

Review

Chitosan Nanocomposite Coatings for Food, Paints, and Water Treatment Applications

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Abstract: Worldwide, millions of tons of crustaceans are produced every year and consumed as protein-rich seafood. However, the shells of the crustaceans and other non-edible parts constituting about half of the body mass are usually discarded as waste. These discarded crustacean shells are a prominent source of polysaccharide (chitin) and protein. Chitosan is a de-acetylated form of chitin obtained from the crustacean waste that has attracted attention for applications in food, biomedical, and paint industries due to its characteristic properties, like solubility in weak acids, film-forming ability, pH-sensitivity, biodegradability, and biocompatibility. We present an overview of the application of chitosan in composite coatings for applications in food, paint, and water treatment. In the context of food industries, the main focus is on fabrication and application of chitosan-based composite films and coatings for prolonging the post-harvest life of fruits and vegetables, whereas anti-corrosion and self-healing properties are the main properties considered for antifouling applications in paints in this review.

Keywords: crustacean waste; chitosan; nanocomposites; films or coatings; antimicrobial activity; anti-corrosion; antifouling; food preservation; fruits and vegetables

1. Introduction

Millions of tons of crustaceans such as shells of crabs, shrimps, lobsters, and krill are produced each year and consumed as protein-rich seafood worldwide. The shells of the crustaceans and other non-edible parts that constitute about half of the body mass, and which are a prominent source of chitin and protein, are generally discarded as waste. Though chitin is the second largest natural polysaccharide on earth, after cellulose, it is not widely used for fabrication of products or as a food commodity due its insolubility in many commonly used solvents [1,2]. Chitosan (CH) can be obtained commercially from crustaceans' wastes and the cell walls of some fungi by the de-acetylation of chitin [3–5]. The United States Food and Drug Administration (USFDA) has recommended chitosan a GRAS (Generally Recognized as Safe) material, which has led to the application of chitosan in food, agriculture, and biomedicine.

Chitosan is a biopolymer, also known as “hydrocolloids”. Although most hydrocolloids are neutral or negatively charged at acidic pH, chitosan is charged positively due to the presence of highly reactive amino groups. Chitosan is a linear polysaccharide composed of N-acetyl, D-glucosamine,

and D-glucosamine units [6]. Chitosan is not extensively available in nature and is thus usually derived from chitin by the partial deacetylation in alkaline solutions at elevated temperatures. Chitosan has attracted attention for food and paint applications owing to its superior characteristic properties, such as degradability, solubility in weak acids, pH-sensitivity, film-forming property, biocompatibility, non-antigenic, absence of toxicity, and low-cost [7–9]. Moreover, because of its natural origin and multiple possible applications, like preparation of biodegradable films, blends, coatings, composites, nanocomposites, etc., it has attracted attention of both the scientific community and various industries, particularly the food and paint industries (Figure 1). This review mainly focuses on the physicochemical properties of chitosan, the development of composite materials, and its various applications in water treatment, paints, and food industries.

