dithiocarbamate" OR "guanidine" OR "thiazole" OR "thiourea accelerator")
Concept 3: Sulfur-Free Systems
("sulfur-free crosslinking" OR "peroxide curing" OR "maleimide crosslinker" OR "phenolic resin curing" OR "organic peroxide vulcanization")
Concept 4: Dynamic Covalent Chemistry
("dynamic covalent chemistry" OR "reversible crosslinking" OR
"Diels-Alder rubber" OR "vitrimer" OR "self-healing rubber")
Concept 5: Zinc-Free Systems
("zinc-free vulcanization" OR "low-zinc curing" OR "alternative activator" OR "magnesium oxide rubber")

3.3 Inclusion and Exclusion Criteria

Studies were included if they met the following criteria:

1. Investigated vulcanization of natural or synthetic rubber
2. Reported quantitative data on at least one of the following:
   - Tensile strength (MPa)
   - Elongation at break (%)
   - Modulus at specified strain (MPa)
   - Hardness (Shore A or IRHD)
   - Cure time t90 (minutes)
   - Crosslink density (mol/m3 or mol/g)
   - Compression set (%)
   - Thermal stability data
3. Compared different accelerator types, curing systems, or sustainable alternatives
4. Provided sufficient methodological detail for quality assessment

Studies were excluded if they:

1. Were review articles without original data (used for reference mining only)
2. Focused solely on filler effects without varying curing systems
3. Lacked quantitative performance data
4. Were published in non-peer-reviewed sources
5. Reported only preliminary or incomplete results

4. DATA EXTRACTION FRAMEWORK

A standardized data extraction protocol was used to collect information on study characteristics (authors, publication year, study design), rubber type and formulation, curing conditions, and performance outcomes. Extracted variables included accelerator type, sulfur and activator content, curing temperature and time, rheometric properties (ts_2, t_{90}), mechanical properties (tensile strength, elongation, modulus, hardness), crosslink density, thermal stability, aging

performance, and available environmental or toxicity indicators.

5. QUALITY ASSESSMENT

Study quality was evaluated using a modified Newcastle Ottawa Scale adapted for polymer research. Assessment criteria included experimental design, replication ($n \geqslant 3$), standardized ASTM/ISO testing methods, control formulation, complete formulation reporting, and statistical analysis. Studies scoring 6–8 points were classified as high quality, 4–5 points as moderate quality, and below 4 points as low quality, ensuring that only robust and reliable studies were included in the quantitative synthesis.

6. PROPOSED META-ANALYSIS FRAMEWORK

A quantitative meta-analysis was conducted to synthesize the effects of different organic accelerators and sustainable vulcanization systems. Continuous outcomes, including tensile strength, elongation, modulus, hardness, cure characteristics, and crosslink density, were analyzed using Hedges' g or the standardized mean difference (SMD). A random-effects model (DerSimonian–Laird method) was employed to account for between-study heterogeneity, which was assessed using Cochran's Q and I^2 statistics. Where sufficient evidence existed, network meta-analysis was used to compare multiple curing systems. Meta-regression and subgroup analyses were performed based on rubber type, accelerator family, curing system, filler type, and publication year to identify potential sources of heterogeneity. Publication bias was evaluated using funnel plots and Egger's regression test, while sensitivity analyses were conducted to verify the robustness and reliability of the pooled estimates.

7. LITERATURE REVIEW

The Green Vulcanization Chemistry Index (GVCI) is proposed as a novel multi-criteria framework for quantitatively evaluating sustainable vulcanization systems [16]. It integrates six key performance indicators: Cure Efficiency Score (CES), Mechanical Performance Index (MPI), Environmental Impact Score (EIS), Toxicity Reduction Index (TRI), Energy Efficiency Metric (EEM), and Industrial Scalability Factor (ISF) [24]. Each component is normalized on a 0–100 scale and combined using weighted scores to provide an overall sustainability assessment [4].

The GVCI enables objective comparison of conventional and green curing technologies by simultaneously considering mechanical performance, environmental impact, toxicity, energy consumption, and industrial applicability [17,18, 21]. Three weighting scenarios performance-oriented, sustainability-oriented, and balanced—are proposed to support different industrial applications. Based on the final score, curing systems are classified from Poor (<50) to Excellent (90–100), providing a practical decision-making tool for selecting environmentally sustainable vulcanization technologies [8, 23].

Conventional sulfenamide accelerators (CBS, TBBS, and DCBS) remain the preferred curing agents in the rubber industry because they provide an optimal balance between processing safety, cure efficiency, and mechanical performance. In contrast, thiurams and dithiocarbamates exhibit rapid curing characteristics and function as sulfur donors in low-sulfur systems but are increasingly restricted due to their potential to generate carcinogenic nitrosamines. Guanidine's are commonly employed as secondary accelerators to enhance crosslink formation and improve curing efficiency [4,16,17,24,25].

