# Polymer Crosslinking Techniques: A Comprehensive Review

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**ABSTRACT**

*Polymer crosslinking is a crucial strategy for enhancing the mechanical, thermal, chemical, and dimensional stability of polymeric materials. It plays a central role in advanced membrane fabrication and energy-related applications, such as fuel cells, batteries, and gas separation systems. This review presents a comprehensive analysis of current crosslinking techniques, including chemical, physical, radiation-induced, and photo/enzymatic methods, with a particular focus on their applications in membrane engineering and energy materials. Recent progress in controlled crosslinking strategies, hybrid approaches, and nanostructured polymers is discussed. Additionally, the review highlights characterization methods for crosslink density and distribution, the challenges associated with scalability and stability, and future directions in designing next-generation crosslinked polymers for high-performance membranes and energy devices. By synthesizing studies from the last five years, this review provides a critical perspective for researchers seeking to optimize polymer crosslinking for advanced functional materials.*

***Keywords:*** *Polymer crosslinking, chemical crosslinking, physical crosslinking, membrane fabrication, energy materials.*

## 1. *INTRODUCTION*

*Polymers are ubiquitous in modern technology due to their versatility, processability, and tunable properties. However, many polymers exhibit intrinsic limitations, such as poor thermal stability, low mechanical strength, and susceptibility to chemical degradation, which restrict their applications in high-performance membranes and energy materials. Crosslinking (the formation of covalent or non-covalent bonds between polymer chains) is a well-established strategy to overcome these limitations [1,2].*

*Crosslinking enhances polymer stability by creating a three-dimensional network that restricts chain mobility, improves dimensional integrity, and provides resistance to solvents, heat, and mechanical stress [3]. The technique is widely used across multiple domains, including membrane fabrication for gas separation and water treatment, polymeric electrolytes in batteries and fuel cells, hydrogels for energy storage, and coatings for corrosion resistance. Among these, membrane-based applications in energy conversion and environmental sustainability have garnered particular attention due to the growing demand for clean energy and efficient separation processes [4].*

*The development of crosslinking methods has progressed from traditional chemical approaches, such as covalent crosslinkers, to advanced strategies including physical crosslinking, radiation-induced crosslinking, and photo/enzymatic techniques. These methods allow for precise control over crosslink density, network architecture, and functional properties of polymers, enabling the design of materials with tailored performance [5,6]. Furthermore, the incorporation of nanomaterials into crosslinked networks has demonstrated significant improvements in permeability, selectivity, ionic conductivity, and mechanical robustness, especially in membrane and energy-related applications [7].*

*Despite these advancements, several challenges remain. Achieving uniform crosslinking in thick or heterogeneous polymer matrices, maintaining long-term stability under operational conditions, and scaling up production without compromising performance are persistent issues. Additionally, precise characterization of crosslink density and distribution remains essential for correlating structure-property relationships in high-performance materials [8,9]. Addressing these challenges is critical for the rational design of next-generation crosslinked polymers suitable for demanding energy and membrane applications.*

*This review provides a detailed discussion of recent advances in polymer crosslinking techniques, emphasizing their role in membrane fabrication and energy materials. It covers chemical, physical, radiation-induced, and photo/enzymatic crosslinking methods, highlights applications in energy and membrane systems, and presents modern characterization approaches. The review concludes with a discussion on current challenges and future perspectives in the field, aiming to guide researchers in optimizing crosslinking strategies for high-performance polymeric materials.*

## *2. Fundamentals of Polymer Crosslinking*

*Polymer crosslinking involves the formation of bonds (either covalent or non-covalent) between polymer chains, producing a three-dimensional network that profoundly influences material properties [9]. Crosslinked networks reduce polymer chain mobility, enhancing mechanical strength, thermal stability, solvent resistance, and dimensional integrity [2].*

*Two major types of crosslinking are generally recognized [10]:*

*1. Covalent crosslinking*

*Permanent bonds between chains that are stable under heat, chemical exposure, and mechanical stress. Examples include chemical reactions with multifunctional crosslinkers.*

*2. Non-covalent (physical) crosslinking*

*Temporary interactions such as hydrogen bonding, ionic interactions, crystallite formation, and entanglements. These interactions are reversible, providing flexibility and self-healing capabilities.*

*Key parameters defining crosslinked polymers include crosslink density, which affects stiffness and swelling behavior, and network homogeneity, which determines the uniformity of mechanical and transport properties. In membrane and energy materials, precise control over these parameters is crucial because crosslink density influences ion transport, gas permeability, and selectivity, as well as electrochemical stability in fuel cells and batteries [11,12].*