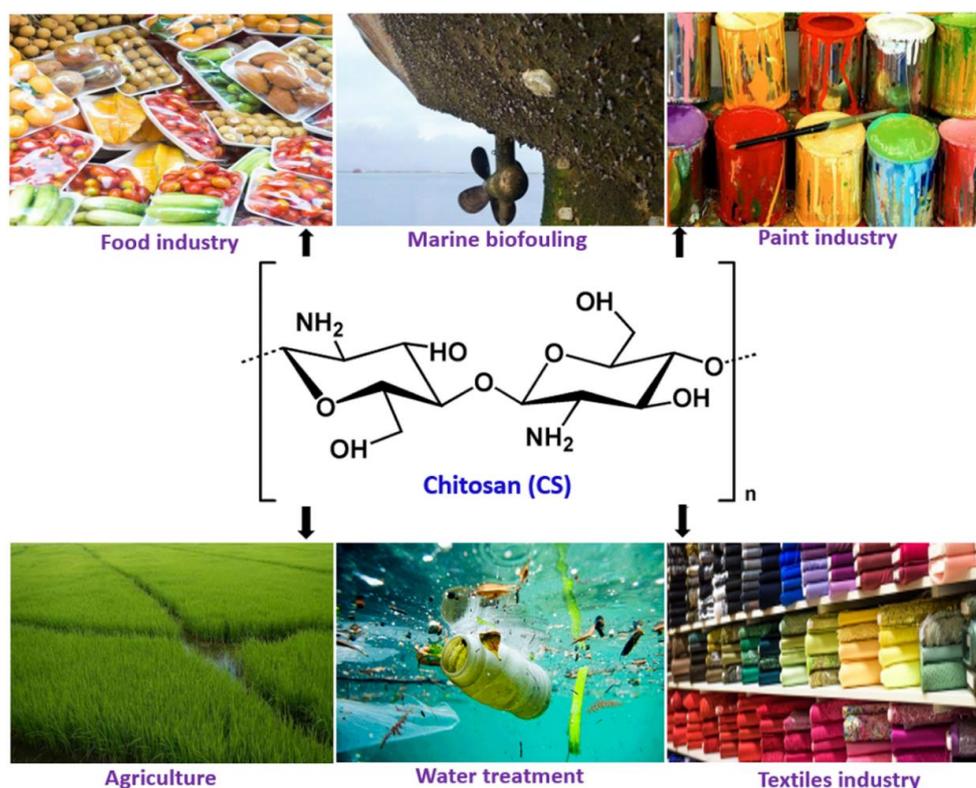


Figure 1. Wide ranging industrial applications of chitosan.

2. Chitosan and its Properties

2.1. Source and Extraction

Chitin is a linear homo-polysaccharide comprising of β-(1, 4)-linked N-acetyl-D-glucosamine units (Figure 2). Chitin is commonly present in invertebrates, such as crustacean shells or insect cuticles, as well as in the cell walls of fungi, some mushroom envelopes, green algae, and yeasts [10–12]. Abdulwadud et al. reported that crustacean shells usually contain 20–30% chitin, 30–40% proteins, 30–50% calcium carbonate/phosphate, and some pigments (astaxanthin, canthaxanthin, lutein, or β-carotene), which varies depending on the sources, or species of sources, and harvesting seasons [13]. Chitosan has been extracted from shrimp shells [14–17], fish scales of *Labeo rohita* [18], squid gladius (*Loligo vulgaris*) [19], locust waste [20], honey bees waste [21], fungus like *Aspergillus niger* [22], silkworm chrysalides [23], fishery waste [18], and blue crab (*Callinectes sapidus*) waste [24], amongst others.

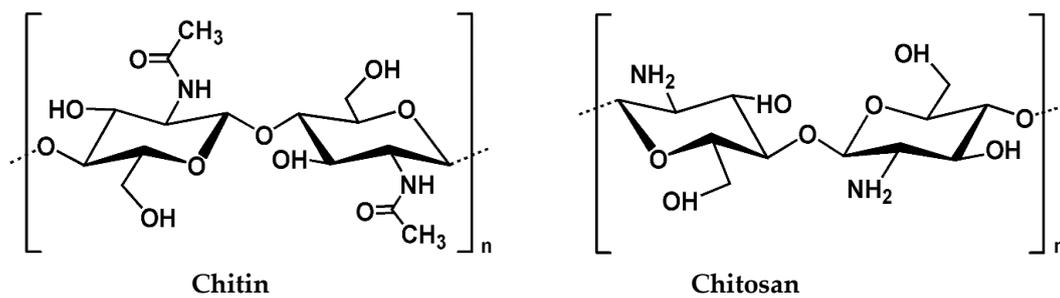


Figure 2. Chemical structures of chitin and chitosan.

Generally, chitin is commercially extracted from the exoskeleton of crustaceans (crab and shrimp shells) by acid treatments, followed by treatment with alkali to remove the calcium carbonates and proteins, respectively. The extraction process includes three major steps: demineralization, deproteinization, and depigmentation/discoloration (Figure 3). The demineralization step comprises of the elimination of calcium carbonate and calcium chloride, which are the primary inorganic compounds in a crustacean's exoskeleton. The digestion reaction is usually carried out in dilute hydrochloric acid (HCl) solution followed by filtration, washing, and drying. The emission of carbon dioxide (CO₂) gas is a significant indicator of the removal of mineral contents from the materials. In the second step, deproteinization is performed using an alkaline solution, such as dilute sodium hydroxide (NaOH), followed by filtration, washing, and drying, similar to the first step, as described above. Proteins that are extracted from crustacean waste shells during this process have found use in animal feed [25]. The final step, depigmentation/discoloration, is a purification process during which colour pigments such as astaxanthin and β -carotene are removed using various organic and inorganic solvents, such as sodium hypochlorite, acetone, and hydrogen peroxide, to obtain a purified chitin [26]. The most common process for deacetylation of chitin is the treatment with concentrated sodium or potassium hydroxide solutions at elevated temperatures. The acetyl (-C₂H₃O) group is removed from the polymer chain of chitin resulting in the formation of an amino (-NH₂) group, thus leading to the *N*-acetyl-glucosamine and *D*-glucosamine copolymer.

