Food and Bioprocess Technology (2026) 19:103
https://doi.org/10.1007/s11947-025-04200-1

REVIEW

Emerging Applications of Adipic Acid in Enhancing Biopolymeric Food Packaging Performance and Sustainability

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Received: 26 September 2025 / Accepted: 30 December 2025
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Abstract

The increasing demand for sustainable and biodegradable food packaging has led to the development of biopolymeric films using green ingredients and strategies. Adipic acid (ADA) has emerged as a promising green crosslinker, capable of enhancing the performance of these films due to its biocompatibility and effectiveness. This comprehensive review provides an in-depth analysis of ADA, detailing its chemistry, synthesis methods, and functional properties. The review also examines various types of biopolymeric films crosslinked by ADA, including those based on polysaccharides, proteins, and biopolyesters. The crosslinking mechanism of ADA, influenced by factors such as content, temperature, and pH, is explored to highlight its impact on the microstructure, thermal, barrier, and mechanical properties of the films. Besides, practical applications of ADA-crosslinked films in packaging for fruits, vegetables, dairy, and meat products are discussed, alongside their potential in coatings and nanofibers. Finally, safety and regulatory considerations, including global standards and migration studies, are critically assessed to ensure the compliance and consumer safety of ADA. In conclusion, ADA, when derived through green or bio-based synthesis routes, offers potential as a more sustainable crosslinking agent. Its incorporation significantly improves the performance of biopolymeric films, presenting promising opportunities for advanced food packaging, particularly as greener production methods evolve.

Keywords Biopolymeric films · Crosslinking · Plasticizing · Adipic acid · Food packaging

Introduction

The global food industry has a significant challenge in creating sustainable packaging solutions that mitigate environmental issues while preserving the prolonged shelf life of food items (Bertolo et al., 2025). Conventional plastic

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Published online: 09 January 2026

packaging, primarily sourced from petrochemicals, effectively preserves food quality; however, it presents considerable environmental issues due to its non-biodegradability and the proliferation of plastic waste. Thus, there is an urgent need for alternative materials that are biodegradable and may offer similar protective capabilities (Wang et al., 2025; Yang et al., 2025). In response to these challenges, biopolymeric films (BFs) have emerged as promising candidates for sustainable food packaging (FP), offering biodegradability, renewability, and the potential for functionalization to meet specific packaging needs (Han et al., 2025; Omer et al., 2025). This shift is fuelled by both growing consumer demand for effective, environmentally friendly packaging and supportive regulatory initiatives (Hadavifar et al., 2025).

BFs, derived from natural sources such as polysaccharides, proteins, and bio-polyesters, decompose naturally, thereby reducing the environmental burden associated with plastic waste (Fan et al., 2024). Despite their environmental advantages, biopolymeric materials often exhibit limitations, such as brittleness, high water sensitivity, and limited mechanical strength, which hinder their widespread application in FP, where durability and barrier properties are critical. To overcome these issues, various strategies are explored, with chemical crosslinking (CL) emerging as a particularly practical approach (Belay et al., 2017). CL involves the formation of covalent or ionic bonds between polymer chains, enhancing the structural integrity, thermal stability (ThSt), and barrier properties of BFs (Cai et al., 2013).

Among the various CL agents (CLAs) investigated, adipic acid (ADA) has garnered significant attention due to its unique chemical properties and effectiveness in improving biopolymer performance. Table 1 provides a comparison of conventional plastic FP materials, biopolymers, polyesters, and polyesters crosslinked with ADA, illustrating the benefits of ADA in enhancing film performance. ADA, characterized by its two terminal carboxyl groups (-COOH), enables esterification with hydroxyl groups (-OH) and amidation with amine (-NH_2) groups in biopolymers (Falamarzpour et al., 2017; Vibha & Negi, 2020). These reactions result in the formation of strong covalent bonds, which significantly enhance the mechanical, thermal, and barrier properties of BFs. This ability to form robust chemical linkages makes ADA a critical component in the development of advanced packaging materials that meet both functional and environmental criteria. Beyond its role as a CLA, ADA also functions as a plasticizer, reducing intermolecular forces and increasing the mobility of polymer chains (Vibha & Negi, 2020; Q. Xu et al., 2018). This dual functionality is particularly beneficial in applications where flexibility and processability are crucial, such as in the packaging of fruits, vegetables, and dairy products.

Incorporating ADA into BFs enhances their mechanical and barrier properties while also increasing their adaptability to various packaging requirements (Hosseini et al., 2021; Ubeda et al., 2020). The application of ADA in CL of polysaccharides, e.g., starch, chitosan (CS), and cellulose, has been extensively studied (Sole et al., 2022; Ubeda et al., 2020). Starch, one of the most abundant and cost-effective polysaccharides, often suffers from brittleness and poor water resistance. CL of starch with ADA, via the formation of ester bonds (R-COO-R′), creates a more interconnected polymer network, thereby enhancing tensile strength (TS) and reducing water solubility. Similarly, CS, derived from chitin, is valued for its biodegradability and antimicrobial properties, but its mechanical properties are often insufficient for broader applications (Rupérez et al., 2024; W. Zhang et al., 2022). The amidation reaction between the amino groups (-NH_2) of CS and the -COOH of ADA results in amide bonds (R-CONH-R′) and significantly improves the flexibility, TS, and moisture barrier properties of CS films (Ghosh & Ali, 2012). Beside polysaccharides, ADA has been effectively utilized to modify protein-based biopolymers. Proteins such as soy protein, gelatin, and collagen are recognized for their exceptional film-forming properties and biodegradability. However, similar to polysaccharides, these protein-based films often exhibit brittleness and limited mechanical strength (Abedinia et al., 2020). ADA facilitates the formation of covalent bonds between protein chains, leading to enhanced structural integrity (Cai et al., 2013; Zhang, Li, Ding, Liu, & Li, 2013). This chemical modification enhances the films' mechanical properties and boosts their resistance to moisture and gas transmission, making them even more suitable for FP applications.

Parallel to its application, the synthesis methods of ADA have also seen significant advancements. Traditional chemical routes, primarily involving the oxidation of cyclohexane, are being complemented by bio-based approaches that utilize renewable feedstocks (Spiridon et al., 2011). These bio-based methods align with green chemistry principles, contributing to reducing the environmental footprint of ADA production (Rios et al., 2021). Such technological advancements ensure the scalability and sustainability of ADA, making it increasingly viable for industrial applications in FP.

Given the importance of ADA in enhancing the properties of BFs, this review aims to provide a comprehensive analysis of its role in FP applications. The objectives of this review are (i) to explore the chemical interactions between ADA and various biopolymers; (ii) to evaluate the resulting improvements in the microstructure, thermal, mechanical, and barrier functions of the films; and (iii) to discuss the practical applications of ADA-crosslinked films in different FP scenarios. The scope of this review encompasses an in-depth examination of the CL mechanisms of ADA with polysaccharides, proteins, and biopolymers, as well as

Table 1 Comparison of food packaging materials, including ADA crosslinked variants

Category	Material types	Key benefits	Limitations	References
Petroleum-based plastics	PP, PE, PS, PET	Good barrier properties; mechanical strength; hydrophobicity; low cost; mature technology for mass production	Non-renewable; non-biodegradable; environmental pollution; health risks from microplastic residues and migration	(Abbas et al., 2025; Chinglenthoiba et al., 2025; Nayanathara Thathsarani Pilapitiya & Ratnayake, 2024)
Biopolymers	Polysaccharides (CS, starch, cellulose); Proteins (soy protein, gelatin); Lipids (beeswax);	Renewable; biodegradable; safe for food contact; low environmental impact; antioxidant and antimicrobial properties (when modified)	Sensitive to moisture; lower mechanical strength compared to plastics; higher cost; Processing challenges; may need additives for enhancement	(Falamarzpour et al., 2017b; Rhim et al., 2013; Siracusa et al., 2008)
Biopolyesters	PLA, PVA, PHA	Biodegradable; good mechanical properties; compostable; suitable for industrial-scale production	Brittle; low thermal resistance; sensitive to humidity and moisture; requires additional plasticizers or modifiers to improve flexibility	(DeMerlis & Schoneker, 2003; Madhavan Nampoothiri et al., 2010; Sharma et al., 2021)
Biopolymers with ADA crosslinking	Starch, CS, Soy protein, Gelatin with ADA as crosslinker	Improved mechanical properties; enhanced barrier against moisture and gases; increased thermal stability; better antioxidant and antimicrobial effects	Costly raw materials; limited industrial-scale production; requires optimization for mass production; potential migration of crosslinker needs regulation	(Y.-C. Chen et al., 2013; Sellivam et al., 2016; Spiridon et al., 2011)
Biopolyester with ADA crosslinking	PLA, PVA, PBS crosslinked with ADA	Enhanced flexibility; improved barrier properties against moisture and gases; increased thermal stability; better mechanical performance	Higher production costs. more research needed for scalability; potential migration of crosslinker needs regulation; slower biodegradation under certain conditions	(Gautam et al., 2022; Shah et al., 2014; Shaikh et al., 2022; Sun et al., 2018)

an exploration of its dual role as a CLA and a plasticizer. Furthermore, this review will cover recent advancements in the synthesis of ADA, particularly those that align with green chemistry principles, and the implications of these advancements for the scalability and sustainability of ADA production.

Abbreviations: *PP* polypropylene, *PE* polyethylene, *PS* polystyrene, *PET* polyethylene terephthalate, *PLA* polylactic acid, *PVA* polyvinyl alcohol, *PHA* polyhydroxyalkanoates, *PBS* polybutylene succinate.

Chemistry, synthesis, and properties of adipic acid

Understanding the chemistry, synthesis, and properties of ADA is crucial to appreciating its role in enhancing BFs for FP. This section examines the molecular structure and chemical properties of ADA, providing essential context for understanding its role in biopolymer CL. Following this, we will explore the synthesis methods and technological advancements that enable the efficient production of ADA. This encompasses both traditional chemical routes and innovative bio-based approaches, which are becoming increasingly important in the context of sustainable and green chemistry. Understanding these synthesis methods is vital for leveraging ADA's full potential in industrial applications. Additionally, we will examine the functional groups relevant to FP applications, highlighting how ADA interacts with various biopolymers. This examination will provide insights into the mechanisms through which ADA enhances the properties of BFs. Finally, we will discuss the biological activity of ADA, emphasizing its safety and effectiveness in FP applications.

Overview of Molecular Structure and Chemical Properties

ADA ($C_6H_{10}O_4$) is a linear dicarboxylic acid composed of a six-carbon chain (hexanedioic acid) with -COOH groups at each terminal end. Its molecular structure, represented as HOOC-$(CH_2)_4$-COOH, gives it a molecular weight of 146.14 g/mol (Luttrell & Klaassen, 2016). This linear and symmetrical structure plays a key role in enabling its efficient reactivity with biopolymers, making it well-suited for use as a crosslinking agent. The six-carbon backbone of ADA provides a flexible yet robust structure, which is crucial for maintaining the integrity and uniform distribution of stress within cross-linked polymer networks. Additionally, the presence of -COOH groups at both ends of the molecule is central to its functionality (Ding et al., 2016). These highly reactive groups participate in esterification and amidation reactions with -OH and -NH_2 groups in biopolymers, respectively. This dual reactivity allows ADA to form strong covalent bonds with various biopolymer chains, creating a 3D network that enhances the film's structural properties (Manjunatha et al., 2010). Besides covalent bonding, the -COOH groups can engage in hydrogen bonding (H-bonding) with other polar groups within the polymer matrix, further strengthening the crosslinked network and contributing to improved barrier properties and mechanical strength (Sun et al., 2018).

ADA is moderately soluble in water, with a solubility of 1.44 g/100 mL at 25 °C, and highly soluble in organic solvents such as acetone, ethanol, and methanol (Lund & Gorwa-Grauslund, 2024; Rios et al., 2021). This solubility profile is advantageous for incorporating ADA into various polymer matrices during the fabrication of BFs. Moreover, ADA has a high melting point of approximately 152 °C, which ensures that the crosslinked BFs remain stable under typical processing and application temperatures, maintaining their integrity and performance (Jin et al., 2020; Skoog et al., 2018). The acidic nature of ADA is defined by its two dissociation constants, $pKa_1 = 4.41$ and $pKa_2 = 5.41$. These values indicate its ability to donate protons in solution, which enhances its reactivity with basic functional groups in biopolymers. Consequently, the acidic nature facilitates efficient CL through condensation reactions, forming ester and amide linkages (Marenich et al., 2012).

Further, the -COOH groups in ADA readily participate in condensation reactions, forming ester bonds with OH groups and amide bonds with amine groups. This reactivity is crucial for CL biopolymers, significantly improving the mechanical properties (e.g., TS and elasticity), ThSt, and barrier properties (resistance to moisture and gas transmission) of the films (Manjunatha et al., 2010; Spiridon et al., 2011; Sun et al., 2018; Ubeda et al., 2020). Adding ADA to BFs can significantly alter their thermal properties. Techniques such as differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) are used to assess these changes, often revealing enhancements in ThSt and resistance to degradation at elevated temperatures. These improvements are essential for extending the shelf life and functional range of biopolymeric FP materials.