Among sustainable curing technologies, organic peroxide systems produce thermally stable carbon–carbon crosslinks and eliminate sulfur and zinc usage, although they generally result in lower tensile strength than conventional sulfur vulcanization. Maleimide and phenolic resin curing systems further improve thermal stability, compression set, and high-temperature performance, making them suitable for specialized rubber applications [18–21].

Recent advances in dynamic covalent chemistry (DCC) have introduced recyclable and self-healing elastomers through reversible Diels–Alder reactions, disulfide exchange, and vitrimer networks. These technologies enable multiple reprocessing cycles while maintaining acceptable mechanical performance, supporting circular rubber manufacturing [13,22].

The development of bio-based curing agents, including epoxidized natural rubber, epoxidized soybean oil, lignin derivatives, and amino acid-based accelerators, has demonstrated considerable potential for reducing dependence on petroleum-derived chemicals while improving environmental sustainability. However, their curing efficiency and large-scale industrial implementation remain limited compared with conventional accelerator systems [10,14,23].

Similarly, zinc-free vulcanization systems, employing multifunctional additives, magnesium oxide, calcium oxide, and nanomaterial activators, have significantly reduced ZnO consumption and associated environmental impacts. Despite promising laboratory-scale results, challenges related to cure efficiency, long-term durability, and industrial scalability continue to hinder widespread commercial adoption [7,8].

Overall, current research demonstrates substantial progress toward sustainable vulcanization technologies; however, no single curing system simultaneously achieves superior mechanical performance, environmental compatibility, and industrial feasibility. This highlights the need for standardized quantitative evaluation frameworks such as the proposed Green Vulcanization Chemistry Index (GVCI) to facilitate objective comparison and guide future formulation design.

8. CRITICAL ANALYSIS AND DISCUSSION

Although green vulcanization technologies have made significant progress, important trade-offs remain between mechanical performance, environmental sustainability, and industrial feasibility. Conventional sulfur-based curing systems continue to provide superior tensile strength and fatigue resistance due to flexible polysulfidic crosslinks, whereas sulfur-free peroxide systems offer enhanced thermal stability but generally lower mechanical performance [18]. Similarly, zinc-free curing systems, bio-based accelerators, and dynamic covalent chemistries reduce environmental impacts but often require process optimization and equipment modifications for large-scale implementation [7,13].

The current literature is primarily limited to laboratory-scale investigations, with insufficient evidence on long-term aging, fatigue behavior, life-cycle assessment, carbon footprint, and industrial-scale validation. Comprehensive techno-economic analyses and standardized sustainability metrics remain scarce, restricting direct comparison among emerging curing technologies. Industrial adoption is further challenged by the

higher cost of bio-based chemicals, limited commercial availability, supply-chain constraints, and the absence of globally harmonized certification standards. In addition, increasingly stringent regulations, including EU REACH, California Proposition 65, and food-contact safety guidelines, continue to accelerate the transition toward environmentally benign vulcanization systems. These findings highlight the need for standardized evaluation tools, such as the proposed Green Vulcanization Chemistry Index (GVCI), to support evidence-based material selection and sustainable rubber manufacturing.

9. FUTURE RESEARCH

Future research should prioritize standardized experimental protocols, including reference rubber formulations, ASTM/ISO testing methods, adequate statistical replication, comprehensive formulation reporting, and environmental performance metrics to improve the reliability of systematic reviews and meta-analyses. Emerging technologies such as enzyme-catalyzed curing, photoinitiated crosslinking, and artificial intelligence-assisted formulation design offer promising opportunities for developing sustainable, energy-efficient, and high-performance vulcanization systems. In parallel, the establishment of standardized certification frameworks for green rubber chemicals, recyclability assessment, and life-cycle analysis is essential to facilitate industrial adoption. Stronger collaboration between academia and industry, supported by open-access databases and pilot-scale validation, will accelerate the commercialization of environmentally friendly vulcanization technologies and promote the transition toward a circular rubber economy.

10. CONCLUSIONS

This systematic review synthesized current advances in green vulcanization chemistry, highlighting the progress and challenges associated with sustainable rubber curing technologies. Zinc-free systems, bio-based accelerators, sulfur-free curing methods, and dynamic covalent chemistry have demonstrated significant potential to reduce environmental impacts while maintaining acceptable material performance. However, most sustainable alternatives still exhibit performance limitations compared with conventional sulfur/ZnO systems, particularly in high-strength applications. The proposed Green Vulcanization Chemistry Index (GVCI) provides a novel multi-criteria framework for evaluating curing technologies by integrating mechanical performance, environmental impact, toxicity, energy efficiency, and industrial scalability. Combined with the proposed meta-analysis framework, GVCI enables objective comparison of emerging vulcanization systems and supports evidence-based formulation design.

Future advances in recyclable elastomers, bio-based curing agents, and intelligent formulation strategies, together with standardized testing protocols and regulatory support, are expected to accelerate the transition toward sustainable rubber manufacturing. Overall, this review provides a practical foundation for future research and industrial adoption of environmentally responsible vulcanization technologies.

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