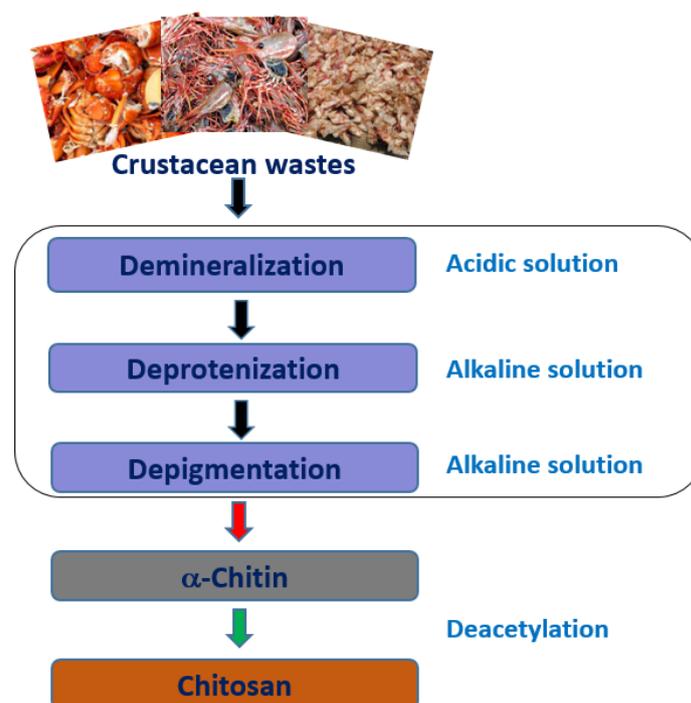


Figure 3. Preparation of chitosan from crustacean wastes.

2.2. Physico-Chemical Properties of Chitosan

2.2.1. Degree of Deacetylation (DD)

The degree of deacetylation is one of the most significant chemical characteristics of chitosan that determines the content of free amino groups ($-\text{NH}_2$) that is formed by the partial replacement of acetyl groups ($-\text{C}_2\text{H}_3\text{O}$) resulting in a copolymer of *N*-acetyl-glucosamine and *D*-glucosamine. Copolymers formed containing higher than 50% *D*-glucosamine units are typically considered as chitosan, whereas copolymers with more than 50% *N*-acetyl-glucosamine units are referred to as chitin. For chitosan, the percentage of *D*-glucosamine units are termed as the degree of deacetylation (DD), whereas for chitin, the percentage of *N*-acetyl-glucosamine units are known as the degree of acetylation (DA). The degree of deacetylation (DD) of chitosan (ratio of *D*-glucosamine to the sum of *D*-glucosamine and *N*-acetyl *D*-glucosamine) provides an indication of the number of amino groups in the polymer chains (e.g., *D*-glucosamine residues of 70% in deacetylated chitin corresponds to a deacetylation degree of 70% and acetylation degree of 30%).

The deacetylation of chitin begins in the amorphous regions prior to the crystalline regions, which occur by hydrolysis through chemical and biological (enzymatic) processes. Chemical treatment methods generally involve acidic or alkaline treatments under a nitrogen environment, or by the addition of sodium borohydride to NaOH solutions, to avoid any undesirable side reaction. Enzyme hydrolysis is environmentally friendly but is comparatively more expensive and thus primarily limited to laboratory-scale experiments [27]. Researchers determine the DD of chitosan using acid-base titration [28], potentiometric titration [29], conductometric titration [30], $^1\text{H-NMR}$ spectroscopy [31,32], elemental analysis [30], Fourier transform infrared (FTIR) spectroscopy [33], UV spectrophotometric analysis [34], capillary zone electrophoresis [35], and Raman spectroscopy [36].

DD indicates the amount of amino groups in chitosan polymer, which will affect the properties of chitosan, such as charge, density, solubility, crystallinity, degradation behaviour, mechanical, barrier, and thermal properties [37–39]. An increased percentage of amino groups in chitosan polymer makes it soluble in weak acids, a characteristic difference from chitin. The amino groups in chitosan polymer, which is highly reactive, contribute towards its versatility for utilization in various industrial applications. Recently, Zhuang et al. investigated the effect of deacetylation degree on mechanical and barrier properties of chitosan films with three different DD values, 81.0%, 88.1% and 95.2%, wherein CH films with higher DD values (88.1% and 95.2%) were found to have better water barrier property and tensile strength compared to films obtained with a chitosan value of 81.0% DD [37]. It has been reported that the antimicrobial efficiency also improves with the increase in the degree of deacetylation of chitosan that is generally attributed to the increase in the number of positive charges from the amine groups [39].

