

Essential characteristics improvement of metallic nanoparticles loaded carbohydrate polymeric films - A review

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ABSTRACT

Petroleum-based films have contributed immensely to various environmental issues. Developing green-based films from carbohydrate polymers is crucial for addressing the harms encountered. However, some limitations exist on their property, processibility, and applicability that prohibit their processing for further developments. This review discusses the potential carbohydrate polymers and their sources, film preparation methods, such as solvent-casting, tape-casting, extrusion, and thermo-mechanical compressions for green-based films using various biological polymers with their merits and demerits. Research outcomes revealed that the essential characteristics improvement achieved by incorporating different metallic nanoparticles has significantly reformed the properties of biofilms, including crystallization, mechanical stability, thermal stability, barrier function, and antimicrobial activity. The property-enhanced bio-based films made with nanoparticles are potentially interested in replacing fossil-based films in various areas, including food-packaging applications. The review paves a new way for the commercial use of numerous carbohydrate polymers to help maintain a sustainable green environment.

1. Introduction

Petroleum-based polymers have been widely used in day-to-day human activities for many years [1–4]. Most synthetic polymers produced from petrochemical products are not degradable, but they can be recycled after their applications and have limitations when used as packaging materials [5–8]. Environmentally benign biopolymers have been extensively studied in recent years as promising candidates for maintaining a sustainable green environment without compromising the essential properties required for their end-use applications [9–12]. Biofilms derived from carbohydrate polymers can be bioplastics developed from biomass such as cellulose, starch, chitosan and poly(lactic

acid) are the most promising materials to replace conventional petroleum-based films and for other applications [13–21]. The essential properties of carbohydrate polymers are to be considered as promising substituting agents for conventional polymers due to their abundance, biodegradability, biocompatibility, and unique features compared to synthetic polymers [22–25].

The biofilms fabricated from carbohydrate polymers have their limitations in processing and end-use applications due to their slow crystallization, poor mechanical strength, low thermal stability, low gas and water barrier properties [26–30]. Recently many attempts have been made to overcome these drawbacks to enhance the processability of biofilms through physical and chemical modifications. Chemical

Abbreviations: CMC, carboxymethyl cellulose; DDA, degree of deacetylation; DSC, differential scanning calorimeter; E, Young's modulus; EB, elongation at break; OP, oxygen permeability; PBAT, polybutylene adipate terephthalate; PLA, poly(lactic acid); SPI, soy protein isolate; T_c, crystallization temperature; T_{dmax}, maximum decomposition temperature; T_g, glass transition temperature; TGA, thermogravimetric analyzer; T_m, melting temperature; T_{onset}, onset degradation temperature; TS, tensile strength; WVP, water vapor permeability; X_c, degree of crystallinity.

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modifications such as grafting, crosslinking, and branching are the commonly used techniques to modify the properties of naturally occurring carbohydrate polymers such as cellulose, starch, and chitosan. Most of these have film-forming abilities. Introducing metal nanoparticles into these biofilms has enhanced their properties further [30–33]. Innumerable techniques have been developed to incorporate metal nanoparticles into these biopolymers, such as tape casting [34–36], solvent casting [37–39], extrusion [40–42], and thermo-mechanical compression [43–45]. Considering the distinctive features of nanoparticles and biopolymers to produce biofilms may be the best way to enhance the limited properties of the biopolymers. To date, a very limited comprehensive review has been published on various property changes of biopolymers with the incorporation of metal nanoparticles. Hence, this review discusses important carbohydrate polymers, their primary sources, recovery methods, and functional modification techniques for film-forming ability. In addition, nanoparticles incorporate techniques, changes in biofilm properties, difficulties in film-forming processes, and future opportunities are discussed in detail.

2. Carbohydrate polymers in biofilms preparation

Naturally existing carbohydrate polymers are good materials in film-preparing processes to seek different applications. Among the many wide-ranging materials, cellulose, starch, chitosan, and carbohydrate-derived polymer, i.e., poly (lactic acid), possess good film-forming properties. Herein, individual natural polymers and their modifications for the preparation of biofilms are covered.

2.1. Cellulose and its modification

Cellulose is a hydrophobic biopolymer formed from thousands of repeating D-glucose units bonded through the unique β -1,4 glycosidic bonds [46,47]. It is found in nature abundantly and is one of the major components of lignocellulose [48]. The major natural sources of cellulose are plants [49], animals [50], bacteria [51], and algae [52]. Cellulose can also be recovered from different sources to offer film-forming properties. Cellulose can be recovered (68.2 %) from water hyacinth (*Eichhornia crassipe*) using the subcritical water pretreatment method [53], and using sequential two-step treatment, 28.3 % recovery of cellulose fibers can be obtained [54]. Green tea can be used as a substrate for the fermentation of *Acetobacter xylinum* bacteria to produce bacterial cellulose nanofibrils with a diameter of 40 nm [55] and 19.7 % of α -cellulose can be recovered from the marine green algae biomass residue of *Dunaliella tertiolecta* [56].

Cellulose and its derivatives have been widely explored to form films for several applications due to their unique physical and chemical properties, of which bio-degradability and bio-compatibility [57], high viscosity [58], nano-fibrillar three-dimensional network structure [59], optical transparency [60] and tunable porous structure [61] are quite attractive for film preparation. In addition, their insolubility in water and other organic solvents, bio-degradable, chirality characteristics, more crystalline nature than starch, and being an odorless polymer with a density of 1.5 g/cm³ [62] are their necessary characteristics. Since cellulose cannot be processed directly as a film-forming material, so attempts have been made to modify its structure by grafting it with other polymers [63]. Other techniques, such as free radical polymerization, ring-opening polymerization, atom transfer radical polymerization, reversible addition-fragmentation chain transfer polymerization, and nitrogen oxide-mediated polymerization, have been widely explored. Cellulose films and cellulose nano-fibers have been cross-linked with glutaraldehyde by tempo oxidation. The modification of cellulose via crosslinking using epichlorohydrin produced a cellulose-based biofilm.

2.2. Starch and its modification

Starch is a linear and a branched biopolymer present as semi-crystalline granules formed through the photosynthesis of plants [64–68], which comprises two major polysaccharides, namely amylose and amylopectin [69]. Films with extra amounts of amylose normally possess higher film-forming properties, mechanical strength elongation, and gas barrier properties [14]. Recently, 40 % of purple yam starch was recovered using ultrasound-assisted extraction from purple yam starch [70]. Starch recovery from turmeric wastes on a dry basis yielded 3.33 % using pressurized liquid extraction and supercritical technology [71]. The amount of starch recovered on a dry basis from three discarded potato varieties, namely Agria, Kennebec, and Neiker was recorded up to 91.2 %, 83.4 %, and 88.6 %, respectively [72]. Starch-rich seed, namely quinoa (*Chenopodium quinoa*), was processed by wet-milling with an absolute starch recovery in the range of 81.9 to 87.2 % [73].

In recent years, starch has been getting much attention as a film-forming biopolymer due to its biodegradability and low cost [74], abundance [75], non-toxicity, and ease of process as edible films [76], and it has thermoplasticity as a packaging material [77]. Like any other biopolymer, starch has many common properties with other biopolymers, among which its unique properties are poor barrier properties [78], low mechanical strength, and moisture sensitivity [76], which are some of the drawbacks associated with starch. Also, it is bio-degradable [79], insoluble in cold water and tends to retrograde [80]. Different techniques have been employed to modify starch into a film-forming biopolymer. Qiao et al. [81] reported that surface modification, i.e., grafting of starch films by different alcohols would increase its carbon to oxygen mass ratio and mobility of the plasticizers. To use starch for different applications, grafting can be done using nitroxide-mediated polymerization [82]. *Triphala churna* has modified starch nanoparticles via graft copolymerization to make it a promising drug delivery polymer [83]. Native starch, i.e., sago, was passed through oxidation, acetylation, and different chemical modifications, which resulted in acetylated starch showing a superior property in biofilm preparations [84].

To alter the property of carbohydrate polymers, another polymeric macromolecule, i.e., protein, is also combined to prepare biofilms. Protein is a complex biomolecule composed of amino acids linked together to each other, which interacts with many types of carbohydrate molecules [85]. Proteins' technological-functional properties are responsible for the look, texture, and stability of food items [86]. The sources of proteins are natural, which include animals, whey protein from dairies [87] and plants such as soy protein from soya beans [88], and red lentils (*Lens culinaris*) proteins [89]. Proteins, like other biopolymers, can be recovered from different protein-rich wastes using different protein-recovering techniques [90,91]. Different extractants, namely deionized water, NaOH, and Na-pyrophosphate, were used to recover soluble proteins from three soil types: Cambisol, Ferralsol, and Histosol. The maximum recovery achieved was 61–80 % and 45–75 % using NaOH and Na-pyrophosphate, respectively [92,93]. It is biodegradable and have good mechanical strength, excellent oxygen barrier properties [87], oil-resisting capacity [94], have the capacity to produce gels, emulsions, and foams, as well as a valuable nutritional index [95] having a high molecular weight and good heat stability [96]. The proteins are stable at different pH, temperatures, and salt levels and have poor water vapor resistance [97], solubility, hydration, and swelling power [98–101].

2.3. Chitosan and its modification

Chitosan is a naturally abundant polycationic polysaccharide next to cellulose composed of poly- β -1,4-linked glucosamine and N-acetyl-d-glucosamine [102]. Chitosan is usually obtained by deacetylation of chitin [103]. It is only soluble in acidic solutions due to the protonation of the NH₂ groups on the C₂ position of the D-glucosamine repeat units

[104]. The natural monomer source of chitosan is chitin which is derived from biological sources like crustaceans [105], insects [106], the cuticle of some arthropods [107], and the cell wall of fungi [105]. There are several other methods to recover and extract chitosan from biological sources.

Using Graviolaextracts as an extractor, chitosan with a high degree of deacetylation, about 94.86 % on the 3rd day, was recovered from commercially available chitin and seafood waste [108]. The chitin from Shrimp shells (*Litopenaeus vannamei*) was treated with 12.5 M NaOH at 65 °C for 12 h to recover chitosan with 56.10–88.76 % degree of deacetylation [109]. On the other hand, chitosan from Shrimp shell waste using a common chemical method was recovered with a degree of deacetylation of 88.89 % [110]. Doğdu et al. (2021) [111] isolated the chitosan from the exoskeleton of the invasive swimming crab *Charybdis longicollis* using chemical methods and the yield of chitosan produced from extracted chitin was 80.23 %. Using chemical techniques, recovery of chitin and chitosan from *Callinectes amnicola* (crab) and *Penaeus notialis* (shrimp) shell wastes was performed. A higher chitin yield of 26.08 %, a higher chitosan yield of 16.93 %, and a higher degree of deacetylation (DDA) of 89.73 % were obtained from the shrimp shell whereas crab shell yielded chitin of 19.36 %, chitosan of 13.29 % and the DDA of 84.20 % [112].