The groups in ADA engage in H-bonding with other polar groups in the polymer matrix, which contribute to the overall cohesion and stability of the crosslinked network (Manjunatha et al., 2010). This is vital for maintaining the structural integrity of BFs under various environmental conditions. H-bonding plays a crucial role in improving the barrier properties of films against moisture and gases, which are essential for effective FP (Ghosh & Ali, 2012; Ubeda et al., 2020). Additionally, ADA is biodegradable, decomposing through microbial activity into non-toxic end products, e.g., water and carbon dioxide (Ghosh & Ali, 2012). Its incorporation into BFs aligns

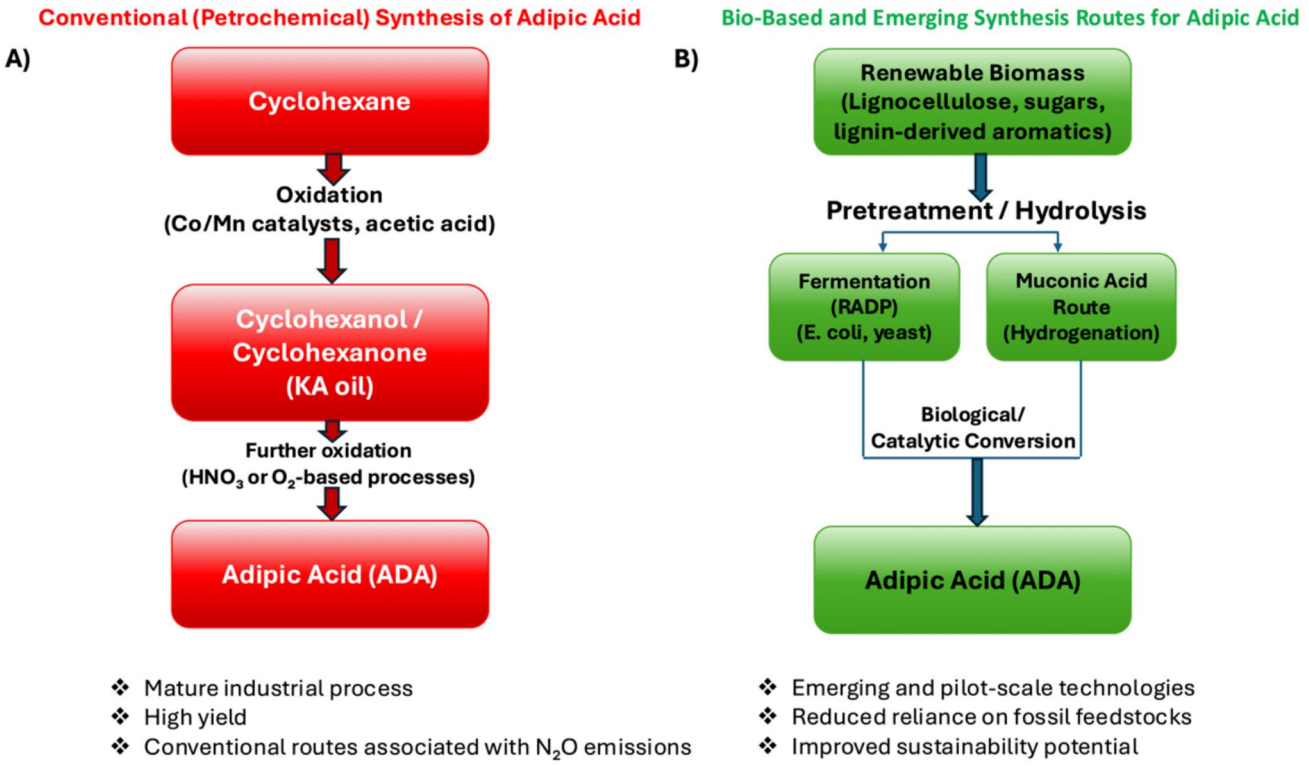


Fig. 1 Schematic overview of conventional petrochemical and representative bio-based synthesis routes for ADA

with sustainability goals by reducing plastic waste and promoting environmentally friendly materials in FP applications. To sum up, the molecular structure and chemical properties of ADA make it an exceptional candidate for enhancing the performance of BFs used in FP. Its ability to form strong covalent bonds and H-bonding, ThSt, and biodegradability support the development of high-performance, sustainable FP solutions.

Synthesis Methods and Technological Advancements

The synthesis of ADA can be achieved through both traditional chemical routes and innovative bio-based approaches. These advancements are crucial for making ADA production more scalable and economically viable, while also supporting a shift toward greener, more sustainable practices, particularly in applications such as FP where environmental impact matters. Figure 1 provides a schematic overview of the principal adipic acid synthesis routes discussed in this section, including the conventional petrochemical oxidation pathway and emerging bio-based approaches derived from renewable feedstocks.

Chemical Routes

The traditional chemical synthesis of ADA primarily involves the oxidation of cyclohexane, a process well-established in industrial settings. This method typically proceeds via the oxidation of cyclohexane to cyclohexanol and cyclohexanone, followed by further oxidation to ADA. The process is catalyzed by a mixture of cobalt and manganese salts in the presence of acetic acid (Mahmud, 2022; Rios et al., 2021). This method, while efficient, is associated with several environmental concerns, including the generation of nitrous oxide (N_2O) as a byproduct, which is a potent greenhouse gas (Ding et al., 2016). Efforts to mitigate these environmental impacts have led to the development of alternative catalytic systems that reduce or eliminate the formation of harmful byproducts.

Recent advancements in chemical synthesis have focused on improving the selectivity and yield of ADA while minimizing environmental impacts. For instance, metal–organic framework (MOF) and ionic liquid as catalysts have shown promise in enhancing the oxidation process efficiency (Mazarı et al., 2022; L. Wang et al., 2023; Wang, Zeng, Wang, & Wang, 2021). These innovative catalysts can potentially lower the reaction temperature and pressure, reduce energy consumption, and decrease the generation of undesirable byproducts, making the process more environmentally

friendly (Lang & Li, 2022). Building on this approach, Feng and co-workers developed a green oxidation process for cyclohexanone to ADA using phosphotungstic acid (PTA) encapsulated in a UiO-66 MOF. This innovative catalyst achieved an impressive ADA yield of 80.3% under mild reaction conditions without any organic solvents or phase transfer agents. The stability and reusability of the PTA@UiO-66 catalyst were also highlighted, with no significant loss in catalytic activity over five successive reaction cycles. This study demonstrates the potential of MOF-based catalysts to enhance the selectivity and efficiency of ADA production while reducing the environmental impact typically associated with traditional oxidation processes (Feng et al., 2019).

Moreover, there have been developments in using renewable feedstocks for the chemical synthesis of ADA. For example, the catalytic conversion of bio-derived furfural to ADA offers a promising route that aligns with the principles of green chemistry (Jin et al., 2020). This approach effectively utilizes renewable resources and integrates more sustainable reaction conditions, significantly lowering the carbon footprint associated with ADA production. Nakagawa and co-workers explored the catalytic conversion of bio-derived furfural and furandicarboxylic acids to ADA using Pt-MoOx/TiO_2 catalysts. It achieved a high yield of ADA, with selectivity reaching up to 99% under optimized conditions. The hydrogenolysis of furandicarboxylic acid resulted in an ADA yield of approximately 85%, demonstrating the efficiency of this method. Additionally, the catalyst exhibited excellent stability, with minimal loss in activity over multiple reaction cycles, making it a promising candidate for sustainable ADA production from renewable feedstocks (Nakagawa et al., 2021).

Bio-Based Approaches

In response to the environmental challenges posed by traditional chemical synthesis, bio-based approaches for the production of ADA have gained significant attention. These methods involve the microbial conversion of renewable biomass into ADA through fermentation processes (Raj et al., 2018). The use of genetically engineered microorganisms, such as *Escherichia coli* and *Saccharomyces cerevisiae*, has been particularly successful in producing ADA from sugars derived from lignocellulosic biomass, like glucose and xylose (Zhao et al., 2018).

A significant study by Zhou and co-workers demonstrated the efficiency of an induction-free system using *E. coli* for ADA production. The authors reconstructed the reversed adipate-degradation pathway (RADP) in *E. coli* and optimized the process to enhance the titer of ADA, achieving a titer of 57.6 g L^{-1} through a fed-batch fermentation process. This approach not only improves the yield but also reduces the reliance on chemical inducers, making the process more sustainable and economically viable. The significance of this study lies in its demonstration of how microbial fermentation, combined with advanced genetic engineering, can serve as a viable alternative to traditional chemical synthesis, offering a greener and more efficient method for ADA production (Zhou et al., 2020). To further illustrate the advancements in bio-based ADA production, Zhao and co-workers successfully engineered *E. coli* to produce ADA through a RADP. Their study demonstrated a remarkable improvement in yield by optimizing metabolic pathways and fermentation conditions, achieving an impressive titer of 68.0 g/L. This represents a significant step forward in the field, showcasing the scalability and sustainability of these innovative synthesis methods, which are increasingly recognized as viable alternatives to traditional chemical processes (Zhao et al., 2018).

In another study, Raj and colleagues (Raj et al., 2018) engineered *S. cerevisiae* to produce ADA from glucose by expressing a bacterial enoate reductase. They achieved a final titer of approximately 2.59 mg/L using a three-stage fermentation process, demonstrating the potential of yeast-based systems for the synthesis of bio-based ADA. By optimizing the fermentation process and metabolic pathways, they attained impressive results, further highlighting the feasibility of microbial systems for producing ADA from renewable feedstocks. Further, the study reported a significant accumulation of *cis*, *cis*-muconic acid, with concentrations exceeding 284 mg/L. This demonstrates the versatility of bio-based production methods and their potential for industrial applications. One of the key advancements in bio-based synthesis is the optimization of metabolic pathways in microorganisms to increase the yield of ADA. Through metabolic engineering, scientists have introduced and optimized pathways that convert simple sugars into intermediates like muconic acid, which can then be hydrogenated to produce ADA (Kruyer & Peralta-Yahya, 2017). Further, advancements in synthetic biology have enabled the design of novel biosynthetic routes that bypass traditional intermediates, leading directly to ADA production with higher efficiency and lower energy requirements.

Another promising bio-based approach involves the use of enzymatic catalysis to convert bio-derived substrates into ADA (Bart & Cavallaro, 2015a). This method leverages the specificity and mild reaction conditions of enzymes, which can operate at ambient temperatures and pressures, thereby reducing energy consumption and the need for harsh chemicals. Enzymatic routes are particularly advantageous for producing ADA from fatty acids and other renewable substrates, offering a more sustainable alternative to petrochemical-based methods (Bart & Cavallaro, 2015a). Zhang and colleagues demonstrated this potential by developing a process that combines enzymatic hydrolysis and microbial

fermentation with chemical catalysis to convert sugar beet residue into ADA. The process achieved an overall yield of 8.4% ADA, with a conversion rate of 0.2 g ADA/g substrate. This approach highlights the efficiency of enzymatic catalysis and exemplifies the successful integration of biological and chemical processes to enhance product yields while reducing the environmental footprint of ADA production (H. Zhang et al., 2016). Despite significant advancements in the bio-based synthesis of ADA, it is important to acknowledge that its predominant industrial production route still relies on the oxidation of petroleum-derived cyclohexane (Bart & Cavallaro, 2015b). While microbial and catalytic processes from renewable feedstocks show great potential, they remain largely confined to research and pilot-scale operations and are not yet commercially dominant. Therefore, although the incorporation of ADA can align with green packaging goals, its overall "green" credentials should be contextualized based on the source and method of production. This nuance is essential for a balanced sustainability assessment of ADA-modified biopolymeric films. Table 2 summarizes the various bio-based synthesis methods for ADA, including sources, synthesis methods, yields, conditions, and benefits.

Abbreviations: *GVL*, γ-valerolactone; *ADA*, adipic acid; *Pt_3Sn/SiO_2*, platinum-tin on silicon dioxide; *Amberlyst 70*, strongly acidic ion-exchange resin; *RuSn/C*, ruthenium-tin on carbon; *IPTG*, isopropyl β-D-1-thiogalactopyranoside; *OD600*, optical density at 600 nm; *RADP*, reverse adipate-degradation pathway; *CatA*, catechol 1,2-dioxygenase; *BcER*, benzoylformate decarboxylase; *GcoAB*, guaiacol monooxygenase complex; *ChCl*, choline chloride; *PEG*, polyethylene glycol; *NaOH*, sodium hydroxide; *SCB*, sugarcane bagasse.

Functional Groups Relevant to Food Packaging Applications

The functional active group is the most important component in packaging materials since it greatly affects the performance and functionality of various packaging systems. The functional properties of any biomaterial depend on the interaction between the polymer chain and stabilizer or CLAs and their parent active molecules (Qiu et al., 2023). Thus, understanding the active groups is helpful during the design of composite FP films for preserving food safety/quality. Various functional groups are associated with the particular properties of the packaging material, which include physic-mechanical, thermal, optical, and barrier properties (Mitra et al., 2012).

The International Energy Agency (IEA) considers ADA to be the most important dicarboxylic acid from an industrial point of view (Deng et al., 2016). From this, IEA includes it in the list of promising platform chemicals for bio-based production as well. Its structural features give unique functional groups that are very important for the modification of (bio)polymer properties, contributing to the sustainability attained in different FP materials (Deng et al., 2016; Skoog et al., 2018). ADA possesses two most important groups at each terminal of its carbon chain, providing active sites for CL with selected (bio)polymers used within the packaging materials. Dicarboxylic acid facilitates the formation of hydroxyl interaction, ester linkages, or amide bonds upon reaction with different active sites (e.g., alcohols or amines), establishing a 3D network within the polymer matrix. This CL structure plays an important role in mechanical properties, chemical resistance, and ThSt to the resulting materials (Deng et al., 2016; Hosseini et al., 2021; Vardon et al., 2015). The addition of ADA as a di-functional carboxylic acid crosslinker is notable in polyesters and polyamides, which are commonly used in packaging materials. This can improve the performance of (bio)polymers during film formation (Falamarzpour et al., 2017).

CS dissolves in dilute solutions of organic acids because of the non-bonding electron pairs found in its -NH_2 groups, which become protonated in acidic conditions. More so, these electrons exhibit strong nucleophilic features, allowing CS to react with functional groups like aldehydes and ketones. The carboxylic groups (–COOH) can function as proton donors to offset the NH_2 groups in CS. Indeed, the ionized version of the –COOH can create an ionic bond with ammonium groups (–NH_3^+) (P.-H. Chen et al., 2007; Cheng et al., 2015). Using ADA to react with CS creates the potential for ionic CL between the -COOH groups of the dicarboxylic acid and the -NH_2 groups in CS chains. These interactions considerably change the characteristics of CS solutions by inducing physical gelation and creating a 3D network (Falamarzpour et al., 2017; Medimagh et al., 2016). Further, Chen and co-workers found that ADA, because of its longer carbon backbone, brings proper flexibility and improves the mechanical strength of CS films (P.-H. Chen et al., 2008).