2.2.2. Molecular Weight (MW)

The molecular weight (MW) of chitosan also influences the physicochemical and antimicrobial properties. Chitosan are categorised in three different forms: high molecular weight (HM-CH) chitosan, low molecular weight (LM-CH) chitosan, and oligochitosan (O-CH, short-chained chitosan) [40]. Jongsri et al. reported the effect of molecular weights on the coating ability of chitosan and the postharvest quality of mango fruit by using three different molecular weight chitosan, namely, high molecular weight chitosan (HM-CH: 360 kDa), medium molecular weight chitosan (MM-CH: 270 kDa), and low molecular weight chitosan (LM-CH: 40 kDa) [41]. HM-CH coatings were found to be effective in delaying the ripening of mango fruit by retaining the titratable acidity, fruit firmness, and slowing down the rate of weight loss, ethylene production, and respiration. Additionally, HM-CH-coated fruits exhibited no incidences of spoilage throughout the reported storage period of 16 days. More recently, Zhong et al. evaluated the effect of MW on film-forming ability, electrostatic spraying atomization performances, and other film characteristics. Chitosan films were prepared with different molecular weights of chitosan (MW 6.55 kDa, 12.93 kDa and 47.70 kDa) using the electrostatic spraying (ES) technique [42]. The results indicated that with an increase in the MW of

chitosan, some film-forming solution properties, such as conductivity, viscosity, surface tension, and contact angle, were raised due to the increase in the proportion of amine-groups and degrees of CH chain entanglements. Moreover, with the increase in MW, the water barrier property and tensile strength of CH films were also found to improve. However, the antibacterial capacities of chitosan films against *Escherichia coli* and *Listeria innocua* were inferior for higher MW chitosan-based coatings [42]. High molecular weight chitosan cannot pass through the bacterial membrane and hence, they stack on the cell surface, which may alter the membrane permeability that effects the transport of nutrients into the microbial cell membrane, resulting in cell lysis, whereas chitosan with a lower molecular weight can proactively penetrate into the nuclei of a microorganism and could bind with DNA, inhibiting synthesis of mRNA and resulting in cell death [43,44]. No et al. however, reported that the antimicrobial activity of chitosan was greater with lower molecular weight for Gram-negative bacteria, but not for Gram-positive bacteria, which is still an area of contention amongst researchers [45].

2.2.3. Solubility

Chitosan is a semi-crystalline polymer due to the strong inter- and intra-molecular hydrogen bonds. Solubility plays a critical role in various applications of chitosan as it is readily soluble in dilute acidic solutions at pH 6.0, but insoluble in most organic solvents. The pKa value of primary amine groups of chitosan is ~6.3, and thus, under acidic conditions the amine groups are protonated, leading to the repulsion between positively charged macromolecular chains, which allows water molecules to diffuse in and solubilize the polymer. Chitosan precipitates from solutions at pH 6.0 or above, limiting the use of chitosan in basic conditions. Derivatives of chitosan such as acyl-chitosan [46], N-alkyl-chitosan [47], hydroxyalkyl-chitosan [48], PEG-chitosan [49], carboxymethyl chitosan acyl thiourea [50], and TEMPO-laccase oxidised chitosan [51] have been synthesized to improve the solubility in water over broader pH ranges. Water-soluble derivatives of chitosan have been reported to be effective in food, paints, and water treatment applications [52–54].

2.3. Antimicrobial Properties

Allan and Hadwiger first reported the antifungal properties of chitosan and suggested that chitosan does not only possess fungicidal properties, but is also more effective on a wider range of fungi than chitin [55]. Since then, several researchers have evaluated the antimicrobial nature of chitosan against different microorganisms and their action mechanisms, but no clear consensus on the mechanism of antimicrobial activity of chitosan has been reached [56–60]. Several mechanisms have been proposed to explain the antimicrobial properties of chitosan. These include: (i) interactions between the positively charged amine groups of chitosan and the negatively charged microbial cell membranes, leading to leakage of cellular constituents; (ii) activation of several defence mechanisms in the host tissue by chitosan molecules acting as a water-binding agent and hindering several enzymes by blocking their active centres; (iii) chitosan as a chelating agent, selectively binding metals and then preventing the microbial growth; (iv) chitosan (high-molecular-weight) forms an impervious polymeric layer on the cell surface that alters cell permeability and ultimately blocks the entry of nutrients into the cell; (v) penetration of chitosan into microbial cytosol that may bind with DNA, resulting in alterations for the synthesis of mRNA and proteins, mostly prevalent with low-molecular-weight chitosan; (vi) adsorption and flocculation of electro-negative materials in the cell, hampering the physiological properties of microorganisms, triggering cell death.