The physicochemical properties that attract the research society to select and process chitosan as a film-forming biopolymer are its molecular weight and DDA [113], antioxidant and antifungal activity [114–116], suitable carrier property for other materials [117], non-toxic, biodegradability and biocompatibility [118]. The specific physico-chemical properties associated with chitosan are it is insoluble in water and common organic solvents, but it is soluble in weak acids, has poor mechanical properties [119,120], has a poor water resistance [40], an excellent oxygen barrier [121], large scale availability [122] and presence of practical groups for chemical adjustments [123].

A unique feature of chitosan that attracts many researchers can potentially be used when it is functionally modified. The most common modification techniques to this particular biopolymer are crosslinking and grafting. The grafting of chitosan film with corrole macrocycle resulted in property enhancement of the chitosan film. Using the carbodiimide-mediated grafting method, chitosan was functionalized by three different hydroxycinnamic acids to bring up the chitosan film into packaging material [124]. Liu et al. (2021) [125] conducted the same grafting method as the previous one, and five different hydroxybenzoic acids were grafted on chitosan with different grafting ratios. Three different dialdehyde derivatives were used to be cross-linked with chitosan to bring up the chitosan films into biomedical applications [126]. Khouri et al. (2019) [127] conducted heterogeneous cross-linking of chitosan with citric acid by varying concentrations of a model cross-linker to obtain its effect on the viscoelastic properties of the chitosan films.

2.4. Poly (lactic acid)

Poly(lactic acid) is bio-based, bio-degradable, and bio-compatible linear aliphatic thermoplastic biopolymer that can be produced by the most well-known production method, which is ring-opening polymerization which results in the production of optical isomers of poly(lactic acid) [63,128–130]. This polymer source is usually lactic acid found in nature, particularly in sugar cane, corn starch, and other bio-mass products [131,132]. Poly(lactic acid) possesses attractive physico-chemical properties to be utilized as a biofilm-making biopolymer and these are its high elastic modulus and high transparency [133] and good mechanical property, i.e., tensile strength [125]. It is non-toxic and bio-compatible for foods [131], good thermal processibility [134]. The properties of poly (lactic acid) include low thermal stability [135], low crystallinity [136], and high brittleness and bio-compatibility [137,138].

In addition to Poly(lactic acid), (butylene-adipate-co-terephthalate)

is a flexible synthetic aliphatic-aromatic co-polymer with outstanding hydrophilic and processing qualities used for biofilm preparation [139]. It consists of two types of comonomers, a rigid butylene terephthalate, composed of 1, 4 butanediol and terephthalic acid monomers. In contrast, the flexible butylene adipate portion is made up of 1,4 butanediol and adipic acid monomers [140]. It is derived from common petrochemicals—purified terephthalic acid, butanediol, and adipic acid, yet biodegradable [141]. Different polymers and their common sources are presented in Table 1. The properties exhibited by this particular polymer play a vital role in utilizing it as a potential film-making and blending polymer and those properties are biodegradability(from aliphatic moiety) [142], good mechanical property (from the aromatic part of PBAT) [143], good crystallization and thermal stability [16], high ductility [144], high elongation at break [145], good processability, high flexibility and toughness [140] has low density [146], good permeability to gas and vapors [147] and good moisture resistance, stretching and impact [148,149]. It is a polymer that possesses different unique physical properties. Among them, the high melt viscosity and crystallization rate [150], low elastic modulus [151], and insoluble in water [152] are the major properties that are usually considered in processing it for different applications. Various biopolymers and their unique features are listed in Table 2.

3. Biofilms preparation methods

Biopolymers can be described as natural polymers with different attractive properties, which direct the need for synthesizing and utilizing them for various applications [218]. Biopolymers can be obtained from plants, animals, and microorganisms or synthesized from different monomers [7]. The commonly available biopolymers, such as starch, cellulose, proteins, gluten, polyaniline, poly-lactic acid, and poly-butylene adipate-co-terephthalate, have widely been focused on preparing biofilms typically utilized in food packaging, tissue engineering and medical applications [103,146]. Various methods have been employed to prepare biofilms with different principles adapting numerous biopolymers [36,42,43,59]. Bio-based film preparation techniques and methods are unique and different based on the type of polymers, application areas and properties of the polymers, and the filler. The elementary biofilm preparation procedures, merits, and demerits of each method are discussed in the following section.

Table 1

Carbohydrate and other potential polymers with excellent film-forming capacities can be extracted, recovered, and produced from different natural sources. It is essential and crucial to derive biopolymers from their natural source and select the polymers for film preparation with different applications.

Biopolymer	Sources	References
Cellulose	Tomato plants	[153–155]
	Bactria	[156–158]
	Green algae	[159–161]
	Animals (tunicates)	[162–164]
Starch	Cassava	[165–167]
	Potato	[168–170]
	Rice	[171–173]
Protein	Whey (diary)	[174,175]
	Soya bean (oil extraction)	[176–178]
	Red lentil	[179–181]
	<i>Moringa oleifera</i>	[182–184]
Chitosan	<i>Vicia villosa</i>	[185–187]
	Marine shell-fish wastes	[188–190]
	Oyster shell	[191–193]
	Fungai	[105,194,195]
	Black soldier fly	[106,196,197]
Poly (lactic acid)	Sugar cane	[198–200]
	Lactose	[201–203]
	Corn starch	[204,205]
	Wheat	[206,207]

Table 2

Carbohydrate and other potential polymers and their unique features are preferable as film-forming materials. These unique features may have a positive as well as a negative impact on the nature of the films and their application. In addition to these features are common features like biodegradability, non-toxicity, and bio-compatibility are possessed by all the polymers with film-forming ability.

Bio-polymer	Unique features	References
Cellulose	<ul style="list-style-type: none"> • 3D network structure • Optical transparency and tunable porous structure • High viscosity • Processable as edible film or coatings 	[39,49]
Starch	<ul style="list-style-type: none"> • Abundance • Non-toxic • Excellent oxygen barrier properties • Ability to form gels, emulsions and foams 	[208,24]
Protein	<ul style="list-style-type: none"> • Valuable nutritional index • Good anti-microbial and anti-fungal properties 	[209,210,211]
Chitosan	<ul style="list-style-type: none"> • Suitable carrier property for other materials • Large-scale availability and presence of practical groups for chemical adjustments • Good thermal processibility 	[108,118,212]
Poly(lactic acid)	<ul style="list-style-type: none"> • Bio-compatible for foods • High transparency • Excellent thermal stability 	[213–215]
Polybutylene adipate terephthalate (PBAT)	<ul style="list-style-type: none"> • Very tough and ductile • Superior mechanical and optical properties 	[16,216,217]

3.1. Tape casting method (spread casting or knife-coating)

The tape-casting method involves casting the film-forming suspension on the polymer-covered tape-casting device. Doctor blades are used for uniform casting of the film-forming suspension [35,36]. A shifting tape casting apparatus with two doctor blades was used to cast a Poly (lactic acid) (PLA)/Mg solution on a glass plate [36]. The tape-casting method of biocomposite film preparation is displayed in Fig. 1. Different polymer compositions were used to prepare a film-forming suspension. Here, cellulosic fibers were used to prepare a fibrous suspension, blended with glycerol and starch to prepare a film-forming suspension at 71 °C. Then, the suspension was spread on the polyethylene terephthalate film-covered tape-casting device using two doctor blades [219].

A film-forming suspension of Soy protein isolate (SPI) and glycerol with different concentrations is formed and cast on polyester film using blades at 60 °C [35]. At 75 °C, at a heating rate of 1 °C/min, a

combination of cellulose fibers, water, starch, and glycerol created a film-forming suspension in a thermal bath, and the suspension solution was cast on the plexiglass plate using three different doctor blade spacing [220]. The advantages associated with this method are the possibility of preparation of films with large dimensions, short drying times, and film thickness controlling ability, but its demerits are that it is costly in operating as well as production, and it is energy-intensive [221].

3.2. Solvent casting method

This method is a classical method of film preparation. A suspension of different components with proper viscosity will be prepared using different solvents and plasticizers. The film-forming suspension is cast on the Petri dish and dried without any external force except the temperature [37,59,222]. The solvent-casting method of biocomposite film preparation is demonstrated in Fig. 2. MgO/Ag nanoparticles with different concentrations mixed with polybutylene adipate terephthalate (PBAT) dissolved in chloroform to prepare PBAT/MgO/Ag solution. The solution was poured into a petri dish and allowed to dry in the open air at room temperature [38]. Biofilms are developed by mixing chitosan and ϵ -polylysine and heating the solution at 50 °C. The heated solution was coated onto glass and allowed to dry for 24 h in the open air [37]. E-Poly-L-lysine biofilm is developed in a way that a given amount of starch, glycerol as a plasticizer, and E-Poly-L-lysine mixture are stirred and heated at 95 °C, then incubated at 37 °C [223].

Bacterial cellulose-based composite films with carboxymethyl cellulose, glycerol, and yeast were developed using a concentration-dependent blending of the components and the blended components were exposed to vacuum and heat at 45 °C [59]. Glucmannan biofilms were formed by adding sulfonyl and sorbitol (surfactant and plasticizer) to the glucmannan solution that was stirred at 6000 rpm and 35 °C. The gel solution was put into a Petri plate and dried at 50 °C with a relative humidity of 11.4 %. MgO/Ag reinforced poly(butylene succinate-co-terephthalate) composite films was developed through a solution-casting method with varying concentrations of nanofillers distributed in chloroform dissolved poly(butylene succinate-co-terephthalate) and the mixture was poured and dried for 2–3 days on a glass plate. The films were approximately 20 μ m thick [224].

Solvent-supported film preparation was used to develop nano-magnesium oxide-reinforced poly (lactic acid) films with the greatest improvement in tensile strength and oxygen barrier characteristics. A bird-type manual film applicator was employed for chloroform evaporation to generate consistent thickness films [225]. The advantageous part of using this method would be easily performed and protects denaturation materials from high operating temperatures, but the demerits of this method are high cost, long drying time, cannot produce a mechanically stable film, control of film thickness hard and it is impossible to produce industrial-scale films [224,225].