One of the most common modifications of starch is CL to improve its functionality for various uses. ADA reacts with at least two -OH groups in starch, forming a link between these two -OH groups. This could result in both crosslinked starches and their monoderivatives. As such, crosslinked starches can improve stability against environmental stress, shear, pH-heat, or freeze–thaw cycles compared to native starches (Shulga et al., 2021; Spiridon et al., 2011). In addition, using ADA as a crosslinker allows modification of polymer thermoplasticity into thermoset materials. This property will be particularly useful in designing packaging that has to withstand very high temperatures during its processing or sterilization. With improved temperature resistance, the materials could exhibit structural integrity even under conditions of storage and transportation that are not ideal (Cai et al., 2013; Shirai et al., 2013). Notably, CS-based films made with ADA could experience a chemical

Table 2 Overview of bio-based synthesis methods for ADA

Sources/feedstocks	Synthesis method	Efficiency	Synthesis Conditions	Benefits	Ref
Corn stover (lignocellulosic biomass)	Catalytic conversion of cellulose and hemicellulose-derived GVL into AD	19.1% overall mass yield (41% biomass to GVL, 46% GVL to AD	443 K for GVL production, 378 K, 60 bar for ADA synthesis; various catalysts used: sulfuric acid, Pt_3Sn/SiO_2, Amberlyst 70, RuSn/C, palladium acetate	Renewable feedstock, reduced GHG emissions, integrated energy recovery from biomass residues, economic feasibility (MSP of $633/metric ton)	(J. Han, 2016)
Glucose	Metabolic engineering of *E. coli* utilizing a synthetic pathway	20-fold increase compared to initial strain	Incubation with enzymes at 30 °C for 12 h	Demonstrated the feasibility of a synthetic pathway; increased biosynthesis through enzyme optimization	(Yu et al., 2014)
Glucose, glycerol	RADP in *E. coli* with enzymes from *T. fusca*	68.0 g/L with 93.1% theoretical yield in fed-batch; 96.86% purity post-purification	37 °C for culturing, 30 °C for expression; 1 vvm aeration at 400 rpm; pH 7.0; IPTG induction at OD600 of 0.6	High yield in *E. coli*, elimination of competing pathways using CRISPR/Cas9, reduced greenhouse gas emissions	(Zhao et al., 2018)
Guaiacol (lignin-derived)	One-pot biotransformation using *E. coli* with engineered enzymes (CatA, BcER, and GcoAB)	14% yield (with guaiacol), 98% yield (with catechol)	24 h at 37 °C, pH 7.2, aqueous media	No byproducts, sustainable feedstock valorization, mild conditions, first synthesis from guaiacol	(Suitor et al., 2020)
Glucose, glycerol	RADP in *E. coli* K12 MG1655 using 5′-UTR Complexes	57.6 g/L AD	Fed-batch fermentation, 5 L, 37 °C for growth and 30 °C for production, pH 7, glycerol and glucose as substrates, 144 h	High yield without IPTG, reduced by-products (lactic and butyric acid), cost-effective bio-based production method	(Zhou et al., 2020)
Glucose	RADP in *T. fusca* genes expressed in *Saccharomyces cerevisiae*	10.09 mg/L	Fed-batch fermentation in a 5-L bioreactor; 30 °C, rotation speed 400 rpm, initial glucose 20 g/L, pH 5.5	Environmentally friendly, renewable glucose substrate, demonstrates yeast-based biosynthesis viability	(X. Zhang et al., 2020)
Corn stover	Deep eutectic solvent (ChCl-PEG10000) + NaOH pretreatment followed by enzymatic hydrolysis and fermentation	0.48 g ADA per g reducing sugars	25 °C for 10 min, saccharification at 50 °C for 72 h	Mild conditions, room temperature, enhanced enzymatic saccharification	(D. Xu et al., 2023)
Sugarcane bagasse	Enzymatic hydrolysis followed by fermentation using *E. coli* MG1655 K12	72.2% of theoretical yield	Room temperature (25 °C), 72 h fermentation time, 3 wt% NaOH, 12 wt% ChCl	High sugar yield from SCB hydrolysates, efficient ADA production, mild conditions	(Wu et al., 2023)
Guaiacol (from Kraft lignin)	Two-step bioconversion using engineered *Pseudomonas putida* strains	57% yield (0.57 mol/mol guaiacol)	Aerobic to muconic acid; oxygen-limited conversion to ADA; 30 °C, 48 h	High efficiency with no need for purification, no special equipment, no byproducts, environmentally friendly	(Lund & Gorwa-Grauslund, 2024)

Table 2 (continued)

Sources/feedstocks	Synthesis method	Efficiency	Synthesis Conditions	Benefits	Ref
Lignin-derived phenols	Electrochemical hydrogenation followed by microbial biotransformation	68% yield of cyclohexanol from phenol, 61% yield of AD	Electrochemical hydrogenation at ambient temperature, biotransformation with *Pseudomonas taiwanensis* VLB120 over 5 h	Renewable feedstock, mild conditions, reduced greenhouse gas emissions	(Morejón et al., 2023)

amidation reaction at higher temperatures. This chemical CL can enhance the properties of CS (Cai et al., 2013).

From another point of view, ADA can improve the barrier properties of packaging films against moisture and oxygen, which is important in packaging to prevent food spoilage and prolong its freshness. Further, the efficacy of pre-added additives, e.g., antimicrobial or antioxidant compounds, can be retained over time upon combination within the crosslinked network. In summary, the functional groups of ADA as a crosslinker are pivotal in advancing FP.

Functionality of Adipic Acid in Biopolymeric Films/Coatings

Its multifunctional nature allows ADA to act both as a CLA, enhancing structural integrity and durability, and as a plasticizer, increasing flexibility and processability (Fig. 2). These roles significantly improve the performance and quality of biopolymeric materials, making them more effective for protecting food items. Under plasticization mechanisms, theories such as chain mobility, free volume, and esterification explain how ADA increases the flexibility and processability of biopolymers, reduces brittleness, and improves film ductility. On the other hand, CL mechanisms involve amidation and H-bonding, where ADA enhances moisture resistance, flexibility, and barrier properties, significantly contributing to the overall performance of BFs. The following sections will explore these roles in detail, demonstrating how ADA contributes to the superior performance of BFs and coatings.

Crosslinking Agent

As a CLA, ADA significantly improves the structural integrity and durability of BFs and coatings. By forming covalent bonds between polymer chains, it creates a 3D network that enhances the mechanical properties, such as TS and elasticity. This crosslinked network also improves the ThSt of the films, making them more resistant to degradation under various conditions. Besides, the CL process reduces the permeability of the films to moisture and gases, which is vital for maintaining the freshness and quality of packaged food products. Through these enhancements, ADA helps produce BFs that are more robust, durable, and effective in protecting food items. In the study by Belay et al. (2017), ADA was utilized to crosslink poly(vinyl alcohol) (PVA), a commonly studied synthetic water-soluble thermoplastic. The CL was facilitated through esterification reactions between the -COOH groups of ADA and the -OH groups of PVA, resulting in the formation of ester bonds. This process established a 3D network within the film, significantly enhancing its mechanical properties such as TS and flexibility. Falamarzpour and co-workers employed ADA in

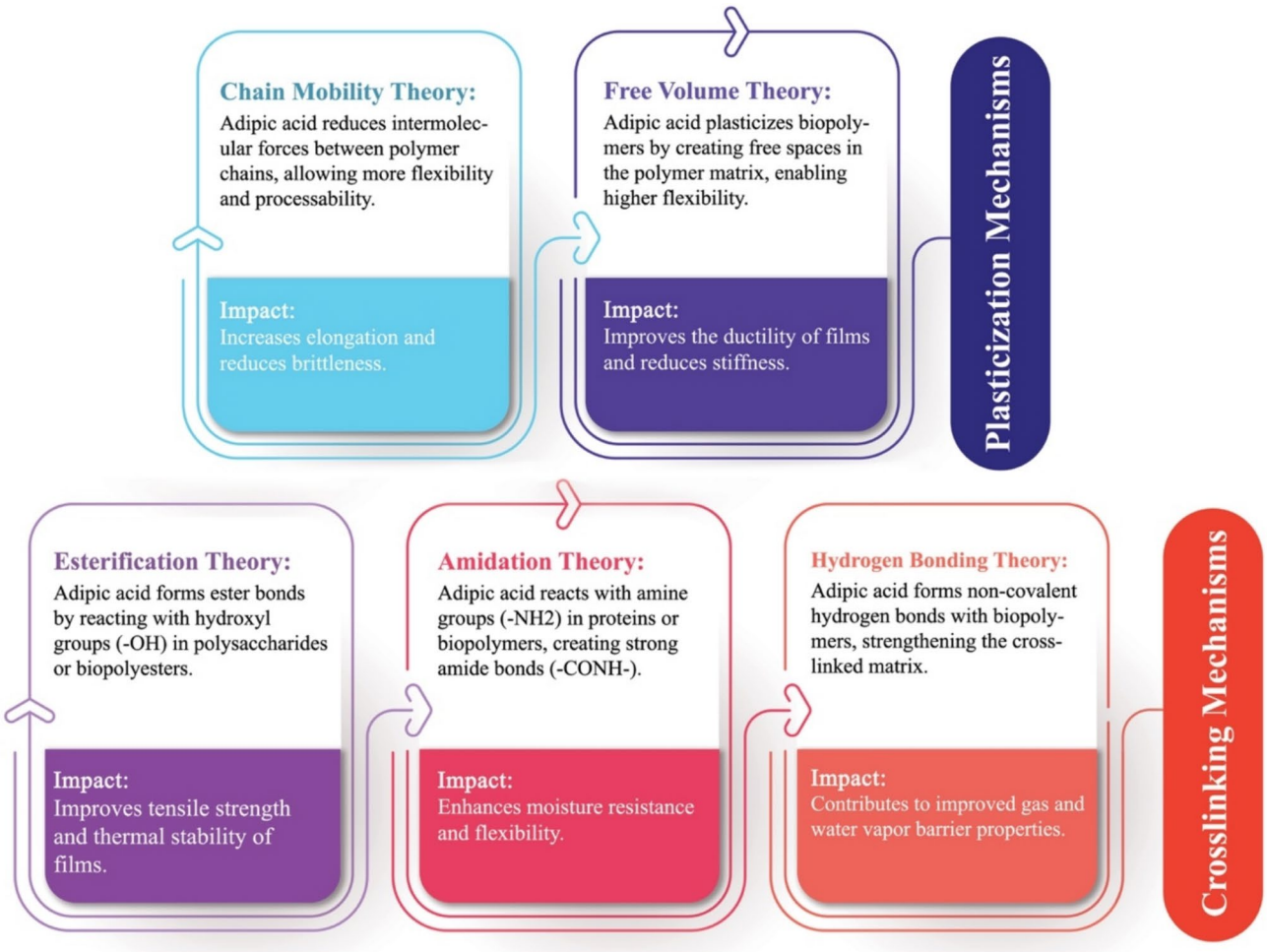


Fig. 2 Adipic acid's role in biopolymeric films and coatings: plasticization mechanisms and crosslinking mechanisms

CS-based films. The CL involved amidation reactions, where the -COOH groups of ADA reacted with the -NH_2 groups of CS, forming stable amide bonds. This chemical interaction was critical for significantly enhancing the structural integrity and mechanical properties of the final films. The successful formation of these amide bonds was confirmed using Fourier transform infrared (FTIR) spectroscopy, which detected characteristic peaks indicative of these new bonds (Falamarzpour et al., 2017).

Cai and co-workers also reported the use of ADA in CS-based membranes, emphasizing the role of amidation reactions in forming stable amide bonds. The formation of these bonds was crucial for enhancing the structural integrity of the membranes. FTIR spectroscopy confirmed these interactions, while changes in the crystalline structure, observed through wide-angle X-ray diffraction (WAXD) analysis, provided additional evidence of successful CL. The study quantified the CL degree (CLD) by measuring the mass of water produced during the reaction, revealing that CLD increased at higher temperatures and longer reaction times, with a maximum CLD = 34.9% at 100 °C for 60 min (Cai et al., 2013). Barbani and co-workers added ADA to gellan gum (GG) films using a dehydrothermal treatment (DHT). The CL process involved esterification reactions between the -COOH groups of ADA and the -OH groups present in GG, forming ester bonds. This chemical modification was verified through FTIR spectroscopy, which detected characteristic peaks corresponding to the ester group (Barbani et al., 2010). Further, WAXD analysis provided insights into changes in the crystalline structure, indicating successful CL. The role of ADA was also highlighted in a study by Nuradibah et al. (2018). Here, ADA was used to crosslink PVA/cellulose nanocrystal (CNC) biocomposites derived from rice straw. The CL was facilitated through amidation reactions, where the -COOH groups of ADA reacted with the -OH groups in PVA, resulting in the formation of ester bonds.

Spiridon and co-workers explored the modification of starch microparticles (SM) in composite films using ADA as a CLA. The process involved esterification reactions, where

the -COOH groups of ADA reacted with the -OH groups present in starch, forming ester bonds. The successful formation of these bonds was verified through FTIR spectroscopy, which revealed characteristic carbonyl peaks, indicating the presence of ester linkages (Spiridon et al., 2011). In research by Sellivam and co-workers, ADA was employed in soy protein isolate (SPI) and kapok husk (KH) biocomposite films. The CL process involved esterification reactions, where the -COOH groups of ADA reacted with the -NH_2 groups of SPI, forming amide bonds. This reaction enhanced the interfacial interaction between the SPI matrix and the KH filler. FTIR spectroscopy confirmed the formation of amide linkages, indicated by the appearance of characteristic carbonyl peaks (C = O) in the spectra (Sellivam et al., 2016). Yeng et al. utilized ADA in CS/corn cob (CS/CC) biocomposite films. The CL process involved amidation reactions, where the -COOH groups of ADA reacted with the -NH_2 groups of CS, forming amide bonds. This reaction improved the interfacial adhesion between the CS matrix and the corn cob filler, leading to enhanced mechanical properties of the biocomposite films. The consistent ability of ADA to form covalent bonds through esterification and amidation reactions showcases its utility in creating robust and durable polymer networks, essential for advanced applications in FP technology (Yeng et al., 2014).

Plasticizer

By integrating into the polymer matrix, ADA reduces intermolecular forces, leading to increased polymer chain mobility. This modification results in films that are less brittle and more pliable, making them easier to handle and apply in various packaging scenarios (Falamarzpour et al., 2017). The plasticizing effect of ADA also contributes to the overall aesthetic and functional quality of the films, ensuring they can be used effectively across a wide range of applications. This dual functionality of ADA as both a CLA and a plasticizer makes it an invaluable component in the development of advanced, high-performance BFs and coatings for FP (Spiridon et al., 2011).