The most widely accepted mechanisms for antimicrobial activity of chitosan is the interaction between the positively charged amine groups of chitosan and the negatively charged microbial cell membranes. In acidic solutions (pH < 6.3) chitosan has a polycationic nature and the positively charged amino groups of chitosan interact with negatively charged components on microbial cell membranes, causing extensive alterations to the cell surface and membrane barrier properties, leading to leakage of intracellular contents that results in cell death. Tyagi et al. have demonstrated this hypothesis through a mechanistic study of chitosan nanoparticles against Gram-positive bacteria, *S. aureus* [56]. As chitosan

has pKa ~6.3, in mildly acidic conditions it is protonated, leading to a reduction in osmotic stability affecting membrane disruption that may efficiently lead to alterations in cell permeability and leakage of intracellular contents, ultimately imbibing rupture of cell. In addition, positively charged chitosan may interact with the negatively charged teichoic acids in the cell wall of gram-positive microbes leading to the formation of small pores on the cell wall and subsequently leading to leakage of the intracellular components [56]. Li et al. suggested that increased permeability of the outer membrane of *Escherichia coli* is the primary reason for antibacterial activity of CH against such Gram-negative bacteria. Increased permeability leads to the release of cellular contents followed by cell lysis, as shown through microstructural analysis using transmission electron microscopy (TEM) (Figure 4) [61].

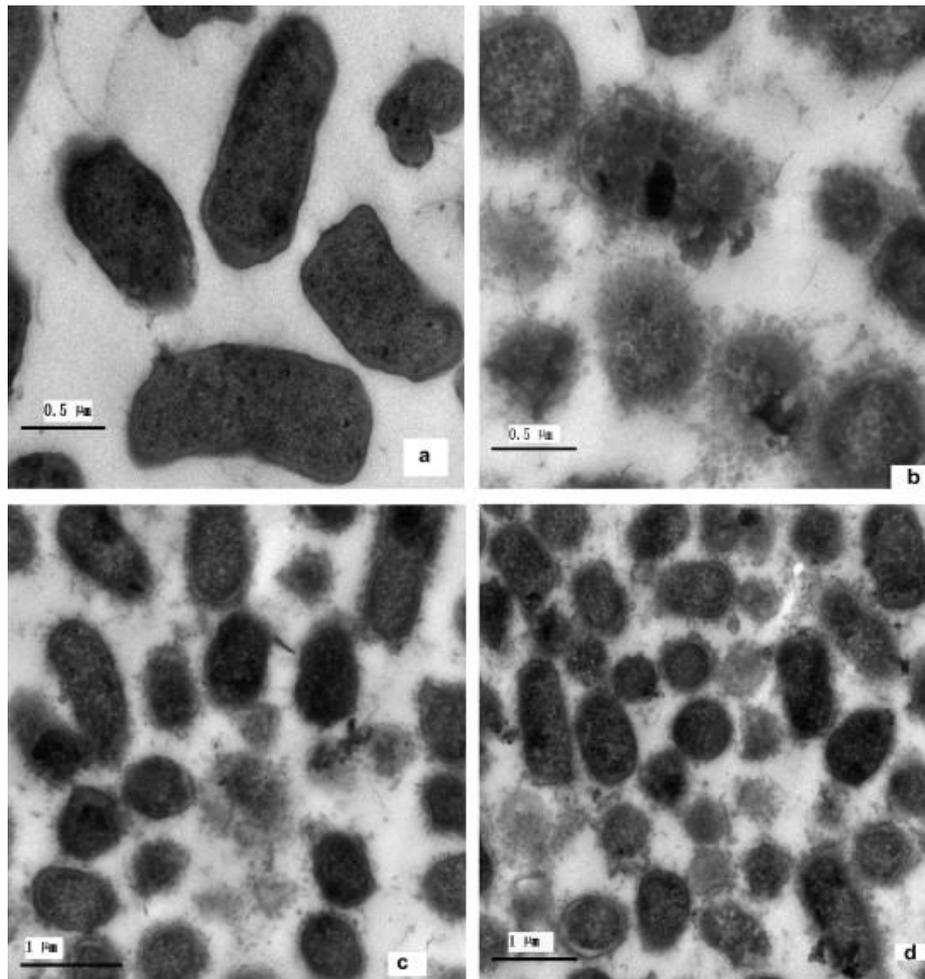


Figure 4. Transmission Electron Micrographs (TEM) of *E. coli* (a) treated with buffer and (b–d) chitosan of 50 kDa molecular weight, (Reproduced with permission from [61], Copyright © 2010, Elsevier).

2.4. Self-Healing Properties

The ability of a material to heal or repair damages automatically or with some external stimulation independently is called self-healing. Numerous polymeric materials with self-healing capabilities have been fabricated in recent years [62–65]. Self-healing materials, being capable of forming reversible bonds or reactions in the networks, enhance the durability of the materials. An example of chitosan-based self-healing materials that have attracted attention are anticorrosion self-healing paints [66–69]. Despite significant achievements in the development of chitosan-based self-healing materials, several challenges still need to be addressed for wider applications. An appropriate balance between mechanical strength, self-healing capacities, and mechanical robustness is required for the fabrication of newly developed, chitosan-based, self-healing materials.