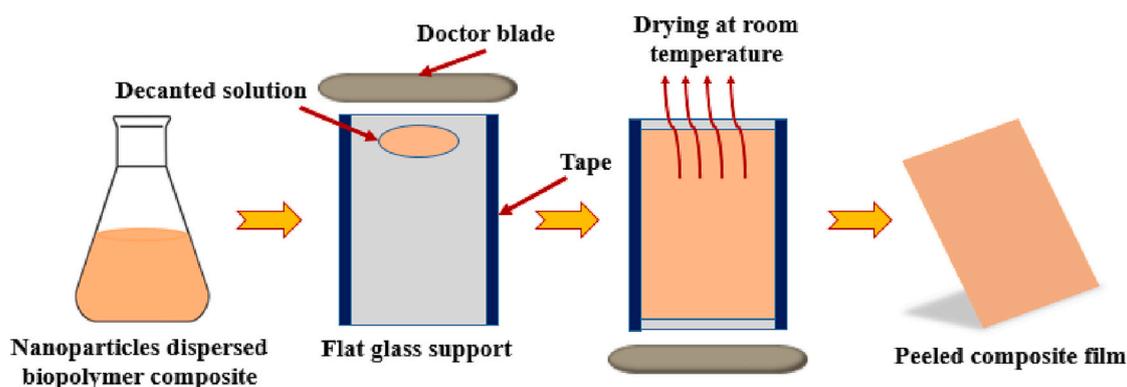


Fig. 1. Tape-casting method of biocomposite film preparation.

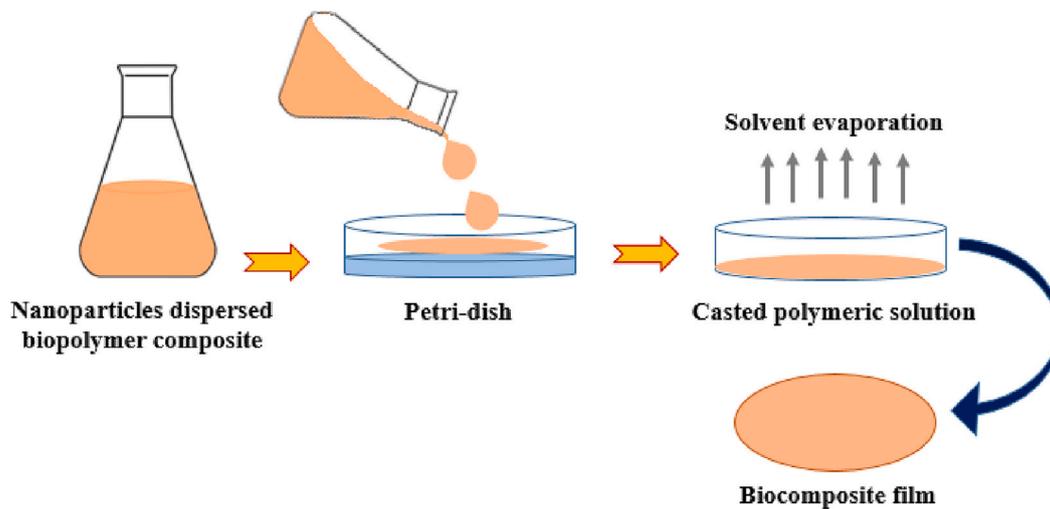


Fig. 2. The solvent-casting method of biocomposite film preparation.

3.3. Extrusion

One biofilm preparation method usually involves blending different components into a homogenized mixture, forming the extrusion strands of the compounded mixture using a single screw, pelletizing or calendaring the extruded strands, and conditioning the films at room temperature. It is a temperature-based process and usually, the temperature profiles associated with this method are known to be a feeder and die temperature [42,226]. The extrusion method of nanoparticles incorporated biocomposite film preparation is illustrated in Fig. 3. Egg white protein films were obtained using extrusion followed by calendaring the extrudates; a mixture of egg powder, water, and glycerol was used as a solution. The need for calendaring using drum dryers after forming extrudates using different feeders and die temperatures is to obtain a plastic-like structure [226]. Polyvinyl alcohol/starch/clay nanocomposite films were developed through an extrusion blowing system, but here, compounding the above mixture came before the blowing to form and conditioned pellets at 25 °C and 53 % relative humidity followed by extrusion blowing at five different temperature zones [13].

A biofilm obtained from corn starch and whey protein blends was successfully produced through extrusion. Here pellets of the above blend were produced using co-rotating twin screws with 7 different heating zones that lie between 70 and 110 °C and then a single screw extruded the pellets with 4 different heating zones to form film sheets, which were

conditioned at room temperature [227]. Poly(butylene adipate-co-terephthalate) films can be developed through the extrusion casting method with a temperature profile of 140 °C and 150 °C for feeding and die zones, respectively. The screw rotation speed was 40 rpm [228]. A homogenized blend of polypropylene-chitosan-poly (lactic-acid) was extruded by a single screw that revolves at 25 rpm where the feeder and die temperature zones were 165 and 175 °C and the thickness of extruded film lies between 0.2 and 0.5 mm [229]. Tear-resistant, biodegradable, as well as flexible films of poly (lactic acid), were formed using different biopolymers and plasticizers through the extrusion technique, where the mixture was extruded at 11 different temperature zones that ended at a drying temperature of 140 °C, which then pelletized and blown at 150 °C, 50 rpm and melt pressure of 150 bar [133].

Poly (vinylidene fluoride) (PVDF)-based piezoelectric films were obtained through the extrusion method by mixing powder of PVDF/GO/PZT with alcohol and followed by drying to condition the composite. Then the conditioned mixture was granulated and extruded at 250 °C, which resulted in film thicknesses of 30–120 μm [40]. Uniformly glycerol, water, and alcohol-diffused starch were extruded with 10 different profile temperatures that ended at a drying temperature of 90 °C followed by pelletizing and hot pressing the extruded strands at 130 °C. The films were conditioned at 23 °C and 58 % of relative humidity [230]. The advantages of extrusion film processing over the classical

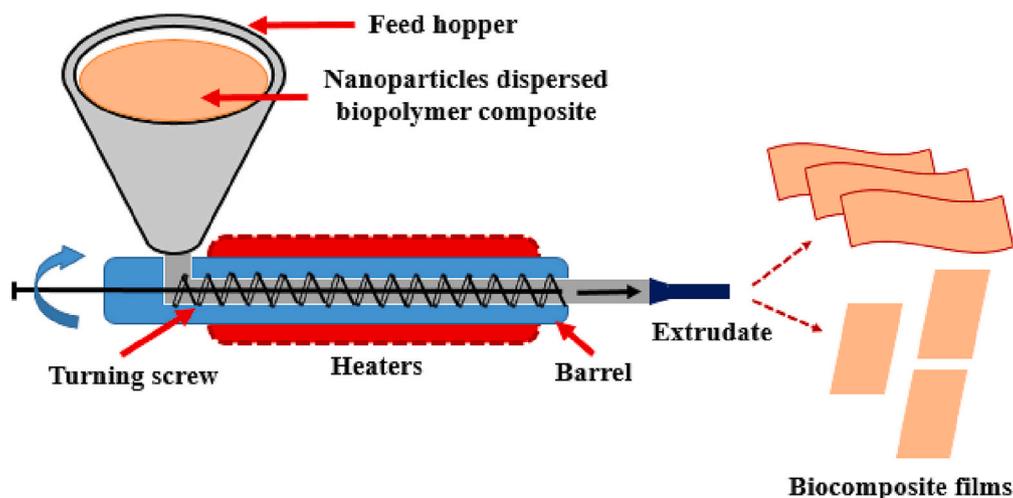


Fig. 3. Extrusion method of nanoparticles incorporated biocomposite film preparation.

ones are the films can be produced with large film thickness and possess high efficiency, flexibility, high tensile strength, commercial viability, dielectric constant, breakdown strength, and low cost compared to the solution casting method, but it is energy-intensive, the films produced through this method has low water permeability [13,40,133].

3.4. Mechanical and thermal compression

Thermal compression is one of the very well-known film preparation methods. It is known as the drying process because the films go through thermal compression and pressure. In low moisture content circumstances, thermoplastic biopolymers are heated above their glass transition temperature [7,43]. The mechanical and thermal compression method of nanoparticles incorporated biocomposite film preparation is illustrated in Fig. 4. Cellulose Nano fibril films were developed by drying the filtered cellulose suspension (wet films) through different drying methods and the dried film was finally compressed thermally at 120 °C and 1.1 MPa for 10 min [7]. Soy protein films were developed through a thermal compression in which soya protein and glycerol suspension were placed between two aluminum plates introduced to the press heated to 150 °C, and pressed at 12 MPa for 2 min [45]. Nano-fibrillated-cellulose-graphene (GNs/NFC) films can be developed from GNs/NFC suspension where a homogenized GNs/NFC suspension was obtained through filtration, and the filtered suspension (wet film) was dried at ambient temperature and further squeezed for 10 min at 150 MPa [231].

A citric acid cross-linked chitosan film was obtained through thermal and mechanical compression of a plasticized chitosan suspension which was placed and pre-heated between aluminum plates at 125 °C and pressed at 2.5 MPa for 2 min [44]. Liquid crystalline monomer-poly (vinyl alcohol) (LCM-PVA) dispersed films can be developed through filtration and mechanical compression of LCM-PVA suspension at 5 MPa and 160 °C for 5 min, then cooled up to room temperature [43]. The advantage associated with this method is to allow less time-consuming manufacturing, its ease of use, and the capacity to produce films without the need for solubilization, but due to high-temperature protein denaturation occurs for protein films [43,232]. A summary of biofilm preparation methods from different biopolymers is presented in Table 3.

4. Improved characteristics of biopolymer-derived films using metal nanoparticles

As discussed in the previous chapter, film-forming biopolymers have their own unique and attractive properties to be utilized as film-forming polymers. However, these biopolymers also exhibited poor properties, making them not to be used as potential substitutes for petroleum-based plastics. For that matter, different researchers have implemented various scientific methods to tackle these poor properties of the biopolymers, for instance, blending with other biopolymers, using inorganic and organic fillers, i.e., nucleating agents. The most recent property improving the technique is using metal nanoparticles as property-enhancing agents.

Table 3

Summary of biofilm preparation methods from different biopolymers. The merits and demerits of the preparation methods are significant factors in determining the properties of films to be produced.