For instance, Falamarzpour et al. utilized ADA as a plasticizer in CS-based films reinforced with cellulose nanofibrils (CNFs). By comparing films with and without ADA, the researchers observed that the di-carboxylic structure of ADA facilitated the plasticization process by reducing intermolecular forces between CS chains. This led to increased flexibility and improved mechanical properties of the films. Specifically, the inclusion of ADA decreased the TS from 78.14 MPa in non-plasticized CS films to 68.32 MPa in plasticized ones, indicating increased chain mobility and flexibility. The elongation at break (EAB) was significantly enhanced, with plasticized films showing an EAB of 18.61%, compared to 3.79% in non-plasticized films. The Young's modulus (YM) also decreased from 4798 to 1736 MPa, further indicating a softer and more pliable material. FTIR spectroscopy confirmed H-bonding between the -COOH groups of ADA and the -NH_2 and -OH groups of CS, disrupting the crystalline structure and reducing the rigidity of the polymer matrix. This study emphasized the potential of ADA to significantly enhance the flexibility and processability of CS-based films, making them more suitable for applications requiring higher flexibility, like FP (Falamarzpour et al., 2017).

Spiridon et al. used ADA as a plasticizer in starch-based composite films, specifically modified SMs integrated with glycerol-plasticized corn starch (GCS). The plasticization occurred through interactions between -COOH groups of ADA and -OH groups in starch, potentially forming ester linkages. This interaction increased chain mobility within the polymer matrix, leading to significant improvements in the flexibility and EAB of the composite films. Although TS of the films decreased slightly due to the plasticizing effect, making them less rigid, the increased flexibility made the films more adaptable for various applications (Spiridon et al., 2011). Vibha and Negi (2020) explored the use of ADA as a plasticizer in a biopolymer blend composed of lignin, styrene, and ADA The plasticizing effect was achieved through esterification reactions, where -COOH groups of ADA reacted with -OH groups of lignin, forming ester bonds. This process, facilitated by lipase-catalyzed transesterification, efficiently integrated ADA into the polymer structure. FTIR spectra confirmed esterification by showing the disappearance of the -OH band at 3437.75 cm^{-1} and the appearance of an ether band between 1300–1000 cm^{-1}, indicative of ester bond formation. ADA improved the material's flexibility and ThSt, evidenced by a higher glass transition temperature (T_g) of 99.7 °C compared to the polystyrene control. Further, the crystallinity of the polymer increased by about 21.88%, as determined by X-ray diffraction (XRD). TGA showed reduced weight loss, indicating improved ThSt, while dynamic mechanical analysis (DMA) revealed a higher storage modulus, suggesting better mechanical strength and stability (Vibha & Negi, 2020).

Martino and colleagues investigated the effect of ADA as a plasticizer in poly(lactic acid) (PLA) films, aiming to reduce the inherent brittleness of PLA for flexible FP applications. ADA disrupted the polymer matrix, increasing chain mobility and flexibility. The plasticization process, facilitated through blending with commercial adipates, significantly decreased T_g of PLA from 58.2 °C in neat PLA to 40.1 °C with 20 wt% ADA. This substantial reduction in T_g demonstrated the effectiveness of ADA in making the polymer more flexible and less brittle. Moreover, the mechanical properties of PLA films improved with ADA. TS rate and YM decreased while the EAB (a measure of flexibility) dramatically increased. Specifically, the percentage deformation at break (ε %) for PLA films

with 20 wt% ADA reached 491%, compared to only 6% for neat PLA. FTIR spectroscopy confirmed the plasticization process by detecting changes in the PLA matrix, including shifts in characteristic peaks associated with ester bonds formed during plasticization. Importantly, the use of ADA did not significantly affect ThSt of PLA films, as the maximum degradation temperature remained relatively unchanged (Martino et al., 2009). In another study, ADA was utilized as a plasticizer to enhance the properties of CS-based films (W. Zhang et al., 2022). Adding ADA into CS matrix was achieved through the formation of H-bonding and potential ionic interactions between -COOH groups of ADA and amino groups of CS. ADA significantly improved the mechanical properties of CS films. TS of the films decreased from 78.14 MPa in the control (non-plasticized) films to 68.32 MPa in the ADA-plasticized films, indicating an increase in the flexibility of the polymer chains. This reduction in TS was compensated by a substantial increase in EAB, which rose from 3.79% in the control films to 18.61% in the plasticized films. This enhancement in elongation demonstrates the effectiveness of ADA in providing the films with increased ductility and flexibility. The thermal properties of CS films were also influenced by ADA. DSC showed a decrease in T_g of the films from 165.33 to 140.22 °C with the addition of ADA, revealing higher chain mobility and flexibility, which are characteristic of plasticized materials.

Adipic Acid Composition in Various Biopolymer Matrices

Incorporation of ADA into various biopolymers leads to improvements in mechanical strength, flexibility, ThSt, and barrier properties, making these materials more effective and durable for FP applications. In the following sections, we will explore the specific effects of ADA on different types of BFs to understand how it enhances their performance and suitability for FP**.**

Polysaccharide-Based Films Crosslinked by Adipic Acid

Polysaccharides, naturally occurring biopolymers, are widely recognized for their biodegradability and film-forming capabilities. However, their inherent limitations, such as brittleness and sensitivity to moisture, necessitate modification for improved performance in FP. CL with ADA has emerged as a promising strategy to overcome these limitations.

Starch

Starch is one of the most abundant and cost-effective polysaccharides available for BF production. Despite its advantages, starch-based films often suffer from brittleness and low water resistance (Gui et al., 2024). CL of starch with ADA enhances the films' mechanical properties by creating a denser and more interconnected network. The result is a more durable and water-resistant film, particularly advantageous in packaging applications where moisture is a concern. This modification retains the biodegradability of starch while significantly enhancing its practical utility (Sun et al., 2018). Figure 3A illustrates the predictive CL reaction that occurs between ADA and starch. In this process, the carboxylic acid groups present in adipic acid interact with the hydroxyl (-OH) groups found along the starch polymer backbone through a method known as esterification. This interaction results in the formation of robust covalent ester bonds, significantly enhancing the mechanical properties of starch-based films. As a result of this CL mechanism, the films show enhanced tensile strength and ThSt, making them more suitable for food packaging (FP) applications. Furthermore, this reaction offers the potential benefit of enhancing the water resistance of the starch matrix by reducing the number of free hydroxyl groups, which primarily contribute to the hydrophilic characteristics of starch. Ultimately, this reduction in hydrophilicity results in improved moisture barrier properties in the modified starch films, thereby optimizing their performance for packaging uses.

Research by Cruz and co-workers demonstrates the remarkable advancements achieved by incorporating ADA into corn starch-based films. The addition of ADA increases ThSt of the films, as TGA shows a higher degradation temperature, approximately 282 °C. This improvement in thermal resistance likely arises from the disruption of starch's crystalline structure. XRD analysis reveals a decrease in crystallinity, which occurs because the formation of ester bonds disrupts the regular packing of starch molecules. This disruption allows for greater flexibility and creates a more resilient polymer network. The likely mechanism behind the improved properties is the esterification reaction between -OH groups of starch and -COOH groups of ADA. This reaction leads to the formation of ester bonds, which create a denser, more interconnected polymer network. FTIR spectroscopy provides further evidence of these chemical interactions, with distinct peaks corresponding to C = O stretching vibrations and O–H stretching, indicating successful ester bond formation. Moreover, scanning electron microscope (SEM) images reveal a more homogeneous film surface, suggesting enhanced miscibility and stronger interfacial interactions between starch and ADA (Cruz et al., 2015).

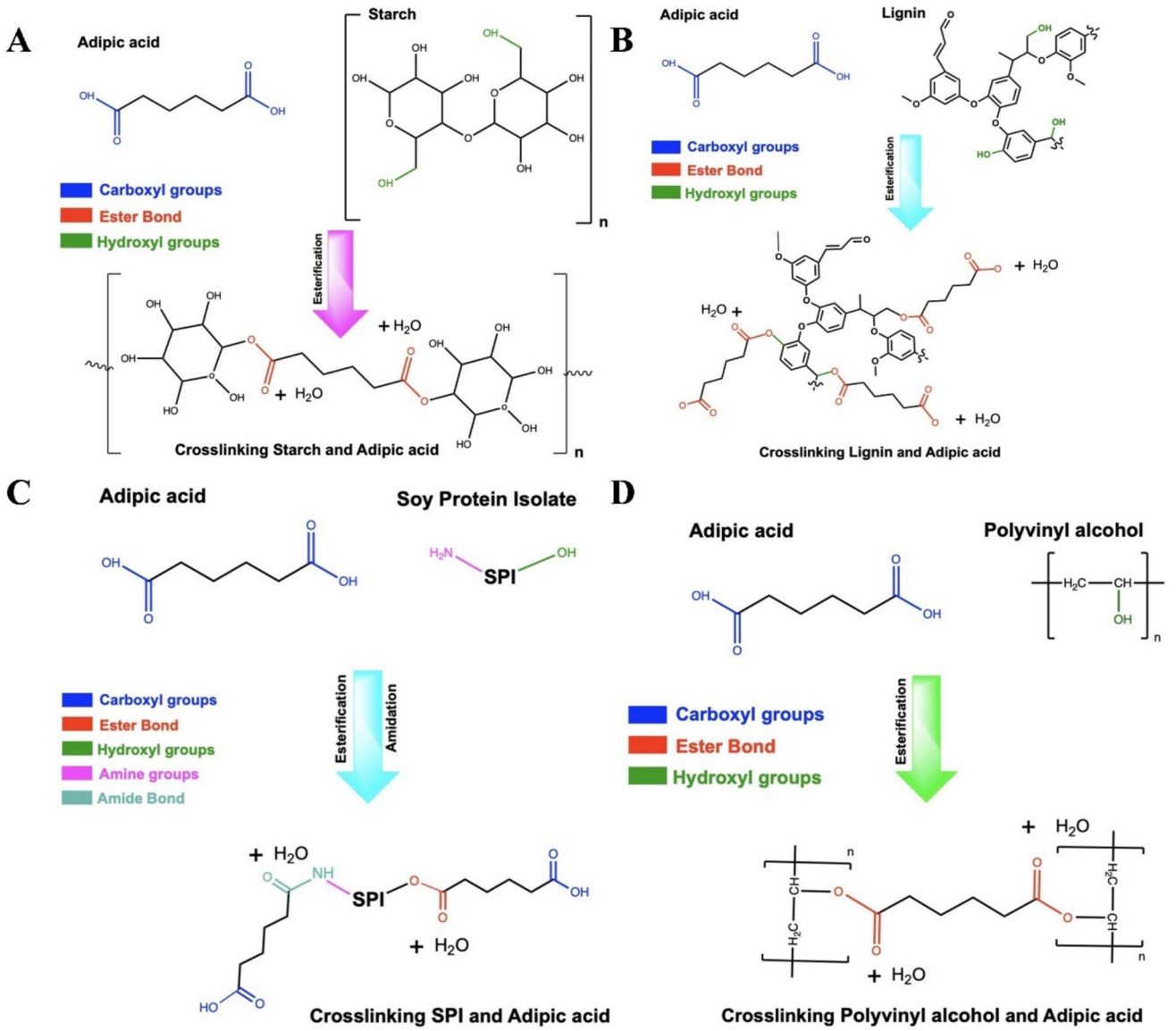


Fig. 3 **A** Predicted crosslinking reaction between ADA and starch, where ester bonds form between the carboxyl groups of ADA and the hydroxyl groups of starch. **B** Proposed esterification between lignin and ADA, forming ester bonds with hydroxyl groups to enhance film strength and thermal stability. **C** Proposed reaction mechanisms between SPI and ADA. **D** Crosslinking mechanism between PVA and ADA through esterification

In a study by Mahmud and co-workers, CL of arrowroot starch with ADA was explored to enhance the mechanical and barrier properties of starch-based films. Adding ADA to starch led to significant improvements in both TS and water resistance. Specifically, the TS of the crosslinked starch films increased to 8.58 MPa, a marked improvement over the native starch films. This enhancement is attributed to the formation of ester bonds between the -OH groups of starch and the -COOH groups of ADA, creating a more robust and interconnected polymer network. The reduction in water absorbency, from > 150% in the native starch to < 60% in the modified films, was also significant. This improvement is likely due to the reduction of free -OH groups in the starch, which are typically responsible for its hydrophilic nature (Mahmud, 2022).

By converting these groups into ester bonds, the starch's hydrophilicity decreased, thereby enhancing its suitability for applications where moisture resistance is crucial, such as FP. Mohammadi and co-workers evaluated the chemical modification of starch with ADA and acetic anhydride, focusing on improving the functional properties of quinoa starch. The modification process involves esterification, where -OH groups in starch react with ADA, forming covalent crosslinks between starch molecules. This CL

significantly enhances the starch's structural stability, leading to improved water retention, swelling power, and reduced syneresis during freeze–thaw cycles. For instance, the starch sample modified with 2% ADA and acetic anhydride demonstrated the highest swelling power (7.91 g/g) and water retention capacity (7.60 g/g), as well as increased solubility (4.45%) compared with other samples and the unmodified control (Mohammadi et al., 2022). These improvements suggest that the crosslinked starch molecules can better interact with water, making the starch more stable and resistant to retrogradation, which is the process where gelatinized starch molecules re-associate, leading to a loss of desirable texture and water release. Further, the modified starch also showed enhanced ThSt, as indicated by DSC: an increase in gelatinization enthalpy and a shift in peak temperature, meaning the starch required more energy to gelatinize, which is a sign of increased structural integrity.

Chitosan

CS, derived from chitin, is a prominent polysaccharide known for its biodegradability and natural antimicrobial properties, making it a valuable material for BFs. However, CS films are typically stiff and lack sufficient mechanical strength for broader applications (Oladzadabbasabadi et al., 2024; Tavassoli et al., 2023). To address these limitations, CL of CS with ADA primarily involves amidation reactions, where -NH_2 groups of CS react with -COOH groups of ADA. This chemical modification leads to the formation of amide bonds (-CONH-), which substantially enhances the flexibility, TS, and EAB of CS films. Further, crosslinked CS films exhibit improved gas and moisture barrier properties, making them more effective in preserving the quality and shelf life of packaged food products.