Applications of self-healing coatings include automotive refinish on the backside of smartphones to stop the development of corrosion in scratches [70], etc. Due to excellent film-forming properties, superior adhesion to metallic surfaces, and self-healing abilities, chitosan-based self-healing coatings have been effective in protecting metal surfaces and metallic pieces [71,72]. Two main approaches have been pursued for corrosion protection in self-healing coatings: (i) the fixing of defects by adding polymerizing agents in polymeric coating matrix and (ii) by using corrosion inhibitors that can protect corroding areas [73]. In anti-corrosion paints, self-healing refers to both dynamic care of the substrate and structural repair of the coatings, offering superior protecting ability and increased longevity of the coating compared to other protective coatings [71]. Chitosan- and cerium (Ce)-based self-healing coatings have been reported to protect aluminium alloy 2024 from corrosion [68,74]. 2-Mercaptobenzothiazole (MBT) has been used as an effective corrosion inhibitor in chitosan-based coating for aluminium alloys 2024. The study revealed that MBT has strong inhibiting ability and even after one week in a full immersion condition no corrosion attack was reported [71]. In the study, the surface properties of the chitosan coatings were also improved by chemical grafting using poly (ethylene-alt-maleic anhydride) (PEMA) and poly (maleic anhydride-alt-1-octadecene) (PMAO) to increase its hydrophobicity, which is important for corrosion protection in atmospheric conditions. Due to good wettability and adhesion properties it was argued that chitosan provides corrosion protection, simultaneously working as a reservoir for the corrosion inhibitor which prevents the formation of pittings on aluminium alloy. The grafting of chitosan at the coating/solution interface with PEMA and PMAO provided an adequate hydrophobic effect, especially in the case of chitosan loaded with MBT, leading to the delay of ingress of electrolyte towards the metal interface. The combination of active corrosion protection due to MBT and the surface hydrophobicity conferred by grafting could be the reason behind the efficient protection from corrosion to aluminium alloy 2024 [71].

3. Chitosan-Based Nanocomposites

Previously, many studies have reported chemical modification of chitosan either by coupling with small molecules or grafting with polymers, for changing/improvement or better use of the intrinsic properties of chitosan. Chitosan has been grafted with different polymers, such as poly-lactide to form polymeric amphiphilic micelles [75] or polyethyleneimine (PEI) to form a branched PEI-g-chitosan with lowered cytotoxicity but higher gene transfection efficiency compared to PEI [76]. More recently, chitosan-based nanocomposites have emerged, where both polymer and nanoparticles contribute to the improvement or enhancement of specific properties. The dispersed nanomaterials contained in chitosan polymer matrix not only improve the physical, mechanical, and thermal stability of chitosan, but also endows the composite with its intrinsic properties, such as high surface area or extraordinary physicochemical properties. Chitosan nanocomposites formed between chitosan and metal/metal oxide, carbon, polymer, or clay materials via physical or chemical interaction and their applications in food, paints, and environmental fields are summarized in Table 1 and Table 2.

3.1. Chitosan-Metal/Metal Oxide

Noble metal, such as silver, are very effective antimicrobial agents [77–79] and can be synthesized in situ in a chitosan matrix due to the metal ion chelation capability of chitosan. Taking into account that silver nanoparticles are well-known for their antimicrobial property, chitosan-silver nanocomposite coatings materials have been studied on metal, glass, wood, etc., surfaces [80–82]. Metal oxide nanomaterials are commonly applied in polymer matrix as filler materials to enhance the antimicrobial and mechanical properties of the films and coatings, which are mainly used for food packaging and food preservation applications [83–86]. Silica nanoparticles in chitosan were reported to selectively absorb rare earth elements [87], while zinc oxide nanoparticles were reported to photo-catalytically generate reactive oxygen species (ROS), leading to high anti-diatom and anti-bacterial activities under sunlight [85,86].

3.2. Chitosan-Carbon Materials

Carbon-based nanomaterials, such as CNT, graphene, and graphene oxide have been well-studied. Incorporation of graphene oxide into chitosan improves the mechanical properties of chitosan in addition to enhancing its antimicrobial and pollutant removal abilities [88,89]. Chitosan being a biocompatible polymer has been applied to improve the solubility of CNT and reduce its toxicity to facilitate practical applications (Table 1) [90].