Film preparation methods	Advantages	Disadvantages	References
Tape casting	<ul style="list-style-type: none"> • Possibility of preparation of films with large dimensions • Short drying time • Film thickness controlling ability • Easy operation 	<ul style="list-style-type: none"> • Energy-intensive • High operating cost 	[36]
Solvent/ Solution casting	<ul style="list-style-type: none"> • Protects denaturation (especially for protein) from high operating temperature 	<ul style="list-style-type: none"> • Long drying time • Lack of film thickness control 	[39,37]
Extrusion	<ul style="list-style-type: none"> • Production of films with large film thickness • Production of films with flexibility, high tensile strength • Less time-consuming manufacturing • Simplicity 	<ul style="list-style-type: none"> • Energy-intensive • Films produced through this method have low water permeability 	[40,41,133]
Compression	<ul style="list-style-type: none"> • Ease of use and capacity to produce films without the need for solubilization 	<ul style="list-style-type: none"> • Due to high temperatures, protein denaturation occurs 	[43,232]

4.1. Crystallization/nucleation property

In theory, polymer nucleation may be described as the initial random generation of a unique thermodynamic new phase that can irreversibly expand into a bigger-sized nucleus within the body of the metastable parent phase [233]. Any polymer's crystallization enhancement is affected by the type of filler and its size and form. The importance of enhancing the crystallization of a specific polymer widens its application area and its processibility (under thermal and stress) and increases the utilization rate of the polymers. The mechanism in shifting the crystallization properties of bio-polymers resulting from the incorporation of metallic NPs is usually related to the changes in the polymer's glass transition temperature (T_g) and melting temperature (T_m). The NPs would affect these specific properties by diverting the chain mobility and crosslinking density of polymer structures. The mechanism of property changes of biocomposite films is illustrated in Fig. 5.

Pure PLA was blended with copper nanoparticles. The crystallization enhancement was significant (from 76.9 % to 79.8 % with 0.043 wt% of Cu and 84.4 % with 0.074 wt% of Cu) [234]. As Nanofillers, graphene

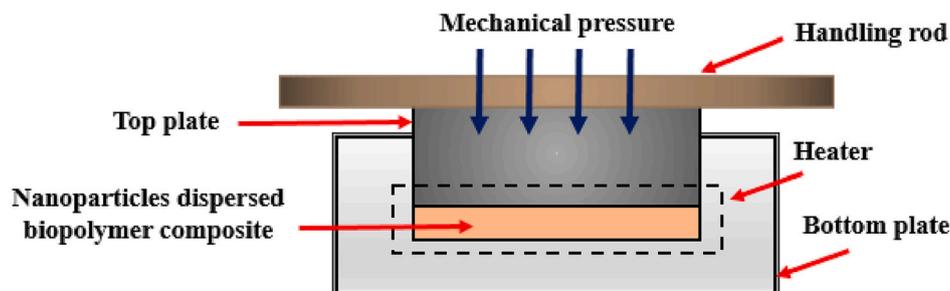


Fig. 4. Mechanical and thermal compression method of nanoparticles incorporated biocomposite film preparation.

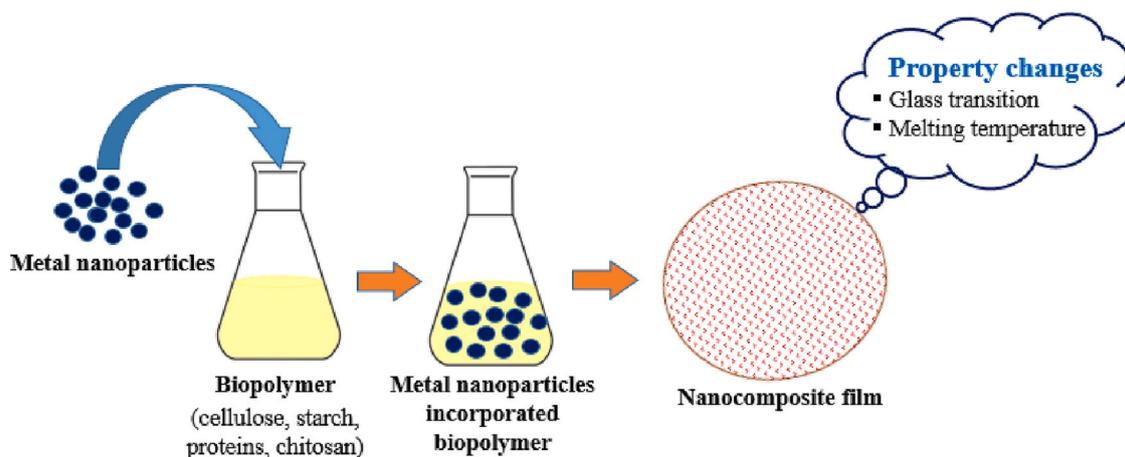


Fig. 5. Effect of metallic NPs on crystallization properties of biocomposite films.

oxide and multiwalled carbon nanotubes containing silver particles in various concentrations were employed to construct nanoparticle-dispersed poly (lactic acid) films. The inclusion of nanoparticles boosted the crystallization rate substantially, whereas the glass transition and melting temperatures of the PLA-based films remained almost constant [235]. The nano-ZIF-8@GO hybrids, which serve as a nucleating agent, were blended with poly(lactic acid) to increase the crystallization property and resulted in slightly higher glass transition temperatures (T_g) and high crystallization temperature (T_c), but the melting temperature (T_m) of the PLA nanocomposites is decreased [26]. A poly (lactic acid) film distributed with silver phosphate, zinc oxide, and nano-hybrids was created. The degree of crystallization rose dramatically from 2.8 % for the clean PLA film to 23.5, 23.2, and 25.4 % for Ag (2 wt%), Ag-Zn (2 wt%), and Ag-Zn (4 wt%), respectively [236].

The crystallinity of PLA increased by 95.6 %, 107.1 %, 118.6 %, 105.3 %, and 110.6 %, with the dispersion percentage of nano-Ag filler ranging from 0 to 15 wt%, respectively, and the crystallization temperature decreased by 2.7 °C in PLA nanocomposites with 15 wt% nano-Ag filler, but there was no significant difference in T_g with increasing nano-Ag content [99]. For a different dispersion rate of silver nanoparticles in PLA, a thermal analysis was performed with a heating rate of 0.1 °C/min. The result showed that the glass transition (T_g) for the pure PLA occurred at around 60 °C, and for the samples loaded with Ag nanoparticles, the glass transition (T_g) temperature proceeded at a higher temperature [237].

Zinc oxide nanoparticles (ZnO-NPs) dispersed with different loading rates in the poly (lactic acid) to develop a biofilm. The melting temperature (T_m) rose very slightly as ZnO NPs concentration increased, but T_c declined as a result of the decreasing influence of ZnO NPs on PLA crystallinity [238]. The nucleation effect of titanium dioxide (TiO_2) was studied on PLA/ TiO_2 films, which resulted in an increment in crystallization temperature, no change in glass transition and melting temperatures as the amount of TiO_2 increased, but the degree of crystallinity was enhanced from 19.63 % (neat PLA) to 22.38 % (PLA/0.6 wt% TiO_2) [239].

By combining thymol and ZnO-NPs with poly (lactic acid)/poly (caprolactone)/thermoplastic starch ternary blends, novel thin sheets were created (lactic acid). The effect of zinc oxide nanoparticles on the crystallization property of the polymer was studied. The integration of ZnO-NPs resulted in a decrement in the cold crystallization temperature and a modest rise in the degree of crystallinity of PLA, which is ascribed to the presence of ZnO-NPs as the nucleating agent [240]. The melt mixing process was used to create polylactic acid-MgO nanocomposite films. The DSC results show that a small amount of 1 % MgO NPs increased the polymer's glass transition temperature (T_g), but higher reinforcement does not result in the expected increase in T_g , which

could be due to agglomeration of MgO NPs at higher concentrations, and the T_m and X_c of blown NC films show no significant change with MgO reinforcement [225].

Poly(lactic acid) has a transition temperature (T_g) of 60.3 °C, while LDHs@PACu(II)/PLA nanocomposites have a slightly higher T_g . This can be attributed to the stronger interaction of PA-Cu (II) coatings to change the mobility of PLA chains related to the glass transition. The χ_c computed of LDHs@PA-Cu (II)/PLA nanocomposites are substantially higher than that of pure PLA as LDHs@PA-Cu(II) concentration rises, illustrating the nucleating action generated by LDHs@PA-Cu(II) (II). Furthermore, the T_m of LDHs@PACu(II) /PLA nanocomposites increases somewhat [241]. PLA film had a glass transition temperature of 58 °C, and the addition of cellulosic nanofiber (CNF) and Ag NPs had no effect. When compared to the clean PLA sheet, the crystallization temperature of nanocomposites rose by 1–2 °C. In contrast, the melting temperature of the nanocomposite was somewhat lower than that of PLA film [153].

The DSC scans revealed that ΔH_c is slightly greater than ΔH_m for all of the films, indicating that there is no crystalline phase in the crystal structure of the films based on the aforesaid relationship. Furthermore, adding MgO NPs had no notable effect on the T_g or crystallinity of the films [225]. The inclusion of ZnO NPs (5 wt%) enhanced the T_g , T_c , and T_m values of PLA/ACNC/ZnO composite films marginally due to the suppression of PLA chain motion. It was caused by the good dispersion of ZnO in composite films, and the X_c values of PLA/ACNC/ZnO composite films initially declined and subsequently climbed when the extra ZnO content was above 5 wt% [242]. The incorporation of TiO_2 nanoparticles enhanced the crystallinity percentage from 14.2 % to 18.2 %. Also, the addition of 2 and 4 vol% NPs, resulted in a modest decrease in the T_c of the clean PLA from 130 °C to 129 and 128 °C [243].

Thermal analysis of PBAT/ZnO nanocomposite films was performed, resulting in a slight increment in T_g values of nanocomposite films than neat PBAT. In contrast, the addition of ZnO nanoparticles doesn't change the T_m but decreases the crystallinity degree (χ_c) [140]. When MgO@CTAB nanoparticles are dispersed (0, 1, 3, 5 and 7 wt%) in biodegradable poly(butylene adipate-co-terephthalate) (PBAT) films, the crystal structure of the film is affected. This effect can be explained so that the initial crystallization temperatures and T_c of PBAT nanocomposites shift to lower values, and the peak melting temperature (T_p) exhibits a decreasing trend. Still, the crystallinity values (χ_c) of PBAT are increased [64].

The DSC test results showed the synergetic effect of both multiwalled carbon nanotube and ZnO NPs on both PBAT and MPBAT. With the addition of 1 % of both NPs, the modified PBAT films exhibit lower crystallization and melting temperature [244]. Copper nanoparticles dispersed PBAT films were thermally analyzed and the degree of crystallinity increased as the concentration of Cu-NPs increased [245]. The

melting temperatures (T_m) of butylene adipate (BA) and butylene terephthalate (BT) segments of neat PBTA film are 58.8 and 114 °C, respectively. As AgNPs@SiO₂ loading increases, the melting temperature of the flexible BA segment becomes 57 °C, while the T_m of the rigid BT segment increases to 110 °C [246].