Medimagh and co-workers demonstrated that incorporating ADA into CS-based films significantly reduced water vapor permeability (WVP), achieving an 85% decrease compared to films prepared with acetic acid. This improvement is attributed to the ionic interactions between -COOH groups of ADA and -NH_2 groups of CS, which create a more tightly crosslinked polymeric matrix, effectively reducing water molecule diffusion. Further, the TS rate of these films increased by approximately 21–27%, while EAB improved by > 60%, indicating enhanced mechanical properties. These improvements are due to the formation of ionic crosslinks that strengthen the structural integrity and flexibility of the films (Medimagh et al., 2016). Building on this foundation, Ghosh and co-workers utilized ADA to crosslink CS through amidation reactions, further demonstrating the benefits of this approach. The resulting amide bonds led to a significant increase in TS, from approximately 13.80 to 16.65 MPa, while EAB improved by > 60%, from 5.17 to 8.43%. These enhanced mechanical properties were attributed to the denser and more interconnected network within the CS matrix created by the ionic CL effect. Besides, ADA significantly reduced WVP, enhancing the barrier properties of the films. This reduction in WVP, coupled with the increased durability and decreased solubility of CS films, underscores their suitability for FP applications where moisture resistance and mechanical integrity are paramount (Ghosh & Ali, 2012). Likewise, Zhang and co-workers explored CL of CS with ADA, focusing on the physicochemical and antioxidant properties of the films. The CL, achieved through amidation reactions, resulted in a more compact and uniform film structure, as evidenced by SEM images and XRD patterns, which showed increased crystallinity and reduced film porosity. The mechanical properties of CS films were significantly enhanced, with both TS and EAB showing marked improvements. The effective CL and the longer carbon backbone of ADA facilitated better interaction with CS chains, contributing to these mechanical enhancements. Moreover, ThSt of the films was improved, with TGA indicating a higher decomposition temperature, reflecting better resistance to thermal degradation, which is crucial for FP applications where the material may be exposed to varying temperatures. Further, the WVP of CS-AD films was reduced, enhancing their effectiveness as moisture barriers, a key factor in maintaining the quality and shelf life of packaged food. Notably, the antioxidant activity of the films was significantly improved, with CS-ADA film showing a DPPH scavenging of 52.19%, likely due to the enhanced availability of functional groups (-NH_2 and -COOH) in the crosslinked structure, which can interact more effectively with free radicals, providing better protection against oxidative degradation (H. Zhang et al., 2016).

Other Polysaccharides

Polysaccharides like cellulose, GG, and lignin significantly benefit from CL with ADA, which enhances their functional properties and broadens their applications in FP. For instance, GG and cellulose films, known for their inherent strength and biodegradability, show marked improvements in TS, ThSt, and moisture resistance through the formation of ester bonds with ADA. This CL creates a denser, more resilient polymer matrix, reducing WVP and enhancing the overall durability of the films. As shown in Fig. 3B, lignin-based films exhibit significant enhancements in mechanical strength and ThSt due to the formation of ester bonds (-O-C = O) with ADA via esterification, which releases water (H_2O) as a byproduct. These improvements in structural integrity and functionality reveal the effectiveness of ADA as a crosslinker, positioning these polysaccharide films as competitive alternatives to synthetic materials in sustainable packaging solutions.

The study on GG and ADA blends, crosslinked by a DHT, provides significant insights into the chemical interactions and resulting properties of these materials. The research demonstrated that the incorporation of ADA into GG, followed by DHT, leads to the formation of ester bonds between -COOH of ADA and -OH groups of GG. This covalent CL process significantly enhances the thermal and mechanical properties of the blends. Key findings include a substantial reduction in WVP, attributed to the tighter network formed by the crosslinked structure. ThSt of the crosslinked materials was notably higher than that of non-crosslinked ones, with TGA showing increased onset and maximum degradation temperatures as ADA content increased. The study also revealed that the crosslinked blends exhibited a lower degree of swelling in water, confirming the enhanced stability provided by the ester bonds (Barbani et al., 2010). In a similar context, Hosseini et al. (2021) utilized ADA as a CLA in the fabrication of cumin seed oil-loaded gliadin-ethyl cellulose (EC) electrospun nanofibers (ENFs), specifically designed for FP. ADA played a crucial role in H-bonding with the mixed biopolymers, significantly increasing the viscosity of the polymer solutions. This increase in viscosity was critical for enhancing the spinnability of the gliadin-EC fibers during the electrospinning process, leading to the production of more uniform fibers with reduced diameters, approximately 250 nm. ADA not only improved the structural integrity of ENFs but also enhanced their ThSt, as evidenced by a higher T_g. Further, FTIR spectroscopy confirmed CL effect, showing characteristic peak shifts that indicated H-bonding between C = O and N–H groups of the polymers and ADA. These CL interactions were pivotal in enhancing the mechanical properties and ThSt of the fibers (Hosseini et al., 2021). Vibha and Negi (2020) demonstrated the versatility of ADA as both a plasticizer and CLA in lignin-styrene-based polymers. ADA significantly improved ThSt, flexibility, and mechanical properties of the polymer through esterification reactions. Specifically, ADA underwent lipase-mediated transesterification with lignin, leading to the formation of ether bonds, as confirmed by FTIR spectroscopy. This chemical interaction significantly increased the T_g of the polymer to 99.7 °C, compared to 92.2 °C for the polystyrene control, indicating improved ThSt. Additionally, the crystallinity of the polymer increased by 21.86%, contributing to enhanced mechanical strength and stiffness, as revealed by XRD. ADA also resulted in a more ordered polymer matrix, reflected in the smooth and homogeneous surface morphology observed through SEM analysis. Furthermore, the polymer exhibited strong antioxidant activity, with a DPPH scavenging of 84.51% at a concentration of 0.1 mg/mL, highlighting its potential for applications requiring durable and biocompatible materials (Vibha & Negi, 2020).

Protein-Based Films Crosslinked by Adipic Acid

Protein-based films are highly valued for their excellent film-forming properties, biodegradability, and nutritional benefits, particularly in the context of edible packaging. However, these films often face challenges related to brittleness, limited mechanical strength, and high water sensitivity, which can hinder their practical application in the FP (Bakhshizadeh et al., 2023). CL with ADA has proven to be an effective strategy for overcoming these limitations, significantly enhancing the mechanical and barrier properties of protein-based films. Proteins like soy protein, wheat-derived protein, collagen, and others have been extensively studied in BFs applications (Sellivam et al., 2016; Xu et al., 2018). Each of these proteins offers unique advantages in terms of film formation and environmental sustainability. Incorporation of ADA into these protein films facilitates the formation of covalent bonds between the protein chains, resulting in enhanced structural integrity. This CL process leads to notable improvements in TS, flexibility, and water resistance, making the films more robust and durable. The chemical modification provided by ADA allows protein-based films to better compete with synthetic plastics, particularly in applications where biodegradability and environmental impact are critical considerations. By improving these key properties, crosslinked protein films are increasingly being recognized as viable alternatives to traditional packaging materials, offering a more sustainable option for the FP industry. Figure 3C illustrates how the incorporation of ADA into SPI leads to esterification and amidation reactions, forming covalent bonds between the carboxyl groups of ADA and the hydroxyl and amine groups of SPI.

Q. Xu et al. (2018) utilized ADA as a CLA to enhance the properties of SPI and kapok husk biocomposite films. ADA significantly influenced the tensile properties of the biocomposite films. As the ADA content increased from 0 to 1.0%, there was a notable improvement in the TS and YM, reaching their peak values at 1.0% ADA Specifically, TS increased due to the formation of amide linkages between ester groups of ADA and -NH_2 groups of SPI, which strengthened the overall film structure. However, when ADA content was further increased to 1.5%, TS and YM decreased while EAB increased. This was attributed to the plasticizing effect of ADA at higher concentrations, which increased chain mobility and reduced interactions between protein chains, thereby enhancing the flexibility of the films. The study also highlighted that the addition of kapok husk contributed to the reinforcing effect, further improving the TS of the biocomposite films. SEM analysis confirmed that CL with ADA enhanced the interfacial adhesion between the

SPI matrix and kapok husk filler, resulting in a more uniform and stable film structure. They claimed that the mechanical properties of SPI films were significantly enhanced through the incorporation of a biological glycerol-based polyester (BGP), where ADA was used as a plasticizer. The study showed that the TS of the SPI-BGP films increased by 12.94–18.22%, depending on the specific ratio of the polyester used, compared to films plasticized with glycerol alone. EAB also improved by 23.63–37.27%, indicating better flexibility. These enhancements are attributed to CL effects of ADA, which disrupt H-bonding between protein molecules, thereby reducing intermolecular forces and increasing the gap between membrane matrices. This process enhances the flexibility of the films and results in a more stable and durable material over time. The study also reported a significant reduction in WVP by up to 14.14%, and the films exhibited lower oxygen permeability (OP), which is crucial for FP applications where moisture and oxygen control are essential. ADA contributed to the formation of a denser network structure within the SPI matrix, leading to these improved barrier properties. Additionally, the contact angle measurements indicated an increase in hydrophobicity, further supporting the film's enhanced barrier properties (Q. Xu et al., 2018).

In a related study, the CL reaction between gliadin, a protein derived from wheat gluten, and various polycarboxylic acids, including ADA, was explored. CL of gliadin with polycarboxylic acids significantly improved the mechanical properties of the protein films. Specifically, TS of the films increased as CLD of ADA increased. The research found that TS of gliadin films crosslinked with ADA was notably enhanced compared to non-crosslinked films. The mechanism behind this improvement is attributed to the formation of covalent bonds between the -COOH groups of ADA and the $-NH_2$ groups of the protein, leading to a more stable and rigid network within the film structure. This chemical reaction primarily involves an S_N2 nucleophilic substitution, where the nitrogen in the $-NH_2$ group attacks the carbon of the -COOH, forming stable (-CONH-) bonds. This CL process effectively increases the molecular weight and the degree of polymerization within the protein matrix, resulting in improved TS and EAB. The study also highlighted the role of molecular structure in the effectiveness of CL. ADA, with its longer carbon chain, provided a flexible yet strong backbone for the protein matrix, allowing for greater tensile elongation compared to shorter-chain acids. This flexibility is critical in applications where both strength and elasticity are required. The researchers observed that the TS of the films increased logarithmically with CLD, indicating a strong quantitative relationship between the extent of CL and the mechanical properties of the films (Shen et al., 2015).

In the study of fish collagen crosslinked with N-hydroxysuccinimide-activated ADA (NHS-AD), significant improvements in the material's properties were observed. As the CLD increased, the collagen's storage modulus (G′) and loss modulus (G″) rose, indicating enhanced structural integrity. A key finding was that collagen with a CLD > 25% exhibited solid-like behavior, accompanied by an increase in denaturation temperature (T_d) from 31.2 to 34.5 °C, reflecting improved ThSt. This enhancement is attributed to the formation of covalent bonds between the collagen's amino groups and the activated -COOH groups in NHS-AD, resulting in a more stable and uniform gel-like network (M. Zhang et al., 2013).

Biopolyester-Based Films Crosslinked by Adipic Acid

Applications of bio-originated polyester films, including PVA and PLA, have been drawing much attention recently due to their sustainability and other potential uses in packaging and elsewhere. These films could be combined with ADA for CL, giving them further advantages. Figure 3D illustrates the CL mechanism between PVA and ADA, where ADA serves as a crosslinker by undergoing an esterification reaction with the hydroxyl groups (-OH) of PVA. This reaction forms ester bonds (-O-C = O), with water (H_2O) released as a byproduct, thereby enhancing the mechanical properties of the resulting films. One of the studies presented the development of biodegradable polymers by reinforcing natural CNCs in a PVA matrix. With different CNC concentrations and the addition of 5 wt% (0.1 g) ADA, an enhancement in the mechanical properties of the films could be achieved. Specifically, ADA-loaded PVA/CNCs films showed higher TS with increased EAB compared to the results for the PVA/CNC films (Nuradibah et al., 2018).

In one such study, blending starch with polyesters created biodegradable films, but the components were often immiscible. Thus, addition of ADA as a compatibilizer at 0.5%, 1.0%, and 1.5% w/w in starch/poly (butylene adipate-co-terephthalate) (PBAT) films was evaluated via reactive extrusion. ADA significantly enhanced the TS from 7.28 to 8.45–9.13 MPa and EAB from 516.51 to 679.10%; YM was within the range between 21.72 and 28.75 MPa, while a lower WVP, from 9.76 to 8.32×10^{-11} $g \cdot s^{-1} \cdot Pa^{-1}$, was observed. The improvement also extended to the microstructure of the films with fewer cracks; however, thermal degradation and crystallinity were unaffected (Silva et al., 2013). A series of aliphatic/aromatic copolyesters were prepared using ADA. Thermal analysis showed that melting, T_m, and crystallization temperatures, T_C, decreased at higher aliphatic content, while the corresponding T_g showed similar trends. Besides, the incorporation of polyethylene glycol and ADA reduced the ThSt and tenacity of the fibers since crystallinity was lowered as a result of disruption in

the regularity of polyethylene terephthalate (PET) chains. Also, the copolymer fabrics exhibited improved moisture regain compared with that of neat PET, and the resistance to hydrolysis and enzymatic degradation was enhanced (Rwei & Lin, 2015).

PLA, in its unmodified form, does not readily crosslink with ADA due to the absence of reactive hydroxyl groups (-OH) on its polymer backbone. PLA primarily consists of ester linkages, which do not easily participate in CL reactions without modification. To enable CL, PLA must first be chemically functionalized, typically by introducing reactive end groups, e.g., hydroxyl (-OH) or amine ($-NH_2$) groups. This functionalization allows PLA to react with ADA's -COOH through esterification or amidation reactions. Alternatively, blending PLA with other polymers or using CLAs like peroxides can facilitate indirect CL by creating reactive intermediates or facilitating radical-based CL. These modifications enable the formation of a crosslinked network between PLA and ADA, enhancing the material's mechanical and thermal properties. In a study done by Kim and co-workers, the mechanical properties and tear resistance of a biodegradable flexible packaging PLA/PBAT-based film were examined using a blown film extrusion process. The inherent brittleness and incompatibility of PLA and PBAT limit their packaging applications. This study assessed various plasticizers, revealing that ADA ester significantly enhanced PLA's flexibility and compatibility with PBAT by promoting chain mobility and improving interfacial adhesion. As a result, the plasticized PLA/PBAT films displayed increased tear resistance, rising from 4.63 to 8.67 $N{\cdot}mm^{-1}$ in the machine direction and from 13.19 to 16.16 $N{\cdot}mm^{-1}$ in the transverse direction (Kim et al., 2020).

Effect of Adipic Acid on the Characteristics of the Packaging

ADA can remarkably improve the thermal, mechanical, and barrier properties of (bio)polymer film/fiber matrices, making it an excellent CLA for FP materials. The bonding process among the polymer chains significantly enhances the mechanical and barrier characteristics of packaging materials, improving their durability and overall performance. ADA further boosts the functional attributes of the matrix by facilitating non-covalent CL interactions. A comprehensive overview of ADA's effects on the physical, mechanical, and microstructural properties of packaging films can be found in Table 3.