3.3. Chitosan-Polymer Mixture or Copolymer

Chitosan is often introduced to other types of polymers to form mixture or copolymers in order to enhance the properties for specific applications (Table 1). Attributed to the chemical reactivity of primary amine groups in chitosan, polymers containing activated carboxyl groups can be covalently linked with chitosan, commonly employing carbodiimide crosslinking reagents to form an amide bond between amine and carboxyl groups [91,92]. For polymers containing abundant hydroxyl groups, such as cellulose, physical mixture/interaction with chitosan is often used for the preparation of chitosan-polymer hydrogels [93], simple Van der Waal interactions occur that do not change chemical structures. Alternatively, chemical linking of polymers containing hydroxyl groups to chitosan works by the activation of hydroxyl into imidazolyl carbamate intermediates, that react with primary amine groups in chitosan to form *N*-alkyl carbamate linkages [94]. Since chitosan is a polycationic molecule, it can easily interact with negatively charged electrolyte/polymers/particles through electrostatic interactions. For instance, a chitosan–nucleic acid nanoparticle complex can be formed by coacervation between the positively charged amine groups and negatively charged phosphate groups in the nucleotide. Similarly, alginic acid/alginate as a natural polysaccharide has a similar chemical structure to chitosan but contains carboxylic instead of amine groups. Due to the opposite charges, chitosan and alginate can easily form electrostatic complexes [95].

3.4. Chitosan-Clay Composites

Clay is finely grained soil that is mainly composed of metal oxides or hydroxides with traces of organic matters. Owing to its small particle size (ca. 1 μm) and excellent colloidal properties, clay has been widely used in different applications, such as for water purification, as an odour absorbent, and as a lubricant in construction industries. Because of the structural characteristics, clay nanotubes and platelets can be used to load active agent to improve the passive barrier performances of anticorrosive coating [96]. Due to the electrostatic interaction between chitosan and clay, the composites are normally combined through adsorption, gelation, or intercalation. In chitosan-clay nanocomposites, clay exhibits its characteristic properties, such as absorption of specific species [97,98] or hemostatic properties [99], while chitosan can provide a higher loading or the cross-linked chitosan network as a support or scaffold for clay (Table 1).

Table 1. Examples of chitosan-based nanocomposites and their applications.

Chitosan Molecular Weight/Viscosity	Type of Nanomaterials in Composite	Name of Nanomaterial/Polymer/Clay	Preparation Method of Chitosan Nanocomposite	Form of Chitosan Nanocomposites	Specific Application	Key/Enhanced Properties	Application Field	Reference
100 kDa	Metal	Ag nanoparticles	In situ reduction on chitosan	Thin film coating on bandage	Antibacterial activity against <i>E. coli</i> and <i>S. aureus</i>	Inactivation bacterial metabolism	Antimicrobial	[100]
Medium molecular weight	Metal	Ag nanoparticles	In situ reduction on chitosan	Ag nanoparticles anchored on chitosan particles	Sensing of ammonia in solution	Sensitive in optical absorption intensity and wavelength	Environment	[101]
Medium molecular weight	Metal oxide	ZnO nanoparticles	Blending	Thin film coating	Antifouling prevention	Anti-diatom activity and antibacterial activity against the marine bacterium	Anti-biofouling	[85,86]
Low viscosity	Metal oxide	SiO ₂ nanoparticles	In situ Stöber method grown on chitosan	Slurry packed in liquid chromatography (LC) column	Adsorption of rare-earth elements	High adsorption efficiency, selectivity, and reusability	Environmental	[87]
190–310 kDa	Carbon	Graphene oxide	Cross-linking	Thin film	Antimicrobial against <i>E. coli</i> and <i>B. subtilis</i>	Improved mechanical and antimicrobial properties	Antimicrobial	[88]
300 kDa	Carbon	Graphene oxide	Cross-linking	Hydrogel	Removal of dyes and metal ions from water	Tunable surface charge; efficient removal of pollutants	Environmental	[89]
N/A	Polymer	low density poly-ethylene (LDPE) film	Grafting	Coating	Significant changes in surface wettability	Improved anti-thrombogenic properties	Antifouling	[92]
N/A	Clay	Halloysite clay nanotubes	Electrostatical adsorption	Coating	Anticorrosive protective	Improved passive barrier protective and self-healing	Environmental	[96]
50–190 kDa	Clay	Bentonite and sepiolite	Blend	Thin film	Winemaking application	Enhanced immobilization of protease but negatively affected catalytic properties	Antimicrobial	[97]
Medium molecular weight	Clay	Bentonite	Gelation and lyophilization	Bead	Carbon dioxide adsorption	High adsorption capacity under moderate condition	Environmental	[98]

4. Applications of Chitosan-Based Nanocomposites

4.1. Water Purification

The use of synthetic dyes is increasing day-by-day for industrial applications, particularly in the textile industry, which leads to severe water pollution because of their discharge into water bodies without suitable treatments. Over 10,000 different colorants (dyes and pigments) are used in textile industries and over 7×10^5 tons of synthetic dyes are annually produced worldwide [102,103]. Most of the synthetic dyestuffs are discharged into the ecosystem without appropriate treatments, which has triggered global environmental problems [103]. Removal of dyes from water bodies is challenging because of their inert nature and existence in low concentrations. Chitosan has received attention in water purification as it is an inexpensive biopolymer and also due to the presence of a large number of reactive amino ($-\text{NH}_2$) and hydroxyl ($-\text{OH}$) groups. Adsorption of acid dyes with chitosan and modified chitosan occurs because of the electrostatic interaction between negatively charged dye ions and the protonated amino groups [104]. Shen et al. demonstrated that the removal of dyes from alkaline effluent involves chelating interactions rather than electrostatic interactions [105].