*Crosslinking can be initiated by several external triggers, including heat, light, radiation, or chemical reagents. The choice of technique depends on the polymer chemistry, intended application, and required performance characteristics. For membranes used in gas separation or proton exchange membranes, covalent crosslinking ensures dimensional stability under high-pressure or high-temperature operating conditions. In energy materials, controlled crosslinking enhances ionic conductivity while maintaining mechanical integrity [13].*

## *3. Chemical Crosslinking Techniques*

*Chemical crosslinking involves the formation of covalent bonds between polymer chains, typically via multifunctional reagents or reactive groups inherent to the polymer. It is the most widely used method for high-performance polymer membranes due to the permanent and stable nature of the resulting networks.*

### *3.1. Crosslinking with Multifunctional Reagents*

*Multifunctional crosslinkers contain two or more reactive sites capable of forming covalent bonds with polymer functional groups. Common reagents include:*

*Epoxides*

*React with hydroxyl or amine groups to form strong covalent linkages. Widely used in membrane and coating applications for chemical resistance [14].*

*Di- or polyisocyanates*

*React with hydroxyl or amine groups to form urethane or urea bonds, enhancing mechanical strength and thermal stability[15].*

*Glutaraldehyde*

*Commonly used to crosslink polymers with amine functionalities, such as polyvinyl alcohol (PVA) or chitosan, improving water and chemical resistance [16].*

*The reaction conditions, such as temperature, pH, solvent, and stoichiometry, significantly affect crosslink density and network uniformity. Over-crosslinking can reduce permeability in membranes, whereas under-crosslinking may compromise mechanical stability [17].*

### *3.2. Self-Crosslinking Polymers*

*Some polymers can crosslink without external reagents due to the presence of reactive groups in their backbone or side chains. Examples include:*

*Poly(vinyl alcohol) (PVA)*
*Can undergo thermal or chemical crosslinking via hydroxyl groups [18].*
*Polyimides*
*Thermal imidization can generate covalent networks that enhance chemical and thermal stability [18].*
*Poly(ethylene oxide) (PEO)-based polymers*
*Can be crosslinked using sulfonation or ether linkages for fuel cell membranes [19].*
*Self-crosslinking is advantageous in membrane fabrication because it avoids residual crosslinkers that may leach into the environment or compromise performance.*

*3.3. Click Chemistry-Based Crosslinking*

*Click chemistry has emerged as a precise and versatile strategy for chemical crosslinking. Reactions such as azide–alkyne cycloaddition, thiol–ene addition, and Diels–Alder cycloaddition allow for highly controlled crosslink density under mild conditions. In energy and membrane applications, click-based crosslinking is employed to fabricate membranes with defined pore structures, improved ionic conductivity, and long-term chemical stability [20].*

*3.4. Ionically Assisted Chemical Crosslinking*

*Incorporating ionic groups during chemical crosslinking provides both covalent and electrostatic interactions(Fig.1). This hybrid approach can enhance $CO_2$ or proton transport in membranes while maintaining mechanical integrity. For example, quaternized poly(phenylene oxide) membranes crosslinked with dihalides show enhanced alkaline stability and ionic conductivity for anion exchange membranes [21].*

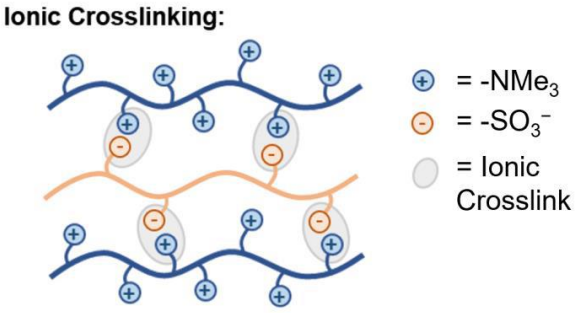


Figure 1- Ionic crosslinking schematic

***4. Physical Crosslinking Methods***

*Physical crosslinking involves non-covalent interactions between polymer chains, such as hydrogen bonding, ionic interactions, crystalline regions, or chain entanglements, creating a reversible network (Fig.2). Unlike chemical crosslinking, physical methods avoid covalent reagents, enabling environmental friendliness, self-healing behavior, and tunable mechanical properties [22].*

*4.1. Hydrogen Bonding*

*Hydrogen bonding is widely used to stabilize polymers with polar functional groups, such as hydroxyl, amide, or carboxyl moieties. For example, polyvinyl alcohol (PVA) membranes can form hydrogen-bonded networks that enhance mechanical strength and dimensional stability while maintaining high permeability for water or gas separation [22]. Hydrogen bonding is also critical in hydrogel-based energy materials, where reversible bonds allow for self-healing and swelling control.*