Thermal Stability

Analyzing the thermal degradation profile of crosslinked films is a valuable approach for assessing their ThSt, which is crucial for understanding the practical limitations of FP films based on their components and molecular interactions. TGA evaluates thermal behavior by measuring mass loss at elevated temperatures, while DSC analyzes melting temperature, crystallization temperature, and enthalpy changes. Research shows that incorporating ADA as a crosslinker enhances the ThSt of these films by promoting intermolecular interactions, which improve their decomposition characteristics across various temperatures.

In a study by Hosseini and co-workers, the thermal behavior of freeze-dried gliadin, ADA, and EC, along with electrospun fibers, was analyzed using DSC. The gliadin protein exhibited a denaturation temperature indicated by an endothermic peak at 58 °C, consistent with its characterization as a monomeric protein with lower ThSt. In contrast, ADA displayed a sharp peak at about 155 °C, while EC showed an exothermic peak around 140 °C and an endothermic peak near 180 °C, correlating with its T_g and melting temperature. The gliadin-EC mixture with ADA presented a peak at 120 °C, suggesting effective CL and an increase in T_g, although the high surface area of the fibers facilitated water evaporation, leading to a lower T_g (Hosseini et al., 2021). In another study, TGA confirmed the mass change and residuals of different samples after thermal degradation. Here, thermal degradation of polyethylene oxide/dendrobium officinale polysaccharide (PEO/DOP) ENFs was intermediate between pure PEO and DOP, with mass loss increasing from 90.23 to 93.42% as DOP content rose. However, ENFs with ADA demonstrated lower mass loss (84.96 to 87.49%), highlighting ADA's role in improving ThSt by enhancing CL between PEO and DOP (Zhu et al., 2019).

Zhang et al. (2022) explored ThSt of CS films containing different functional acids, including glacial acetic acid, succinic acid, ADA, tartaric malic, and citric acid. Reportedly, ThSt of AD/CS film had a three-step decomposition. The first, around 75–120 °C (5–10 wt%), corresponds to the loss by evaporation of water. An important degradation, corresponding to the release of residual carboxylic acids, was also observed between 180 and 250 °C, while the crystalline structures degraded from 270 to 375 °C. AD/CS film illustrated lower ThSt than the other crosslinked derivatives, probably reflecting changes in H-bonding and chain mobility due to the action of ADA. This indeed manifested in a higher T_g compared with succinic acid/CS film, but a lower value compared to citric acid/CS. Building on these findings, ThSt of packaging films infused with ADA can be influenced by several factors, including the type of polymer, the interactions between the film components, and the amount of added ADA. However, since data correlated with ThSt of packaging systems reinforced with ADA are still limited, further studies are needed to perceive the ThSt of films, indicating a complicated interaction between chemical and thermal properties of ADA as crosslinker (W. Zhang et al., 2022).

Table 3 The effect of ADA on the physical, mechanical, and functional properties of biopolymer-based packaging films

Film base	Adipic acid content	Barrier properties	Mechanical strength	Functional properties	Ref
Chitosan	0.73 g (0.05 M)	↓ (~22 → 13.07 g mm/m^2 d kPa)	EAB; ↑ (~4% → ~8%), TS; ↑ (~85 MPa → ~48 MPa)	DPPH; 52.19%	(W. Zhang et al., 2022)
Chitosan	NS	↓ (1.50 to 0.23 g mm/m^2 d kPa)	EAB; ↑ (5.17% → 8.43%), TS; ↑ (~13.80 MPa → ~16.65 MPa)	*E. coli* (IZ; 00 mm), *S. typhimurium* (IZ; 0 mm), *S. aureus* (IZ; 27.5 mm), *E. faecium* (IZ; 26.83 mm), *S. agalactiae* (IZ; 28.00 mm)	(Medimagh et al., 2016)
Starch	1% wt	NA	EAB; ↑ (~4.89% → 5.84%), TS; ↑ (7.58 MPa → 8.73 MPa), YM; ↓ (653.17 MPa → 533.79MPa)	NA	(Mahmud, 2022)
SPI	NS	↓ (32.46 to 24.98 g·mm/m^2·d·kPa)	EAB; ↓ (~50% → ~15%), TS; ↓ (~5 MPa → ~3.5 MPa)	NA	(Q. Xu et al., 2018)
SPI	0, 1, and 1.5% v/v	NA	EAB; ↑ (~24 → ~26%), TS; ↑ (~4 MPa → ~6 MPa), YM; ↓ (~55 MPa → ~120 MPa)	NA	(Sellivam et al., 2016)
Gliadin	NS	NA	EAB; ↑ (~8% → ~11%), TS; ↑ (~0.25 MPa → ~1.00 MPa)	NA	(Shen et al., 2015)
Chitosan	0.5% and 1.0% w/v	↓ (1.27 to 0.12 10^{-7} g m^{-1} h^{-1} Pa^{-1})	NA	NA	(Sole et al., 2022)
Starch/PBAT	0.5, 1, and 1.5% v/v	↓ (9.76–8.32 9 10^{-11} g per s.m.Pa)	EAB; ↑ (516.51–679.10%), TS; ↑ (7.28 to 8.45–9.13 MPa), YM; ↑ (21.7–28.7 MPa)	NA	(Silva et al., 2013)
Starch/polyester	NS	↓ (5.9×10^{-6} to 5.0×10^{-6} g m^{-1} Pa^{-1} day^{-1}	EAB; ↓ (395% to 36%), TS; ↑ (4.7 to 5.7 MPa), YM; ↑ (28 to 121 MPa)	NA	(Shirai et al., 2013)
PVA/CNC	5 wt% (0.1 g)	NA	TS; ↓)41.3 MPa to 29.3 MPa(, YM; ↑ (~30 MPa – ~50 MPa)	NA	(Nuradibah et al., 2018)
Chitosan/zein	0.5% w/v	↓ (0.071 to 0.022 g mm/cm^2 24 h)	EAB; ↑ (3.185% to 22.31%), TS; ↑ (1.353 MPa to 1.608 MPa), YM; ↑ (0.088 MPa to 0.137 MPa)	DPPH (315.4) and ABTS (455.5) E. coli (~35 mm) and S. aureus (IZ; ~50 mm)	(Zhu et al., 2019)

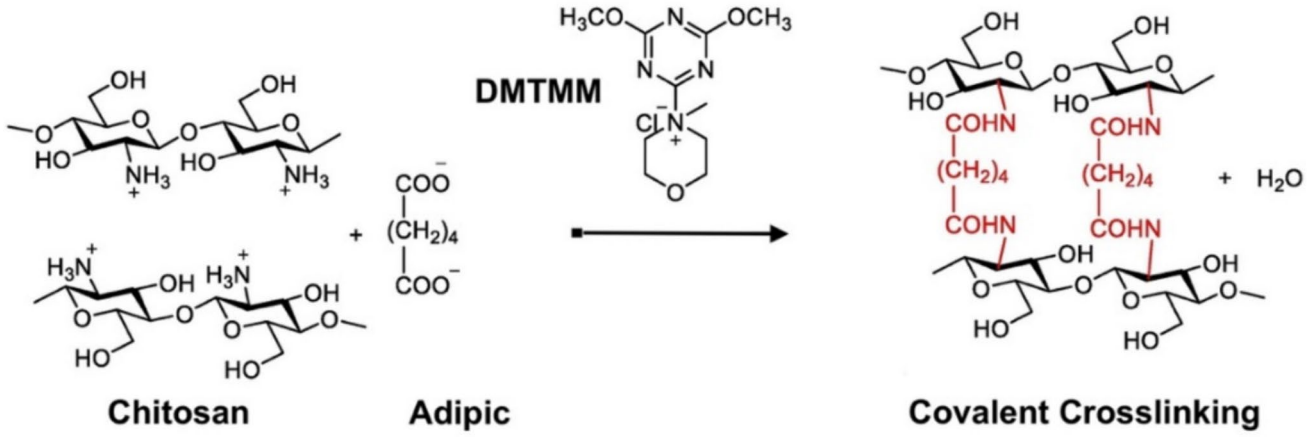


Fig. 4 Covalent crosslinking of chitosan with ADA through DMTMM. Adapted from (Sole et al., 2022) with permission from Elsevier Science Ltd

Barrier Properties

An essential requirement for effective FP systems is the ability to create a barrier that safeguards against harmful environmental factors, e.g., water vapor, oxygen, and UV light. These characteristics are vital as they help preserve the quality of packaged foods by preventing oxidation and dehydration, as well as unwanted rehydration. Enhancing barrier properties is crucial because the exchange of gases or moisture can adversely impact food products and their safety. The effectiveness of reinforced FP is linked to how well functional and technological additives are mixed into the polymer matrix (Khezerlou et al., 2023). Recent studies show that adding ADA to packaging can enhance WVP rates. All research indicates a reduction in WVP, which is attributed to several parameters. In one such study, CS films crosslinked with ADA showed a much lower WVP than pure CS films, which is likely due to the limitations imposed by the CL reaction and to enhanced H-bonding (W. Zhang et al., 2022). In another study, ADA in CS films led to an 85% decrease in WVP (from 1.50 to 0.23 g mm/m^2 d kPa). The improvement in barrier properties was accounted for by the ionic interactions between the two COOH groups of ADA and the NH_2 group of CS, which gave rise to a crosslinked polymeric matrix. As a result, the free volume was reduced, hence lowering the rate of water molecule diffusion through the films (Medimagh et al., 2016).

In an interesting study, a biological glycerol-based polyester (bio-poly glycerol: ADA = 1:2 (BGP I), 1:1 (BGP II), and 2:1 (BGP III); w/w) was utilized as a plasticizer in SPI films. The authors indicated the barrier properties of SPI films significantly improved after adding BGP I, II, and III (D. Xu et al., 2023). Reportedly, SPI films rich in glycerol had the highest WVP at 32.46 g·mm·m^{-2}·d^{-1}·kPa^{-1}, while BGP III-plasticized SPI film exhibited the lowest WVP at 24.98 g·mm·m^{-2}·d^{-1}·kPa^{-1}. Compared to control films, BGP decreased the WVP by 1.25%, 9.37%, and 14.14% for BGP I, BGP II, and BGP III, respectively. The presence of long-chain fatty alcohol esters in BGP limits its water diffusion, contributing to lower WVP. Glycerol, being hydrophilic, disrupts the dense network in SPI films, leading to increased permeability over time. Zhang and co-workers [16] evaluated the physical–mechanical properties of 4-(4,6-dimethoxy-1,3,5-triazin-2-yl)−4-methylmorpholinium chloride (DMTMM)/CS films after adding ADA solution (0.5% and 1.0% w/v). ADA significantly improved the barrier properties of films, decreasing them from 1.27×10^{-7} in the pure film to 0.32×10^{-7} gm^{-1} h^{-1} Pa^{-1} for CS films rich in 0.5% ADA, owing to ionic CL. A further reduction to 0.12×10^{-7} was observed in 1.0% ADA/CS films with high DMTMM content (30% wt). The covalent CL reaction between CS and ADA, facilitated by DMTMM, not only improved barrier properties but also enhanced the hydrophobicity of the films, leading to lower WVP. This CL mechanism, illustrated in Fig. 4, strengthens the films by increasing the intermolecular interactions between CS and ADA, forming stable covalent bonds and reinforcing the film structure. While glycerol increased WVP slightly in plasticized films, covalent CL counteracted this effect, leading to superior barrier properties and mechanical strength. Therefore, ADA effectively reinforces the films by enhancing their physical properties and reducing WVP through improved hydrophobicity (W. Zhang et al., 2022). OP in FP is important as it influences off-flavors, off-odors, and nutritional loss due to oxidation. Lower OP is preferred in films to mitigate these issues (Yekta et al., 2023). Xu and co-workers found that the OP of SPI films decreased by 5.38%, 8.47%, and 1.68% for the BGP I, BGP II, and BGP III formulations, respectively, compared to glycerol-plasticized films. As such, BGP significantly improved gas resistance because of

its ability to interlink with SPI molecules, creating a tangled structure that improved gas barrier features. Therefore, SPI-BGP II films indicated excellent potential for applications in FP systems (D. Xu et al., 2023).

Mechanical Properties

The proper mechanical properties can help in the bearing of different stresses with regard to storage, handling, and transportation. Two of the major indices characterizing the mechanical performance of a film are TS and EAB. ADA could act as a very efficient crosslinker in enhancing these properties, including TS, EAB, stiffness, and toughness, owing to the reinforcement it imparts (Table 3).

In an interesting study, the mechanical properties of CS films were explored after incorporating different crosslinkers, including glacial acetic, succinic, ADA, tartaric, malic, and citric acids (W. Zhang et al., 2022). Here, the mechanical properties of ADA-rich CS films exhibited a mix of brittle and rubber-like traits, differing from those containing tartaric, malic, and citric acids. This behavior is attributed to the presence of -OH groups, which enhance chain mobility and decrease intermolecular forces, resulting in greater EAB. Films containing ADA exhibited superior TS compared to succinic acids/CS, likely due to ADA's longer molecular structure, which minimizes spatial hindrance. These findings highlight the significant impact of intermolecular forces and compositional variations on the rigidity and flexibility of the films. In a comparative study, the effects of different dicarboxylic acid solutions (e.g., ADA, suberic acid, succinic acid, and sebacic acid) on the mechanical properties of CS films were examined (Medimagh et al., 2016). Reportedly, dissolving CS with ADA increased EAB by > 60% (increased from 5.17% to 8.43%). Along the same line, TS of films prepared with ADA (16.65 MPa) was higher than those prepared with acetic acid (13.80 MPa) or succinic acid (14.46 MPa). The enhanced mechanical properties are highly related to the ionic CL effect of dicarboxylic acids. Both TS and EAB increase with the number of carbon atoms in the acid. With longer carbon chains, a stronger ionic CL effect is developed. Thus, a more flexible network and higher EAB are seen with ADA, $n = 6$, compared with succinic acid, $n = 2$.