With the increased pattern of MgO/Ag NPs content from 0 wt% to 5 wt%, T_c of PBAT film increased from 74 °C to 81 °C and there was no change in the glass transition, and melting temperatures [247]. Improving the crystallinity (nucleation) property is a determining factor for utilizing the bio-polymers for different applications. In measuring the enhancement of the nucleation property of biopolymers, four essential parameters that should be considered are cold crystallization temperature (T_{cc}), degree of crystallinity (χ_c), and glass transition temperature (T_g) and melting temperature (T_m). It can be concluded that a biopolymer's nucleation property is enhanced with a positive change in those parameters. The effect of different NPs on the crystallization property of bio-based films is indicated in Table 4.

4.2. Mechanical property

Mechanical characteristics are physical qualities a substance shows when forces are applied. The modulus of elasticity, tensile strength, elongation, hardness, and fatigue limit are examples of mechanical parameters. These properties of biopolymers change dramatically with temperature, limiting their application area in industries. Therefore, enhancing the mechanical properties of any biopolymers is essential in that it helps produce eco-friendly materials that can resist any force applications. Many scholars have been using metal nanoparticles to enhance these particular properties of biopolymers. The following section discusses the effect of the metal oxide nanoparticles on the mechanical property of biopolymers.

The mechanical properties of cellulose film, leaf-extracted cellulose film, and cellulose nanocomposites were studied, which resulted in high elongation at the breakpoint as the concentration of bimetallic (Ag and Cu) nanoparticles increased, high tensile strength and tensile modulus observed up to some concentration of NPs but starts to decline as the concentration increases more [30]. The tensile strength (TS) was significantly increased for carboxymethyl cellulose film when ZnO-NPs were incorporated into the films. However, the TS and stiffness of the film decreased with the high content of NPs, but no significant change was observed in the flexibility of the film [28]. Incorporating metallic NPs involves shifting the mechanical properties of any polymeric bio-films. In such a way, adding NPs might create additional structure, i.e., cracks and holes, to the biopolymers, which either lowers the stress-withstanding ability or it might help in resisting stress on the composite films. In some cases, it might be inserted between cross-linked chains of the polymers, which results in the shifting elongation property of biofilms. The mechanical property changes of biocomposite films with metallic nanoparticles are shown in Fig. 6.

The incorporation of MgO up to 1.5 % had no discernible impact on the tensile strength of the prepared CMC/PVA film containing MgO

Table 4
Effect of different NPs on crystallization property of bio-based films.

Polymer and incorporated NPs	Crystallization property	Reference
Poly (lactic acid) and Cu	• X_c from 76.9 % to 84.4 % at low concentrations of NPs	[234]
Poly (lactic acid) and Ag	• Significant change in crystallinity occurred, yet there was no change in T_g	[99]
Poly (lactic acid) and TiO ₂	• Degree of crystallinity was enhanced from 19.6 % to 22.4 %	[239]
PBAT and MgO/Ag	• T_c increased significantly, but there was no change in T_g and T_m	[146]
PBAT and ZnO	• Slight change in T_g , T_m was not affected and X_c was decreased	[140]

[248]. The untreated and free paper sheets results reveal that metal deposition is unsuitable for strength properties. However, it causes a decrease in the tensile index. This undesirable observation is reduced by pre-treating paper sheets with chitosan (Ch) and cellulose diacetate (CDA) [249]. The CMC film's tensile strength (TS), elongation at break (EB), and elastic modulus (EM) were 42.8 MPa, 16.7 %, and 1.56 GPa, respectively. The addition of ZnO NPs did not affect the strength or stiffness of the CMC film, but the TS rose to 49.3 MPa when 0.5 wt% curcumin and 1.0 wt% ZnO were added [250]. The plain has a tensile strength and an elongation at break of Tensile strength and elongation at break of cellulose acetate (CA) film were enhanced from 35.5 MPa and 1.6 % to 49.2 MPa and 2.9 %, respectively, with 7 wt% Cu-MOF incorporation [251]. The addition of AgNPs on tempo-oxidized nanocellulose (TNC) significantly enhanced the mechanical property of the film, causing the AgNPs to occupy the TNC film's interspace [27].

Ghozali et al. (2020) [78] investigated the thermoplastic starch/metal oxide biocomposites prepared and the effect of these metal oxides on the mechanical property of the film. The tensile strength of TPS has been enhanced as the concentration of ZnO and TiO₂ NPs increases but declines as the loading rate maximizes. The ZnO-chitosan nanocomposite was produced and incorporated into starch films to enhance the mechanical property of the films and resulted in high tensile strength when the loading rate of NPs was 3.0 wt%. On the contrary, the elongation at break decreased [79] and Hajizadeh et al. (2020) [252] observed that the TS values of films loaded with both Ag-modified SiO₂ and TiO₂ NPs were considerably greater at all concentrations and the EB values of films containing SiO₂ and TiO₂ NPs considerably lower than those of pure starch film at all concentrations.

The addition of nano-SiO₂ greatly improved the tensile strength of potato starch films, with the 100 nm nano-SiO₂ providing the best tensile strength [26]. The results revealed that 0.5 % ZnO-NPs had no influence on the TS but dramatically increased the YM and EB values of the SC film. When the concentration of the NPs was increased to 1 %, the TS value reached 4.74 MPa while the YM value dropped to 17.31 MPa, and when the concentration of the NPs was increased to 2 %, the TS and EB grew even more, but the YM value reduced [253]. By adding 0–5 wt% of OEO, the tensile strength and tensile modulus of PSt-Ag NPs were significantly increased by up to 80.88 % and 88.63 %, respectively, due to the reinforcing action of homogeneously dispersed Ag-NPs in the matrix. Ag NPs aggregation at 7 % OEO reduced tensile strength and modulus [254].

Copper nanoparticle dispersed PLA film was studied and found to have a greater Cu content (0.082 wt%), lowering the EB value by 14.9 wt % compared to PLA-Pr. There are no statistically significant variations in Young's modulus. Copper nanoparticles resulted in a significantly greater strain at break [234]. PLA/PBAT composite film with TS of 14.5 MPa was analyzed and the TS of PLA/PBAT films rose to 23.4 ± 3.9 and 26.9 ± 2.8 MPa when ZnONP^{ZA} and ZnONP^{ZC} were added, respectively but increased to 16.8 ± 3.7 MPa when ZnONP^{ZN} was added. The modulus of elasticity, on the other hand, (EM) (stiffness) increased considerably, but elongation at break (EB) was significantly decreased, in which the EB (flexibility) of PLA/PBAT film was 199.9 ± 32.2 %. But when ZnONP^{ZA}, ZnONP^{ZC}, and ZnONP^{ZN} were incorporated into PLA/PBAT film, it decreased to 118.1 ± 23.9 %, 121.8 ± 14.1 %, and 142.3 ± 26.8 %, respectively [255].

When 1 wt% LDHs@PA-Cu is added to LPCP1, exhibiting a 53.0 % improvement in elongation at break and an 18.9 % increase in tensile strength when compared to pure PLA (II). Still, it shows a decrement in TS and EB when the LDHs@PA-Cu(II) concentration exceeds 5 wt%, yet better mechanical properties than pure PLA [241]. The tensile strength of pure PLA film is 4.2 MPa. After the incorporation of 0.25 wt% of NiO with PLA, the tensile strength increased to 7.12 MPa, and the maximum value of tensile strength of 16.54 MPa was achieved at 1 wt% of NiO [256]. Zinc oxide nanoparticle was dispersed in Poly (lactic acid). Its effect on the mechanical property was investigated, which resulted in declining tensile strength as the ZnO loading rate increased. PLA with 1

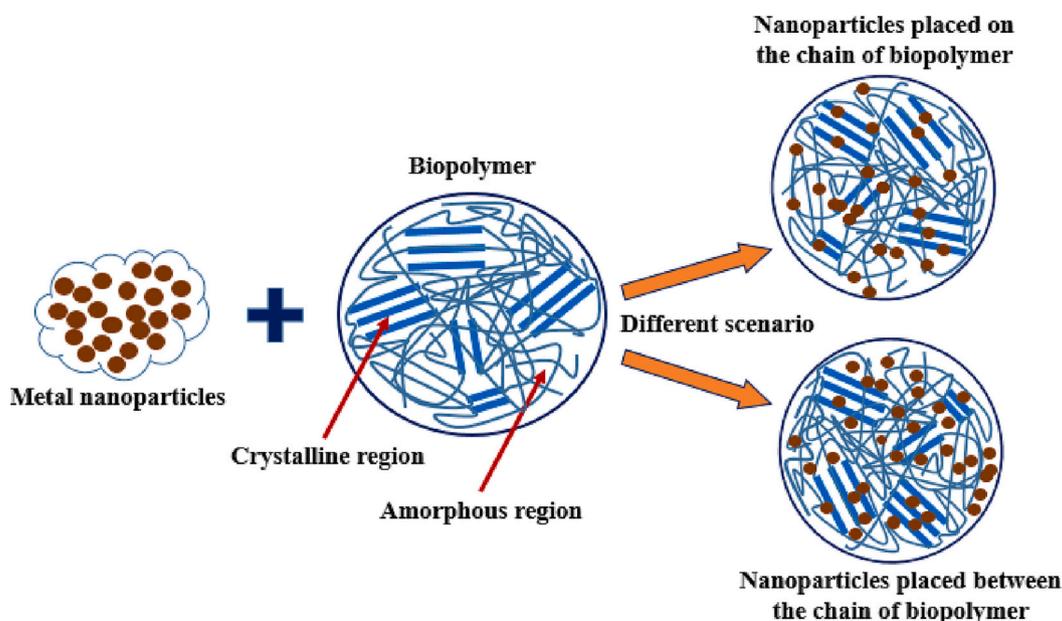


Fig. 6. Mechanical property changes of biocomposite films with metallic nanoparticles.

% of ZnO shows the highest PLA elongation at break and Young's modulus [257].

The incorporation of TiO₂ with PBAT and thermoplastic cassava starch (TPS) blended films was performed to enhance the mechanical property of the film. The results showed that small but YM and TS increased with the incorporation of TiO₂ [258]. MgO@CTAB nanoparticles dispersed (0, 1, 3, 5, 7 wt%) in biodegradable PBAT films, and the TS and EB (%) were increased up to optimal loading of the nanoparticle (3 wt%) but decreased as the loading rate increased, but Young's Modulus increased as the loading rate increases [64]. Multi-walled carbon nanotube-zinc oxide (MWCNT-ZnO) NCs were synthesized and dispersed in PBAT and its derivative. The MPBAT/MWCNT-ZnO nanocomposite film displayed the best mechanical performance at 0.2 % MWCNT-ZnO. (17.74 % increase in tensile strength, 22.17 % in yield strength, and 14.29 % in elongation at break) [244]. The tensile strength and elongation of Cu NPs incorporated PBAT film increase with 1 % load NPs but not in the elastic range [245]. Mechanical properties of Starch/PBAT/AgNPs@SiO₂ (SPA) films can be summarized as TS and EAB values increasing (2 wt%) and then decreasing (> 2 wt%) with increasing AgNPs@SiO₂ loading [246].