*4.2. Ionic Interactions*

*Ionic crosslinking utilizes electrostatic attractions between oppositely charged groups in polyelectrolytes. For example, sulfonated polymers can be crosslinked with multivalent cations ($Ca^{2+}$, $Al^{3+}$) to enhance*

*mechanical robustness and control swelling in water purification or fuel cell membranes [23]. Ionic crosslinking is especially advantageous for membranes in energy devices because it improves dimensional stability while preserving ionic conductivity.*

*4.3. Crystallite Formation*

*Semi-crystalline polymers can form physical crosslinks via crystalline domains, which act as junction points. Polyethylene, polypropylene, and poly(ethylene oxide)-based polymers exploit crystallites to reinforce mechanical strength without chemical modification. Crystalline domains provide thermal stability, which is crucial in high-temperature membrane and battery applications [24].*

*4.4. Entanglements and Polymer Blends*

*High-molecular-weight polymers naturally form entanglements that act as reversible crosslink points, contributing to toughness and elasticity. Blending complementary polymers can enhance interchain interactions, creating semi-interpenetrating networks (semi-IPNs) that combine mechanical strength with functional properties, such as ion or gas transport in membranes [25,26].*

*Physical crosslinking is generally less permanent than covalent crosslinking but offers unique advantages in applications requiring flexibility, self-healing, or environmentally responsive behavior. Hybrid approaches combining chemical and physical crosslinking are increasingly adopted in energy membranes to balance mechanical integrity with transport performance [25].*

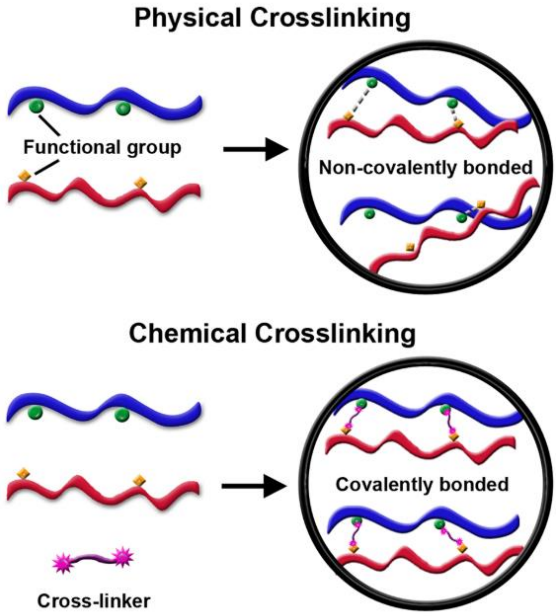


*Figure 2. The difference between physical and chemical cross-linking*

***5. Radiation- and Photo-Induced Crosslinking Techniques***

*Radiation- and photo-induced crosslinking provide advanced, tunable methods for forming polymer networks without additional chemical reagents. These methods involve high-energy photons or ionizing radiation that generate radicals on polymer chains, leading to covalent crosslinking [27]. The figure 3. Shows this schematic.*

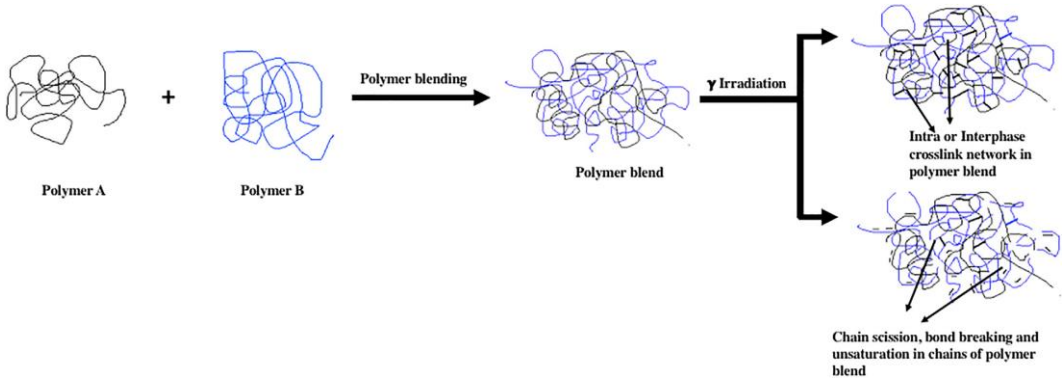


Figure 3. Schematic diagram of the effect of gamma irradiation on chain scission and chain crosslinking in intra and intraphase in polymer blends.