Mahmud and co-workers measured the mechanical properties of ADA-modified starch films by tensile, flexural, and hardness tests. Accordingly, TS showed a marked increase in the range of 7.83–8.58 MPa, well above that of virgin pure starch films (3–6 MPa). Modified starch also presented rigidity. EAB was lower, within the range of 4.89–5.84%, showing some improvement given by increased crystallinity and bond reformation. Meanwhile, the modulus reached 533.79 and 653.17 MPa, showing higher stiffness than unmodified starch (Mahmud, 2022). Silva and co-workers found that higher concentrations of ADA in starch/PBAT films significantly reduced WVP from 9.76 to 8.32 9 10–11 $g \cdot s^{-1} \cdot m^{-1} \cdot Pa^{-1}$. Films containing 0.5-AD and 1.0-AD indicated similar permeability, but 0.5-AD was more permeable than 1.5-ADA. This improvement is ascribed to ADA enhancing polymer interactions, leading to better adhesion and more compact films, which decreased permeability. More so, the single esterification reaction with ADA reduced the starch's hydrophilic properties, creating more hydrophobic chains. They also reported that ADA improved TS of starch/PBAT films (from 7.28 to 8.45–9.13 MPa), with ADA levels of 0.5% and 1.5% showing similar results. This enhancement was likely because of CL between polymers, resulting in a more compact and homogeneous structure. Higher ADA content did not significantly affect the EAB compared to pure films (Silva et al., 2013).

Q. Xu et al. (2018) utilized biological BGP I, II, and III to improve the mechanical strength of SPI films. Here, TS initially enhanced but then significantly reduced after 20 days, which were affected by CL and the Maillard reaction. After 60 days, TS of SPI films containing BGP I, BGP II, and BGP III declined by 9.28%, 4.00%, and 14.29%, respectively, while glycerol films dropped by 22.22%. Reportedly, optimal ratios of BGP I, BGP II, and BGP III enhanced TS stability, but excessive ADA reduced protein interactions, increasing mobility and decreasing TS. However, over 60 days, EAB of control SPI films declined significantly to 72.83%, while SPI-BGP I, SPI-BGP II, and SPI-BGP III films experienced reductions of 49.2%, 35.56%, and 52.5%, respectively. Thus, BGP greatly improved the EAB (23.63% to 37.27%). The -OH groups in modified glycerol enhance H-bonding within the SPI matrix, creating a more elastic network. As such, moderate ADA in the BGP complex can maintain film strength and flexibility, while excess may undermine structural integrity (Q. Xu et al., 2018). In a similar study, Sellivam and co-workers found that adding ADA (1% (v/v)) increased TS and reduced ductility because of enhanced CL. But, at 1.5% (v/v) ADA, TS decreased owing to increased plasticity and dropped protein chain interactions. More so, YM increased with ADA content up to 1% (v/v), showing greater stiffness. However, elasticity decreased at 1.5% (v/v) ADA because of excessive flexibility. Therefore, moderate ADA (1% (v/v)) improved mechanical strength, whereas higher levels have the potential to generate the reverse result (Sellivam et al., 2016).

In a study, gliadin from wheat gluten was used as a model protein for assessing the tensile properties after CL with different carboxylic acids, including malic acid, ADA, citric acid, and 1,2,3,4-butanetetracarboxylic acid. Gliadin without heat, heated gliadin, or gliadin without carboxylic acid had lower mechanical strength than crosslinked versions (Shen et al., 2015). However, ADA's longer caron chain and lower mobility hindered CL with gliadin, leading to lower

TS and enhanced EAB. In contrast, acids with more -COOH groups (e.g., citric acid) enhanced TS via better CL. Overall, the weak interactions from ADA led to less cohesive gliadin films, as opposed to stronger options like malic acid. Thus, ADA's properties diminished gliadin's mechanical performance.

Functional Properties

The antioxidant properties of the FP system are crucial for preserving food safety/quality. Antioxidants help prevent oxidative degradation, which can lead to spoilage and loss of flavor, texture, and nutritional value (Tavassoli et al., 2024a, b). By reducing the creation of free radicals, FP materials rich in antioxidants prolong food shelf life and maintain its freshness. This is particularly important for oil-rich foods or perishable products that are sensitive to oxidative processes. More so, antioxidant activities improve the overall safety and quality of food items by minimizing the risk of harmful compounds forming during the supply chain (Tavassoli et al., 2024a, b). Ultimately, incorporating ADA into packaging films can significantly improve their antioxidant capacity. Zhang and co-workers indicated that ADA-crosslinked CS films recorded the highest radical scavenging at 52.19%, while films with other acids (citric acid/CS at 11.99% and succinic acid/CS at 47.14%) indicated less variation. The more enhanced antioxidant activity of ADA-crosslinked CS films might be due to the stabilizing effect on free radicals by $-NH_2$ and -OH groups present in CS with a larger molecular structure. The -OH groups present in ADA could enhance H-bonding with CS to develop a structure that is more homogenous and denser, which may reduce DPPH scavenging (W. Zhang et al., 2022).

The antimicrobial properties of FP systems are very important for ensuring food safety/quality and prolonging their shelf life. Indeed, this ability contributes to inhibiting the proliferation and growth of pathogenic microorganisms, minimizing the risk of foodborne illnesses. By controlling microbial populations, antimicrobial FP systems can maintain the quality and freshness of food items, ensuring they remain safe for consumption. The need for such technology is heightened by increasing consumer demand for minimally processed foods with natural ingredients. Further, effective antimicrobial FP systems can reduce food waste by keeping products usable for longer periods. Thus, integrating antimicrobial abilities in FP systems can support both public health and sustainable practices (Tavassoli et al., 2024a, b). In a study by Medimagh and co-workers, CS films with ADA were quite effective against Gram-positive bacteria such as *S. aureus*, *S. agalactiae*, and *E. faecium* and Gram-negative bacteria such as *S. typhimurium* and *E. coli*, whereas CS films containing acetic acid were not have this respect, increases in acidity favored interactions between positively charged $-NH_2$ groups and negatively charged bacterial membranes, causing cell damage. Besides, the structural properties of ADA contribute to such efficiency, allowing better performance compared to other acids (Medimagh et al., 2016). Hosseini and co-workers also reported that adding ADA to gliadin-EC ENFs improves antibacterial activity, with minimal inhibitory concentrations (MIC) for cumin seed oil (CSO) of 0.12 mg/mL for *S. aureus* and 0.15 mg/mL for *E. coli* (Hosseini et al., 2021).

Abbreviation: *NS* not stated, *EAB* elongation at break, *TS* tensile strength, *NA* not analyzed, *IZ* inhibitory zone, *YM* Elastic modulus, *SPI* soy protein isolate, *PBAT* poly (butylene adipate-co-terephthalate), *CNC* cellulose nanocrystal.

Application of Adipic Acid-Crosslinked Packaging for Food Applications

Considering its promising outlook, research on the use of ADA as a crosslinker in biopolymeric FP is scant. To date, only two research works have been conducted exploring its application: one for reducing microbial load in pork products and another for maintaining the preservation of apple slices. Zhu and co-workers prepared novel PEO/DOP ENFs for enhancing pork packaging. ADA enhanced the fibrous structure of nanofibers with high DOP content. During antimicrobial tests, ENFs with the highest weight ratio of DOP, 3:3 (without AD) and 3:3 (with 3% (w:w) AD), reduced *E. coli* populations on pork to 7.11 CFU/g for 3:3 and 6.68 CFU/g for 3:3. More so, the release rate of DOP from ENFs with ADA reached 70.29% after 12 h, outperforming those without ADA by 14%. Sensory evaluations showed that pork treated with PEO/DOP ENFs retained better color and overall quality, indicating their effectiveness in pork meat preservation (Zhu et al., 2019). Adili and colleagues developed an oleogel using reinforced EC combined with ADA and assessed its potential application in cakes and beef burgers. Their research revealed that the oleogel formulation containing EC at 2% and ADA at 4% exhibited the highest oil binding capacity at $97.33 \pm 2.58\%$, making it the optimized choice for cake and beef burger formulations. As a result, food models created with the EC 2%/ADA 4% oleogel demonstrated a pleasing texture profile, as well as acceptable color and sensory characteristics (Adili et al., 2020). PBAT is a thermoplastic polymer that combines both aliphatic and aromatic characteristics. It is synthesized through the copolymerization of adipic acid, 1,4-butanediol, and terephthalic acid. PBAT is known for its excellent tear resistance, significant flexibility, and favorable processability, making it widely utilized in various packaging applications (Sant'Anna Andrade et al., 2025).

In a study by Venkatesan and colleagues, porous carbon nanoparticles (CNPs) were created through the carbonization

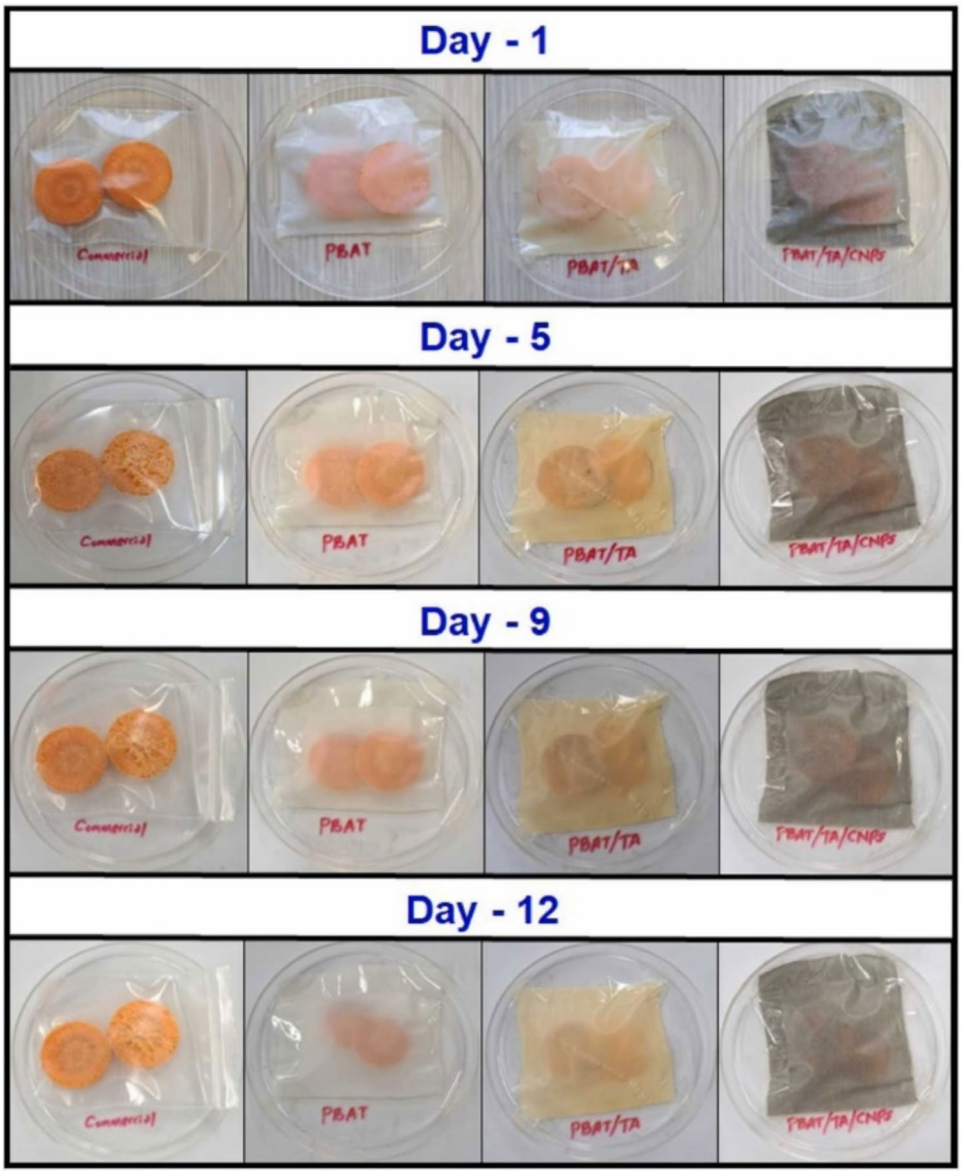


Fig. 5 Application of PBAT-based films and PBAT/TA/CNPs composite films during storage at 25 °C on carrot samples, Ref. (Venkatesan et al., 2023), with reproducing permission from Elsevier under license number 6166870583457

and activation of potassium hydroxide (KOH). The solution casting technique was employed to fabricate composites of PBAT with CNPs and tannic acid (TA). The resulting PBAT/TA/CNPs composites exhibited enhanced antimicrobial properties against food-borne pathogens such as *E. coli* and *S. aureus*. Furthermore, food quality assessments demonstrated that the PBAT/TA/CNPs composites significantly improved the shelf life of sliced carrots, extending it from 1 to 12 days compared to control samples, commercial films, and PBAT films (Fig. 5) (Venkatesan, Sivaprakash, Kim, Eldesoky, & Kim, 2023).

In another study, Xu and co-workers fabricated SPI films plasticized with BGP for fresh-cut apple preservation. This polyester, with variable ratios of bio-poly glycerol to ADA (1:2 → BGP I, 1:1 → BGP II, and 2:1 → BGP III), showed higher efficiency compared with ordinary glycerol. Reportedly, apples packaged in SPI-BGP films were assessed for a total period of 60 days; films containing BGP inhibited mold growth. The mold growth eventually appeared on day 22 in glycerol films, although the BGP I and III films retarded the mold growth to the 34th and 39th days, respectively. Meanwhile, color metrics (ΔE) were significantly higher in control films, indicating quality deterioration, while this value remained lower for SPI-BGP films even after 60 days ($\Delta E = 10.88$, 10.14, and 11.18, respectively, for BGP I, BGP II, and BGP III). Similarly, microbial counts were also below commercial thresholds of quality loss in SPI-BGP films, with an extended shelf life of 28, 34, and 40 days for BGP I, BGP II, and BGP III, respectively (Fig. 6). Results indicated that SPI-BGP films were effective for maintaining and extending the freshness life of fresh-cut apples (Q. Xu et al., 2018).

Although these research projects provide important information, they cannot be regarded as sufficient to reach a general conclusion about the efficacy of ADA for food preservation and its wider applicability. In view of the increasing interest in sustainable FP solutions, more careful studies are urgently needed to test the potentiality of ADA in different kinds of food systems. Its future research should be directed toward more types of foods and storage conditions to establish a better foundation for its application in the FP industry.