The results for E, σ_b , and ϵ_b for PBAT/SiO₂-EO initially grow and then plateau, but the highest comprehensive attributes occurred for 0.6 vol%, encompassing stiffness, strength, and elongation at break values of 59 MPa, 35 MPa, and 675 %, respectively [259]. With 5 wt% incorporation of ZnO/Rgo nanocomposites into the PBAT film, the TS and elastic modulus increase from 7.65 MPa and 5.31 MPa (neat PBAT film) to 27.43 MPa and 9.36 MPa, respectively [260]. A significant enhancement in the mechanical properties of PBAT film was found at the loading of 3 wt% MgO/Ag. As the content of NPs increases, it tends to form big agglomerations in the composite film [247]. Because of its hydrophilic character, the chitosan film exhibited the lowest tensile strength of 8.29 MPa compared to the other produced films. The tensile strength of CCZ2 with 5 % (w/w) ZnO was approximately 60 % greater than that of CCZ1 [261]. The addition of NiO-NPs influenced the mechanical characteristics of chitosan film significantly. This resulted in an increase in tensile strength, Young's modulus, and a decrease in elongation at a break of up to 6 % [29].

To investigate the impact of metal oxide (GO, MgO, TiO₂, and ZnO) nanoparticles on the mechanical properties of chitosan film, metal oxides were incorporated into the chitosan film. GO (0.1–5 %) exhibits enhanced TS. The TS and elastic moduli of 5 % MgO incorporated

chitosan film is 86 % higher and 38 % compared to neat chitosan films, respectively. After introducing 30 % TiO₂, the tensile strength of the CS-glycerol films increased 5.83 times. Furthermore, the elastic modulus of these chitosan-glycerol-TiO₂ nanocomposite films was 11.76 times greater than that of the chitosan-glycerol films. With loading concentration (0.5–4 %) of ZnO elastic moduli of bio-based films was improved [262]. Rodrigues et al. [263] reported the mechanical effect of ZnO-NPs on chitosan film, resulting in enhanced elongation, Young's modulus, and tensile strength that 8 wt% of NPs. Whereas chitosan film containing TiO₂ shows high Young's modulus (348.13 MPa), tensile strength (TS) (20.76 MPa), and elongation at break (% EB) (37 MPa) compared to neat chitosan film [264]. The effect of different NPs on the mechanical property of bio-based films is displayed in Table 5.

4.3. Thermal stability

The capacity of a polymeric substance to withstand the effect of heat is characterized by its thermal stability. The polymer structure, kind of covalent or noncovalent bonds, and degree of unsaturation are all factors to consider when dealing with biopolymers' thermal stability [265]. Bringing out multifunctional biopolymer products to enhance thermal stability is as important as producing biopolymer products [266]. Recently, using NPs to enhance the thermal resistance of polymers is becoming preferable due to the significant impact of the metal and metal oxide NPs. The following research reports address how the NPs incorporate with the polymers and how they affect the thermal stability of the polymers. The thermal stability of biocomposite films could easily be altered by incorporating thermally active metallic NPs. Often, the metallic NPs lower the thermal stability of composite films by increasing their thermal conductivity and decreasing their resistivity to thermal factors. The effect of incorporated metallic NPs on the thermal stability of biocomposite films is depicted in Fig. 7.

Leaf-extracted cellulose (LECF) and green synthesized bimetallic nanoparticles, namely Ag and Cu, were mixed to prepare nanocomposite films, and their thermal stability was analyzed. The thermal stability drastically decreased from 50 to 100 °C as the moisture was released from the film, and then no variations were found up to 350 °C for all films prepared with different concentrations of bimetallic NPs. The neat LECF exhibited different thermal properties after 350 °C, but NPs dispersed films showed different thermal stability around 300 to 350 °C due to thermal stability brought by the nanoparticles [30].

Table 5
Effect of different NPs on the mechanical property of bio-based films.

Polymer and incorporated NPs	Mechanical properties			Reference
	TS	EAB	YM	
Cellulose and Ag/Cu	• Started to decline as the concentration of NPs was high	• Increased at high concentration of NPs	• NA	[30]
Chitosan-starch blend and ZnO	• Increased with 3 % incorporation of NPs	• Declined as the loading rate of NPs increased	• NA	[79]
PLA with Cu	• NA	• Lowered by 15 % compared with neat PLA	• No change	[234]
Chitosan and TiO ₂	• Increased	• Increased	• Increased	[264]

NA – not available.

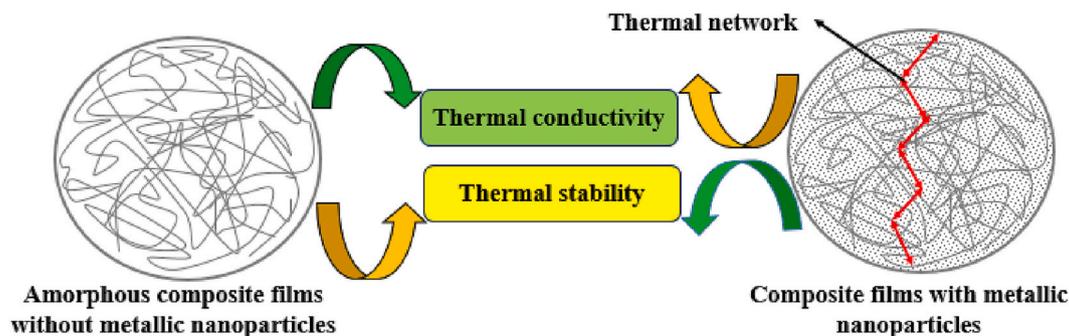


Fig. 7. Effect of incorporated metallic NPs on the thermal stability of biocomposite films.

Priyadarshi et al. [28] studied CMC films' thermal stability, which resulted in a two-step weight reduction strategy: 1) moisture removal and 2) decomposition of glycerol and CMC polymer matrix. Adding ZnO-NPs does not substantially differ from the CMC-based films' thermal degradation pattern compared to bacterial cellulose films. Cu-MOF nanoparticles dispersed cellulose acetate (CA) film's thermal properties were analyzed. As the concentration of Cu-MOF increases, the onset degradation temperature (T_{onset}) declines. At the same time, the maximum degradation temperature (T_{max}) was not much affected. Compared to the neat CMC film, the maximum decomposition temperature ($T_{0.5}$) of CMC/ZnO film decreased slightly. However, there was no significant change in T_{onset} [267].

In the case of biofilms prepared using the composite of Ag-NPs and sugar palm starch, the degradation temperature rose as the quantities of Ag-NPs increased because of their (Ag-NPs) excellent heat-storage capacity [268]. Zhu et al. (2021) [269] studied how ZnO-NPs and SiO₂-NPs alter the thermal stability of the films. The T_{max} observed for starch-ZnO, starch-SiO₂, and starch-ZnO-SiO₂ films were 322, 318, and 322 °C, respectively. Here, the ZnO-NPs dispersed starch film showed maximum thermal stability than SiO₂-NPs. However, both NPs exhibited higher thermal stability when compared to granules of starch (314 °C) and the neat film (318 °C). The pure starch–chitosan films presented a typical endotherm from 50 to 150 °C, characteristic of water loss. The dispersion of the TiO₂-NPs into the starch–chitosan film does not interfere with the thermal behavior of the composite films, according to Vallejo-Montesinos et al. (2020) [270].

In the temperature range of 300 to 382 °C, neat PLA film lost 96 % of its weight, corresponding to removing the ester group. When ZnO-NPs dispersed, the T_{onset} , T_{50} , and T_{endset} values of PLA films showed a decrement pattern [238,271]. However, TiO₂-NPs dispersed film showed a higher decomposition temperature when compared to pristine PLA film. Still, there was no significant change with adding more concentrations of TiO₂-NPs [239]. Yu et al. (2021) [123] reported that neat PLA's T_{max} was 379 °C but slightly increased when it was incorporated with acetylated cellulose nanocrystals. However, ternary composite film T_{max} values decreased and shifted further with a high concentration of ZnO NPs. The addition of TiO₂ to PLA provides no change in the T_{onset} , but alters the T_{max} , while the Ag-NPs dispersion also decreases the thermal resistance of the film [272].

The decomposition curves of MgO@CTAB with PBAT shift to lower

temperatures with the increasing loading of MgO@CTAB [64]. Zhang et al. (2021) [224] also stated that the initial breakdown temperature was also reduced from 377 to 322 °C due to the catalytic effects of MgO on PBAT matrix. In contrast, the weight loss of PBAT showed a decrement pattern from 420 to 560 °C, increasing MgO and Ag nanoparticle content in the ternary nanocomposite. This might be the synergetic effect of scattered nanoparticles in PBAT films. At 421 °C, the neat PBAT lost 1 % of its weight, while PBAT/Cu film presented a weight loss of around 3 %. The final thermal decomposition was 98.9 %, 97.5 %, 95.4 %, and 96.8 %, respectively, to pure PBAT matrix, nanocomposites of PBAT/Cu with the loading of Cu-NPs 1, 3, and 5 % at the temperature range 420–427 °C [245]. It can be noticed that the thermal decomposition percentage was decreased than the neat PBAT film when the composite was formulated with 3 % Cu-NPs.

When 6 % NiO-NPs were incorporated into chitosan film formulation, the T_{max} increased to 257 °C from 232 °C, i.e., for neat chitosan. It shows the enhanced intermolecular interactions between chitosan chains and NiO-NPs [29]. Zahiri et al. (2021) [273] performed the thermal stability analysis of ZnO-NZnO-NPs-loaded chitosan films. The result showed that at a lower concentration of ZnO-NPs the film's thermal stability was improved. Still, as the ZnO-NPs concentration increased, the thermal stability of the film started to decline. It could be concluded from the results discussed that integrating metal nanoparticles into biopolymer films might result in the increment or decrement pattern of thermal stability according to the nature of polymers and their interactions with the nanoparticles. The thermal stability of biopolymers could be adjusted during the processing based on their applications. However, developing thermally stable bio-polymeric films is directly related to creating process-able films with good enhanced properties. The effect of different NPs on thermal stability of bio-based films is presented in Table 6.