*5.1. Ultraviolet (UV) and Visible Light Crosslinking*

*UV-initiated crosslinking utilizes photoinitiators that generate free radicals when exposed to light, enabling rapid polymerization and network formation at room temperature. Common photoinitiators include benzophenone, camphorquinone, and acetophenone derivatives [28].*

*Visible light crosslinking with LED sources or photoredox catalysts is gaining attention due to energy efficiency and reduced UV damage to polymer chains. This approach is increasingly applied in fabricating membranes and hydrogel electrolytes for fuel cells and batteries.*

*5.2. Gamma and Electron Beam Irradiation*

*Gamma rays and electron beams generate radicals along polymer chains, enabling crosslinking without chemical additives. This method is suitable for bulk materials and membranes where uniform crosslinking is required [29].*

*5.3. Photo-Click and Thiol-Ene Reactions*

*Photo-click reactions, such as thiol-ene or azide-alkyne cycloaddition, combine light initiation with high reaction specificity, enabling precise network control. These methods are especially useful for membrane fabrication, allowing the introduction of functional groups while controlling crosslink density and pore structure [20].*

*5.4. Advantages and Applications in Energy Materials*

*Radiation- and photo-induced crosslinking methods allow for rapid, clean, and tunable network formation, which is highly desirable in polymeric electrolytes, gas separation membranes, and nanostructured membranes. By controlling light intensity, wavelength, and irradiation dose, researchers can fine-tune mechanical and transport properties while avoiding chemical residues that may interfere with membrane performance [29,30].*

***6. Crosslinking in Membrane Fabrication and Energy Materials***

*Crosslinking is central to developing high-performance membranes and energy materials due to its ability to enhance mechanical strength, chemical stability, and selective transport. Recent advances have demonstrated that controlled crosslinking strategies, combined with nanostructured additives, can optimize membranes for gas separation, water purification, fuel cells, and batteries [31].*

*6.1. Gas Separation Membranes*

*Polymeric membranes for $CO_2$, $H_2$, and other gas separations often face a trade-off between permeability and selectivity. Crosslinking addresses this by [31]:*

*1. Reducing chain mobility, which prevents plasticization by gases like $CO_2$.*
*2. Stabilizing free volume and microporous structures to maintain selectivity.*
*3. Enhancing thermal and chemical stability under high-pressure operation.*

*6.2. Fuel Cell Membranes*

*In polymer electrolyte membranes (PEMs) for fuel cells, crosslinking enhances proton conductivity, dimensional stability, and chemical durability. Crosslinked networks prevent excessive swelling in hydrated conditions, maintaining ionic pathways for proton transport [33]. Strategies include:*

*1. Chemical crosslinking of sulfonated poly(ether ether ketone) (SPEEK) with multifunctional agents.*

*2. UV-induced or radiation-assisted crosslinking to form covalent networks without compromising ionic domains.*

*3. Nanocomposite crosslinking with functionalized silica or graphene oxide to improve mechanical stability and proton conductivity.*

*6.3. Battery and Energy Storage Materials*

*Crosslinked polymer electrolytes (CPEs) in lithium-ion and sodium-ion batteries prevent dendrite formation, enhance ionic conductivity, and improve thermal stability. Techniques include [33]:*

*1. Chemical crosslinking of poly(ethylene oxide) with multifunctional acrylates or epoxy-based crosslinkers.*

*2. Radiation-induced crosslinking to form stable ion-conducting networks.*

*3. Hybrid crosslinking with nanofillers for simultaneous enhancement of mechanical and electrochemical properties.*

*6.4. Water Purification and Nanofiltration Membranes*

*Crosslinking in polymeric nanofiltration and reverse osmosis membranes improves Salt rejection and chemical resistance, Chlorine and fouling resistance through covalent and ionic crosslinking and Dimensional stability during high-pressure filtration. For example, PVA-based membranes crosslinked with glutaraldehyde or citric acid exhibit enhanced water flux and salt rejection, while crosslinked polyamide thin-film composite membranes demonstrate long-term operational stability [34].*

## *7. Characterization Techniques*

*Understanding and controlling crosslinking requires precise characterization methods to measure crosslink density, distribution, and network structure. Common techniques include [9,35]:*

*7.1. Spectroscopic Methods*

*1. Fourier Transform Infrared Spectroscopy (FTIR)*

*Detects formation of new covalent bonds, functional group conversion, and crosslinking reactions.*

*2. Nuclear Magnetic Resonance (NMR)*

*Provides information on chemical structure and reaction completion, particularly in solution or solid-state NMR for network polymers.*