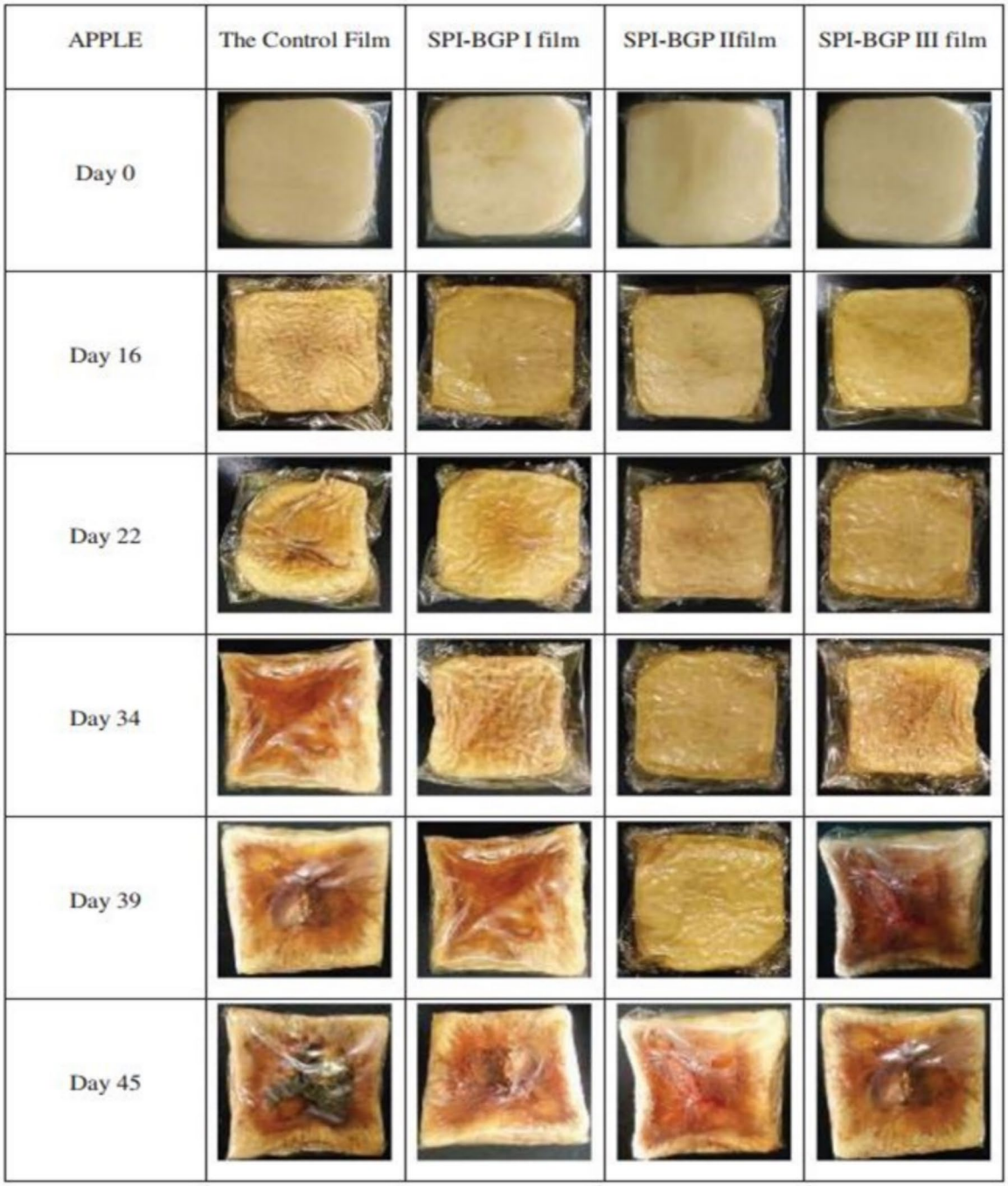
Challenges and Future Prospectives

ADA is a vital compound in the development of BFs, which are increasingly utilized for FP due to their sustainable properties. However, the application of ADA in these materials requires thorough attention to various aspects, including potential migration of the substance, implications for food safety, and adherence to regulatory standards. The regulatory framework governing the use of ADA in food contact materials is shaped by multiple international authorities, most notably the US Food and Drug Administration (FDA) and the European Food Safety Authority (EFSA) (EFSA Panel on Food Contact Materials et al., 2015). The FDA classifies ADA as a Generally Recognized as Safe (GRAS) substance under 21 CFR 184.1009. This designation indicates that ADA can be deemed safe for use in food packaging under specific conditions. Specifically, the FDA allows ADA to be utilized as a plasticizer in polymeric materials intended for contact with food, but this is contingent upon strict compliance with designated concentration limits. These limits are established to ensure that any migration of ADA into food products remains within acceptable safety thresholds. Therefore, manufacturers must conduct rigorous testing and adhere to regulatory guidelines to demonstrate that their biopolymeric films meet safety standards and do not pose risks to consumers. While ADA offers significant advantages in the formulation of biopolymeric films for food packaging, its use must be carefully managed to ensure both compliance with regulatory requirements and the safety of food products (Bart & Cavallaro, 2015b; Vardon et al., 2015). EFSA has also evaluated ADA's safety as a food additive and its application in food contact materials. The specific migration limit (SML) set by EFSA for ADA ensures that its migration into food does not exceed safe levels for human consumption, typically capping the migration at 5 mg/kg of food. Compliance with these limits is mandatory for FP materials within the EU (EFSA Panel on Food Contact Materials et al., 2015). Moreover, global bodies like the Joint FAO/WHO Expert Committee on Food Additives (JECFA) have established acceptable daily intake (ADI) levels, guiding international regulatory standards and ensuring the safe use of ADA in food-related applications (Joint et al., 2011).

The safety of ADA in food packaging is carefully checked through detailed toxicological studies. These studies examine potential health risks, including cancer, genetic changes, and reproductive effects (Kennedy Jr, 2002). Animal studies have consistently shown that ADA has a low toxicity profile, with no significant adverse effects observed at concentrations commonly used in FP. In addition, both in vitro and in vivo studies have been conducted to evaluate the potential migration of ADA from packaging materials into food (Rupérez et al., 2024; Ubeda et al., 2020). The results show that migration levels are usually much lower than the safety limits, which supports the safe use of ADA in food packaging. ADA is also biodegradable and has minimal environmental impact, making it a good choice for sustainable packaging. Risk assessments for ADA in food packaging include a close look at both exposure and hazard. Exposure assessments measure how much ADA might move from packaging into food under normal conditions, often using tests that mimic real-life contact with different foods. At the same time, hazard assessments review the toxicology of ADA and its possible health effects. By combining these findings, experts decide if using ADA in certain food packaging is safe for consumers. If migration levels stay within the limits set by regulators, ADA is considered safe to use (EFSA Panel on Food Contact Materials et al., 2015).

Migration studies are an essential component of ensuring ADA's safety in food contact materials. These studies assess how much ADA migrates from packaging materials to food under varying conditions, such as different temperatures, storage durations, and food types. Recent research indicates that ADA migration is influenced by factors, including the polymer matrix, the presence of other additives, and the nature of the food product itself. For example, foods with higher fat content may increase the migration rate of ADA from packaging. Nonetheless, when used within recommended guidelines, the migration levels of ADA consistently remain within safe limits (Ubeda et al., 2020; Vrolijk et al., 2020). In conjunction with migration studies, exposure assessments estimate consumers' potential intake of ADA from materials containing the compound. These assessments consider factors, e.g., the frequency of consumption, the amount consumed, and the concentration of ADA in the compound. The estimated daily intake (EDI) is then compared to the ADI to determine whether the exposure poses any health risks. In most cases, the exposure levels of ADA from materials are significantly below the ADI, indicating that its use in these materials does not present a significant risk to consumer health. This conclusion is supported by extensive data from both migration and toxicological studies (Mahmud et al., 2023; Ringbeck et al., 2021).

The comprehensive regulatory landscape, safety assessments, and risk evaluations collectively highlight the suitability of ADA for use in FP materials. With its



	ΔE					
FA	10 days	20 days	30 days	40 days	50 days	60 days
Control	3.85 ± 0.47^{Fa}	6.53 ± 0.20^{Ea}	8.24 ± 0.19^{Da}	10.59 ± 0.08^{Ca}	11.45 ± 0.12^{Ba}	12.38 ± 0.17^{Aa}
SPI-BGPI	3.79 ± 0.76^{Fb}	5.57 ± 0.19^{Eb}	7.45 ± 0.22^{Eb}	8.50 ± 0.17^{Cc}	9.58 ± 0.06^{Bc}	10.88 ± 0.19^{Ab}
SPI-BGPII	3.15 ± 0.50^{Fb}	5.90 ± 0.15^{Eb}	7.11 ± 0.13^{Ec}	8.35 ± 0.12^{Cc}	9.33 ± 0.13^{Bc}	10.14 ± 0.11^{Ac}
SPI-BGPIII	4.11 ± 0.50^{Fb}	5.12 ± 0.19^{Ec}	7.75 ± 0.10^{Eb}	9.13 ± 0.23^{Cb}	10.27 ± 0.27^{Bb}	11.18 ± 0.20^{Ab}

Note. Data are expressed as mean ± standard deviation ($n = 10$). $^{A\text{–}F}$Means with different superscripts in the same row are statistically different ($p < 0.05$). $^{a\text{–}d}$Means with different superscripts in the same column are statistically different ($p < 0.05$).

◂**Fig. 6** **A** Visual analysis of fresh-cut apples when wrapped with a control SPI film and SPI film samples containing BGP I, BGP II, and BGP III. **B** Effects of BGP I, BGP II, and BGP III on the color measurements over 60 days of storage. Ref. (Q. Xu et al., 2018) with permission to reproduce from Wiley Online Library under license number 6170380306543

well-documented safety profile, low migration potential, and adherence to global standards, ADA remains a valuable component in the development of sustainable, high-performance BFs for FP. Continued research and regulatory oversight will ensure that its use remains safe, effective, and aligned with both industry standards and consumer safety expectations.

The future of ADA in FP BFs is highly promising, driven by advancements in sustainable materials and green chemistry. One significant trend is the development of bio-based synthesis methods for ADA. Researchers are focusing on producing ADA from renewable resources like biomass and agricultural residues. This shift reduces reliance on fossil fuels and lowers the environmental impact of production. Moving towards bio-based sources not only supports sustainability goals but also meets the growing consumer demand for eco-friendly packaging solutions. Future research should prioritize optimizing these bio-based processes to make them more cost-effective and scalable for industrial applications. Following the development of bio-based synthesis methods, green synthesis methods for ADA are also a critical area of focus. These methods involve using environmentally friendly processes and renewable resources to produce ADA, minimizing harmful byproducts and reducing energy consumption. Green chemistry principles can lead to more sustainable and efficient production methods essential for large-scale industrial applications. Researchers should continue to develop and refine these green synthesis techniques to ensure they are economically viable and environmentally friendly.

Another promising direction is the development of multifunctional biopolymeric films and coatings incorporating ADA, which aim to combine antimicrobial activity, improved barrier performance, and enhanced mechanical strength in a single film. Combining ADA with bioactive compounds and nanomaterials could lead to innovative packaging solutions that extend shelf life, improve food safety, and reduce waste. Future studies should focus on exploring the synergistic effects of these combinations and their practical applications in FP. Additionally, the ongoing evolution of regulatory support and safety standards is crucial for the widespread adoption of ADA in FP. As regulatory bodies emphasize sustainable and safe packaging materials, ADA, with its proven biodegradability and low toxicity, is well-positioned to become a preferred choice. Future efforts should focus on achieving compliance with global regulatory standards and conducting extensive safety assessments to ensure consumer confidence and industry acceptance. Continued research and development are essential for realizing the full potential of ADA in FP BFs. Key areas for future exploration include optimizing bio-based synthesis processes, developing more efficient and scalable methods for producing bio-based ADA, and investigating the combined effects of ADA with other bioactive compounds and nanomaterials. Ensuring that ADA-based materials meet evolving safety and environmental standards will also be critical. Ultimately, the future of ADA in FP BFs holds numerous opportunities for innovation and growth. By focusing on these key areas, researchers and industry stakeholders can develop advanced, sustainable packaging solutions that address both environmental and consumer needs.

Conclusion

Recent research has revealed promising results using ADA as a CLA in degradable FP films. ADA is a green, low-toxicity, easily accessible, and cost-effective agent that significantly enhances various properties of FP films. Studies on animals have consistently shown that ADA has a low toxicity profile and no significant adverse effects at commonly used concentrations in FP. Adding ADA to FP BFs has been found to bring about comprehensive improvements, including enhanced UV light barrier properties, gas barrier properties, water sensitivity, mechanical properties, as well as antioxidant and antimicrobial properties. The effectiveness of ADA in enhancing these properties primarily depends on its concentration and reaction state, which affect the cohesion of the polymer network through CL with the polymer molecules. This CL process primarily occurs through physical interactions, such as H-bonding, hydrophobic interaction forces, electrostatic interaction forces, and *van der Waals* forces, and can be influenced by the temperature and pH of the environment. Furthermore, combining ADA with other additives or plasticizers can synergistically enhance the properties of the film. Also, ADA crosslinked FP BFs and coatings can effectively preserve fresh foods due to the enhanced stability, barrier properties, adhesion properties, antibacterial properties, and oxidation resistance. Additionally, ADA has been demonstrated to enhance the thermal stability and interfacial miscibility of various biopolymers. ADA, being a type of dicarboxylic acid, dissolves in water, with its solubility increasing as the temperature rises. It is also used in mixtures to create crosslinks between polysaccharides and proteins. ADA crosslinked biopolymer matrices offer several advantages over non-crosslinked matrices, including heightened resistance to heat, shear, pH, and freeze–thaw cycles.

Looking into the future, it is essential to focus on the industrial production of ADA crosslinked films as a significant challenge in replacing plastic-based FP films. Additionally, emerging technologies such as cold plasma, pulsed light, and 3D printing require further investigation for ADA-based FP films, as they have the potential to further improve film performance. To further enhance the application of ADA in FP, research efforts should focus on enhancing safety and preventing toxicity in the films that may result from degradation upon exposure to the natural environment. Effective sources for extracting ADA, such as food waste from various processing industries and non-edible parts of plants, can be explored to develop sustainable packaging. These studies can potentially create substantial economic value and offer more sustainable packaging solutions.

Acknowledgements The research protocol was approved and supported by the Student Research Committee, Yasuj University of Medical Sciences, Yasuj, Iran (Grant number: 4040281).

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Funding The research protocol was approved and supported by the Student Research Committee, Yasuj University of Medical Sciences, Yasuj, Iran (grant number: 4040281).

Data Availability Data will be made available on request.

Declarations

Ethical Approval Not applicable.

Competing interests The authors declare no competing interests.

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