4.4. Barrier properties

Barrier properties include permeability of gases, optical barrier properties, and water vapor permeability, i.e., related to the film materials' qualities, such as the component chemicals' hydrophilicity or hydrophobicity [274–276]. To avoid degradation, food packaging materials must have low permeability to moisture or gas molecules. The water barrier properties of films can be assessed by measuring water

Table 6
Effect of different NPs on the thermal stability of bio-based films.

Polymer and incorporated NPs	Thermal stability	Reference
Chitosan and NiO	• T_{max} increased by 25 °C	[29]
PLA and ZnO	• T_{max} shifted to lower temperatures	[123]
Starch and Ag	• Degradation temperature increased at a high loading rate of NPs	[268]
PBAT and MgO	• Decomposition curves shifted to lower temperatures	[64]

vapor permeability (WVP), moisture absorption, and swelling ratio. The effect of active metallic NPs on the barrier properties of polymeric films essentially depends on the dispersion, size, and type of NPs. The hindering and blocking the activity of the films that are raised from the structural changes resulted from the incorporation of metallic NPs. The NPs involve lowering the movement of water and air molecules into the structure of the films and blocking the penetration of UV-light. The Effect of incorporated metallic NPs on the barrier properties of bio-composite films is illustrated in Fig. 8.

With GO-NPs loading, the permeability coefficient of O_2 improved dramatically, successfully preventing O_2 penetration into the cellulose nanocomposite films [277]. The produced alumina nanocomposites improved the moisture resistance of the CMC-based film. As a consequence, the nanoparticle-based films that were produced demonstrated strong barrier characteristics against oxygen infiltration [278]. Uncoated paper's WVP was determined to be 11.7410×10^{-10} g/m² Pa. Compared to uncoated paper, the WVP of CMC/CNC@Ag-NPs coated paper was reduced by approximately 32 %. It could be further enhanced by increasing CMC/CNC@Ag-NPs content. Similarly, the air permeability of paper was decreased from 1363 L/min m², i.e., for uncoated CMC paper when the films contained CNC@AgNPs, as reported by He et al. (2021) [279]. The clean CMC film had a WVP of 2.04–9 g/m² Pa. Adding ZnO-NPs to the CMC films lowered the WVP and water contact angle considerably [28]. The WVP of presiten cellulose acetate films is 8.86 g/h.Pa, however, lowers WVP considerably to 5.82 g/h.Pa for CA/7 %

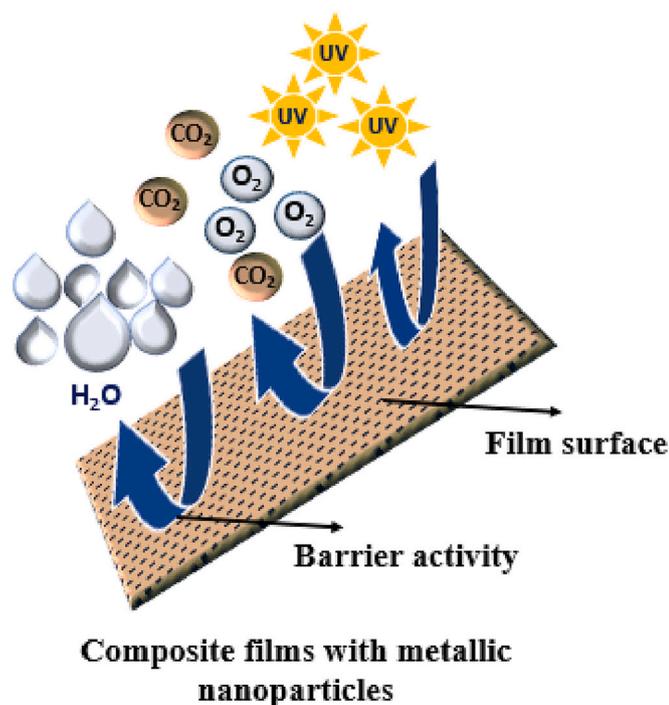


Fig. 8. Effect of incorporated metallic NPs on barrier properties of bio-composites films.

Cu-MOF films [251].

Semi-refined iota carrageenan's WVP was reduced as more SiO_2 and ZnO-NPs were added. However, compared to films with only one kind of nanoparticle, films containing a mix of SiO_2 -ZnO nanoparticles had a lower WVP value [280]. Roufegarnejad et al. (2022) [281] reported that the WVP of neat SPI film was 49.11×10^{-10} g/s.Pa and it significantly decreased by incorporating TiO_2 and CuO-NPs by increasing their concentrations. A similar outcome was seen by Lee et al. (2021) [282] for chicken skin gelatin tapioca starch composite film prepared with ZnO-NPs. The WVP of the gelatin film was decreased from 3.94×10^{-9} to 3.36×10^{-9} g/m²Pa after reinforcing the gelatin matrix with magnetic iron oxide nanoparticles from 0 to 10 % [283].

The permeability of the alginate film is 1.48×10^{-9} g/m².Pa, following the integration of sulfur nanoparticles, a substantial decrease in WVP values was found for the resultant nanocomposite films up to a concentration of 2 wt% nanoparticles. The WCA of the clean alginate film, on the other hand, was 56.14°, which increased when sulfur nanoparticles were added [28]. The WVP and WCA were used to determine the water vapor barrier and surface wettability characteristics of the alginate-based films, which resulted in decreasing and increasing trends, respectively, as the CuS-NPs were incorporated with the film [267]. As the fraction of ZnO nanoparticles increased, the water absorption capability of the Soy protein-based film decreased. As a result, they lost their superabsorbent properties [284]. The WCA of the starch/alginate films, i.e., control films, was 22.35°, which has a high level of hydrophilicity. The addition of g-C₃N₄ or Cu5%-g-C₃N₄ increases the WCA to 47.26° and 49.86°, respectively. On the other hand, the WVP of the neat film changed from 7.5×10^{-13} to 4.13×10^{-13} and 3.05×10^{-13} kg/m².s.Pa, respectively when incorporated with g-C₃N₄ and Cu5%-g-C₃N₄ [285]. The PVA/S-TiO₂ film was found to have significantly higher moisture content (MC), while the PVA/S-ZnO film has low MC value than PVA/S-control [286].

Oxygen transmission rate (OTR) and WVP values of pure PLA were 69.42 μm·mL/m²·day·mbar and 1.14×10^{-11} g/m².Pa.s, respectively. The OTR and WVP values of the PLA/ACNC/ZnO ternary composite films declined further when the ZnO nanoparticle loading was increased to 5 % by weight [242]. PLA film reinforced with 9 % ZnO decreased WVP up to 40 % and OTR up to 33.5 %, while at 15 % ZnO loading decreased the WVP up to only 20 % and there was no change in OTR [287]. The PLA/PBS film had a low water absorption capacity due to a porous surface created when ZnO was incorporated into the film PLA/PBS/ZnO.1 showed the highest values in the swelling test. [288]. The pure PLA film's OTR was reduced from 1439.07 to 841.20 cc/m².day. Pure PLA WVP reduced from 8.24 to 3.96 g/m²/day, with a rising trend of NiO-NPs [256]. ZnO-NPs dispersed PLA film significantly enhances water solubility and swelling capacity compared to pure PLA film [224].

Oxygen permeability and WVP values altered with increasing Ag@SiO₂-NPs loading in starch/PBAT films. The minimum oxygen permeability and WVP values were observed at 2 wt% loading Ag@SiO₂-NPs [246]. But, the oxygen and CO₂ permeability increased with increasing TiO₂-NPs in PBAT-based film and reached maximum permeability at 3 % TiO₂-NPs [258]. The oxygen and WVP coefficients were lower in comparison to clean PBAT. TiO₂-NPs and PBAT/hybrid nanocomposite fillers had little effect [289]. The inclusion of TiO₂/Ag-NPs reduced the WVP and OTR. When compared to PBTA-0 film, the WVP and OTR of PBTA-5 film were lowered by roughly 37 % and 71 %, respectively [290].

The clean chitosan film had a WVP of 0.59 to 0.03 g/m².Pa.s, which reduced to 0.51 to 0.03 g/m². Pa.s following the addition of tannic acid. It was drastically lowered further to 0.46 to 0.03 g/m².Pa.s after integrating 1 wt% TiO₂ [291]. Water vapor transmission and permeability were investigated in TiO₂-dispersed chitosan (CS) film and decreased both barriers as the concentration of TiO₂ increased [292]. Neat chitosan film with $2.41 \pm 0.32 \times 10^{-6}$ g/m·s.Pa WVP, but the addition of NiO-NPs at high concentrations had a widespread effect on lowering WVP values [29]. Pristine chitosan film with oxygen permeability and

WVP of 0.18×10^{-16} mol m/m².s.Pa and 1.54×10^{-11} mol m/m².s.Pa was incorporated with ZnO-NPs. Incorporating ZnO-NPs negatively affects both properties [293]. Similarly, the presence of Ag-NPs increased the chitosan-based films' WVP, as per the report of Shankar et al. (2015) [228]. The oxygen permeability and WVT of chitosan-based films dispersed with reduced graphene oxide and Ag-NPs were smaller than that of CS, CS/rGO. In general, the barrier properties of the films are enhanced, which means that the WVP and OTR coefficients are minimized compared to neat film. Improving the barrier qualities of bio-based films is critical for keeping meals fresh, unpolluted, and nutritious for an extended period. The effect of different NPs on the barrier properties of bio-based films is showed in Table 7.

4.5. Antimicrobial activity

Naturally, the anti-microbial activity of bio-based films is the capacity of the specific biopolymers to act against microbial activities when used in different applications. In this context, biopolymer-based films have no significant antimicrobial activities except those from chitosan [295,296]. Therefore, incorporating organic or inorganic active components into the biopolymers is crucial for enhancing the application of biofilms with a broad range of antimicrobial properties. The anti-microbial activity of metallic NPs incorporated in polymeric films work in a way that the NPs involved in attacking the cell wall of microorganisms, which leads to microbial mortality. Besides, the charges of the NPs incorporated play a vital role, specifically for Gram-positive and Gram-negative bacterial species. The anti-microbial activity of incorporated nanoparticles in the composite films is illustrated in Fig. 9.

The cellulose nano crystals-based films exhibited the highest inhibition zone against *E. coli* RB and *S. aureus* when the Ag₂O than ZnO and TiO₂ were incorporated [297]. Roy et al. (2020) [250] studied the effect of ZnO and curcumin added to the CMC-based films against foodborne pathogenic bacterial species, such as *L. monocytogenes* and *E. coli*. The investigation revealed that ZnO-NPs showed good antibacterial activity, whereas curcumin exhibited a bacteriostatic effect for the studied pathogens. The cellulose/elastomeric polymer bitumen-based hybrid biofilms with Ag-NPs displayed more bactericidal activity for Gram(-) bacterial species than Gram(+) [298].