*7.2. Thermal Analysis*

*1. Differential Scanning Calorimetry (DSC)*

*Evaluates glass transition and melting behavior, which correlates with crosslink density.*

*2. Thermogravimetric Analysis (TGA)*

*Measures thermal stability and degradation temperatures, indicating network robustness.*

*7.3. Mechanical Testing*

*Tensile, compression, and nanoindentation tests assess the effect of crosslinking on stiffness, elasticity, and toughness. Crosslinked membranes often show higher modulus and improved elongation at break, essential for pressure-driven separation processes.*

*7.4. Swelling and Solvent Uptake Measurements*

*Swelling tests in water or organic solvents indicate crosslink density and network integrity. Lower swelling ratios correspond to higher crosslink density, which is critical for membrane stability in aqueous and electrochemical environments.*

*7.5. Microscopy and Porosity Analysis*

*1. Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM)*
*Visualize morphology, filler dispersion, and network uniformity.*
*2. Porosimetry (BET, mercury intrusion)*
*Quantifies pore size distribution and free volume, especially important in gas separation membranes.*
*These characterization tools allow researchers to correlate crosslinking conditions with performance metrics such as gas selectivity, proton conductivity, or ionic transport, guiding the rational design of advanced polymer networks [34].*

## *8. Challenges and Future Perspectives*

*Despite significant advancements in polymer crosslinking for membrane and energy applications, several challenges remain.*

### *8.1. Control of Crosslink Density and Uniformity*

*Achieving uniform crosslinking throughout thick polymer films or nanocomposite membranes is difficult. Inhomogeneous networks can result in mechanical weak points, reduced selectivity, and variable ionic or gas transport properties. Advanced fabrication techniques, including controlled photopolymerization, in situ crosslinking during membrane casting, and nanofiller surface functionalization, are under investigation to address this issue [36].*

### *8.2. Trade-offs Between Permeability and Mechanical Strength*

*Increasing crosslink density generally enhances mechanical and thermal stability but can reduce gas permeability or ionic conductivity due to decreased free volume. Hybrid strategies combining moderate covalent crosslinking with nanostructured fillers or physical crosslinks are being developed to optimize both properties [37].*

### *8.3. Long-Term Stability under Harsh Conditions*

*Membranes and polymer electrolytes often operate under high temperature, pressure, and chemical exposure. Stability against plasticization, hydrolysis, oxidation, or ionic degradation remains a challenge. Radiation-induced or photo-crosslinking methods have shown promise in enhancing long-term stability, but scalability and cost are limitations [38].*

### *8.4. Scalability and Manufacturing*

*Translating laboratory crosslinking strategies to industrial-scale production requires careful consideration of reaction kinetics, energy input, solvent use, and environmental impact. Methods such as UV-initiated or radiation crosslinking offer rapid processing but require specialized equipment [39]. Green and solvent-free approaches, including thermal or ionic crosslinking, are preferred for sustainable manufacturing [36].*

### *8.5. Future Directions*

*Future advancements in crosslinked polymer systems are expected to focus on creating more versatile, adaptive, and sustainable materials. Hybrid crosslinking approaches that combine chemical, physical, and nanocomposite strategies can yield multifunctional membranes and electrolytes with precisely tailored mechanical, thermal, and transport properties [40]. The integration of smart and responsive polymers, capable of reacting to external stimuli such as pH, temperature, or light, offers opportunities for developing adaptive membranes and next-generation energy devices [41]. Furthermore, computational design tools, including machine learning and molecular modeling, can accelerate material discovery by predicting crosslink network behavior and guiding experimental optimization [42]. Finally, the pursuit of sustainable materials, through the development of biodegradable or recyclable crosslinked polymers, will be essential to minimize environmental impact and promote circular material economy strategies [43].*

## *9. Conclusion*

*Polymer crosslinking remains a fundamental and versatile strategy for enhancing polymer performance in membrane and energy applications. Recent advances in chemical, physical, radiation-induced, and*

*photo/enzymatic crosslinking techniques have enabled the fabrication of membranes with high selectivity, ionic conductivity, and mechanical stability. Nanostructured fillers, hybrid networks, and controlled crosslinking methods have further improved performance metrics, addressing the traditional trade-offs between mechanical strength and transport properties.*

*Challenges such as uniform crosslinking, long-term stability, and industrial scalability remain, but ongoing research in hybrid strategies, responsive polymers, and computational-guided design holds significant promise. Overall, crosslinking remains an indispensable tool in developing next-generation polymeric membranes and energy materials capable of meeting the demands of sustainable energy, environmental protection, and high-efficiency separation technologies.*

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