Madvoli et al. (2022) [299] observed that Ag-NPs@cellulose nanofibers composite films have an antagonistic effect on different bacteria, namely *P. mirabilis*, *B. subtilis*, *C. albicans*, *S. aureus*, *P. aeruginosa* and *E. coli*. However, more potency was measured against *B. subtilis*. About 96 % of the inhibition rate was determined for *S. aureus* and *E. coli*, while 51.2 % was observed for *C. albicans* by ZnO-NPs@Zn²⁺/Cel composite films under colony count test for six hours [300]. The structure of bacteria, such as *L. innocua* and *E. coli* was altered and also showed a high rate of antibacterial activity by CuO-NPs dispersed with cellulose-based biofilms [301]. The TiO₂-NPs and MWCNTs resulted in a maximum inhibition zone against *E. coli* than *C. albicans* and *S. aureus* when incorporated into cellulose-based films [302].

Table 7
Effect of different NPs on the barrier properties of bio-based films.

Polymer and incorporated NPs	Barrier properties	Reference
Starch/PBAT and Ag@SiO ₂	• Minimum oxygen permeability and WVP at 2 wt% loading of NPs	[246]
Chitosan and Ag	• WVP of the film increased	[294]
PLA and ZnO	• With 9 % ZnO decreased WVP up to 40 % and OTR up to 33.5 %	[287]
Gelatin and FeO ₃	• WVP of the gelatin film decreased from 3.9×10^{-9} to 3.4×10^{-9} g/m ² Pa	[283]
Cellulose and alumina NCs	• Strong barrier characteristics against oxygen infiltration	[278]

Peighambaroust et al. (2019) [303] investigated the individual and synergetic antibacterial effect of Ag, CuO and ZnO-NPs dispersed into starch-based films. According to the findings, the synergistic impact was four times greater for *S. aureus* and two times more for *E. coli* than the individual effect. Besides, Ag-NPs resulted in a maximum inhibitory effect for both bacterial species among the nanoparticles used. The antimicrobial activity of ZnO-chitosan nanoparticles dispersed starch film was analyzed. The film exhibited positive antibacterial activity against both gram-positive and gram-negative bacteria [292]. The agar diffusion method investigated the antimicrobial activity of ZnO-NPs dispersed starch films against *S. aureus* and *E. coli* was investigated by [304]. The film exhibited an inhibition zone of 5 ± 0.1 and 4 ± 0.1 mm against *S. aureus* and *E. coli*. Antibacterial activity of MgO dispersed starch-based films was 4–6 mm zone of inhibition against *E. coli* and *S. aureus* [305]. Gelatin/starch/ZnO-NPs film was tested against *S. aureus* and *E. coli*, resulting in 67.28 ± 17.28 and 85.30 ± 18 mm² zones of inhibition, respectively [306].

The antibacterial activity of Chitosan-based films incorporated with ZnO-NPs was studied against *S. aureus* and *E. coli*. The zone of inhibition increased from 0 to 22.85 ± 6.81 and 0 to 23.51 ± 3.12 mm for *S. aureus* and *E. coli*, respectively [125]. The NiO-NPs incorporated chitosan films inhibited the growth *S. aureus* and *S. typhimurium* [29]. The antimicrobial activity of Ag-NPs/PLA composite films observed was 31.7 and 35.6 mm for *S. aureus* and *C. albicans*, respectively [237]. The synergetic antimicrobial effect of ZnO/rGO on PBAT films against *E. coli* and *S. aureus* was analyzed. The measured maximum inhibition zone for *E. coli* was 19.2 ± 0.81 mm and for *S. aureus* was 20.0 ± 0.38 mm [260]. The broader the inhibition zone, the greater the antibacterial activity of the films. Generally, the antimicrobial activity of metal and metal oxide nanoparticles is related to how efficiently these particles inhibit the growth of different bacterial species. The microbicidal effect depends upon the size and shape of NPs, ions releasing ability of the NPs, the cell wall composition of the bacteria, and the interaction way between the films and the bacteria. Based on their response to the antimicrobial test, the nanoparticles incorporated in biofilms could be applied for bacteriostatic or bactericidal purposes. The effect of different NPs on the antimicrobial properties of bio-based films is presented in Table 8.

5. Constrains for developing metal nanoparticles dispersed biofilms

In recent decades, metal or metal oxide nanoparticles have been utilized in different research areas to achieve high-end applications. Using those nanomaterials as fillers to enhance the limited properties associated with biopolymers is unavoidable. Nowadays, it is a very attractive research area due to its positive impact on improving the properties of biofilms [307,308,309,310]. Although all their significant impact in enhancing the properties of biobased polymer-driven films, they exhibit some limitations. There are some limitations commonly existing for most nanomaterials. Also, some biofilm developmental constraints are associated with specific nanoparticles. A few common limitations include the migration effect [99,153], toxicity, and the incompatibility nature of nanomaterials [244]. The specific constraints possessed by particular nanoparticles include weak photocatalytic activity, limited to enhance the flexibility of composite films [140], poor processability, formation of aggregates [26] and toxicity to prokaryotic cells [303]. For instance, GO failed to enhance the glass transition and melting temperature of PLA film [235] and ZnO-NPs have limitations in encompassing reactive oxygen species and reactive nitrogen species, which are more hazardous [240]. Dealing with the above-mentioned limitations of different metal and metal oxide nanoparticles is crucial for improving their processability and producing compatible biofilms for various applications.

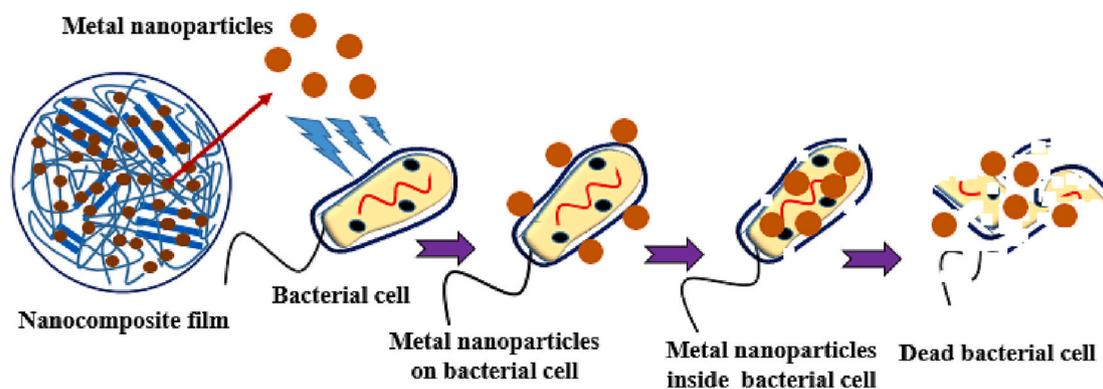


Fig. 9. Anti-microbial activity of incorporated nanoparticles in the composite films.

Table 8

Effect of different NPs on the anti-microbial properties of bio-based films.

Polymer and incorporated NPs	Anti-microbial properties	Reference
Cellulose and ZnO-NPs@Zn ²⁺	<ul style="list-style-type: none"> About 96 % of the inhibition rate was determined for <i>S. aureus</i> and <i>E. coli</i>, while 51 % was observed for <i>C. albicans</i> 	[300]
PBAT and ZnO/rGO	<ul style="list-style-type: none"> Maximum inhibition zone for <i>E. coli</i> was 19 mm and for <i>S. aureus</i> was 20 mm 	[260]
Starch and MgO	<ul style="list-style-type: none"> Zone of inhibition was 4–6 mm against <i>E. coli</i> and <i>S. aureus</i> 	[305]
PLA and Ag	<ul style="list-style-type: none"> Antimicrobial activity of films observed was 32 and 36 mm for <i>S. aureus</i> and <i>C. albicans</i> 	[237]
Gelatin/starch and ZnO	<ul style="list-style-type: none"> <i>S. aureus</i> and <i>E. coli</i>, resulting in 67.28 ± 17 and 85.30 ± 19 mm² zones of inhibition, respectively 	[306]

6. Future prospects of metal and metal oxide nanoparticles dispersed biofilms

Biopolymeric materials have been extensively used to manufacture various composite products for different environmental applications, such as dye removal [311,312], heavy metals detection and capturing [313–316], and nutrient recovery from wastewater [317–319]. Besides, several studies have been conducted based on using carbohydrate-based polymers in composite film preparation and enhancing their properties. They have provided magnificent results to the research communities in directing the options to extract, utilize, and recover carbohydrate polymers and improving their properties using different techniques. However, the research related to the incorporation of metal nanoparticles for biofilm preparation is yet to be discovered extensively because, for every particular research, there would be a new finding along with enhancing and making better biopolymer-based films. Several cutting-edge research opportunities could be created due to the potential application of metal and metal oxide nanoparticles to develop biofilms commercially. Some of the future prospects of these biofilms include,

- Processing as packaging material with enhanced barrier and anti-microbial properties
- Bringing biobased films to industrial-level processibility and applicability by enhancing their mechanical and thermal properties is essential with novel nanomaterials.
- Many researchers investigated the effect of individual nanoparticles with different carbohydrate polymers. To develop multifunctional biofilms economically and environmentally friendly for various applications, the synergetic impact of different nanomaterials could be performed with different biopolymer sources.

- Controlling the migration and toxicity of nanoparticles in biofilms is to be investigated to design suitable drug-carrying and delivering media, including typical packaging applications.
- Due to the abundance, biodegradability, and biocompatibility of biopolymers, the nanomaterials incorporated biofilms could be a promising option to replace fossil-based polymer products.

7. Conclusions

The effect of petro-based films emerged as a threat to the environment. To solve this emerging effect, biodegradable films have been investigated for their enormous potential to replace petroleum-based polymers. Even though these carbohydrate polymers exhibit good film-forming capacity, they have their limitations. One new trend evolving as the best way to tackle these drawbacks is using metal nanoparticles. The nanoparticles exhibited positive and negative enhancing impacts on the properties of carbohydrate polymers. Nonetheless, it can be concluded that employing various film manufacturing processes and nanoparticles to increase the capabilities of biopolymers is by far the greatest answer to these restrictions. Using NPs as fillers to enhance the properties of biofilms can be seen as a potential factor, yet these NPs also have limitations when dispersed into the films. Resolving the limitation of the NPs and carbohydrate polymers can open up broad future prospects related to biofilms. Thus, this review serves as a good source for producing bio-based films with enhanced properties using metal and metal oxide nanoparticles.

Declaration of competing interest

The authors declare that there is no conflict of interest.

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