The removal of dye from wastewater using nanocomposites of chitosan has been achieved using several processes, such as physical adsorption, ion-exchange, hydrogen bonds, hydrophobic attractions, and chemical bonding. Chitosan-based composite fibres (MNPs/ZnPc-CS) and pellets of zinc photocatalysts (ZnPc)-supported metallic and bimetallic nanoparticles have been synthesized by Ali et al. for metal ions uptake [106]. The MNPs/ZnPc-CS fibres were used as dip-catalysts for the reduction of nitrophenols and azo dyes like methyl orange (MO) and cango red (CR). The results showed that the developed composites exhibited excellent catalytic efficiency and recyclability in the reduction of these dyes. Recent studies conducted by many researchers on the removal of dyes from wastewater using chitosan-based nanocomposites are summarized in Table 2.

Table 2. Chitosan-based nanocomposites for removal of dyes from wastewater.

Chitosan-Based Nanocomposite	Dye/Metal	pH	Removal Process	References
CS/AgNPs	Methyl orange	3–11	Photocatalytic decolourization	[107]
CS/AuNPs	4-nitrophenol	No data	Catalytic reduction	[108]
CS/TiO ₂	Rhodamine-B, Congo red	3–11	Photocatalytic degradation under visible light irradiation	[109]
CS/Polyaniline/CdS	Reactive Blue-19	6	Adsorption	[110]
CS/PVA/ZnO	Acid Black-1	No data	Adsorption	[111]
CS/SiO ₂ /CNT	Direct Blue-71, Reactive Blue-19	0–12	Adsorption	[112]
CS/lignin/titania	Brilliant Black	No data	Adsorption	[113]
CS/Bio-silica	Acid Red 88	1–12	Adsorption	[114]
CS/Palladium	4-Nitrophenol	No data	Catalytic hydrogenation	[115]
CS/SnO ₂	Methyl orange, Rhodamine-B	No data	Photocatalytic degradation	[116]
CS/Ag ₃ PO ₄ /CdS	Methyl orange	3–8	Photocatalytic decolourization	[117]

Table 2. Cont.

Chitosan-Based Nanocomposite	Dye/Metal	pH	Removal Process	References
CS/zirconium tungstate	Reactive Blue-21, Reactive Red-141, Rhodamine-6G and binary mixture; RB+RR, RR+RH, RB + RH	No data	Photocatalytic degradation	[118]
CS/SnO ₂ intercalated polyaniline (PANI)	Methylene blue Reactive yellow-15	No data	Photocatalytic degradation under direct sunlight irradiation	[119]
CS/TiO ₂ fibers supported zero-valent metal nanoparticles (like Cu ⁰ , Co ⁰ , Ag ⁰ and Ni ⁰)	Methyl orange, Congo red (CR), Methylene blue, Acridine orange, 4-nitrophenol, 2-nitrophenol, 3-nitrophenol, 2,6-dinitrophenol	No data	Catalytic reduction	[120]
CS coated cotton cloth supported zero-valent metal nanoparticles	Methyl orange, Methylene blue, 4-nitrophenol, Rhodamine-B	No data	Catalytic reduction	[121]
CS/MoO ₃ /TiO ₂	Methyl orange	No data	Photocatalytic degradation under solar light	[122]

Zhou et al. fabricated three low-cost polypyrrole-based composites, i.e., polypyrrole-chitosan-lignosulfonate (PPY-CS-LS), polypyrrole-chitosan (PPY-CS), and polypyrrole-lignosulfonate (PPY-LS) via polymerization (in-situ) of pyrrole monomers with chitosan (CS) and/or lignosulfonate (LS) as dispersants in order to improve the dye adsorption capacity of polypyrrole [123]. The selective adsorption properties of these composites were investigated for six dyes, i.e., methylene blue (MB), malachite green (MG), amaranth (AM), acid orange 10 (AO10), congo red (CR), and acid blue74 (AB74). The synergistic effects of amino/hydroxyl-containing functional groups from CS, sulfonic groups from LS, and nitrogen-containing functional groups from PPY chains resulted in high adsorption performance (feasible and spontaneous) for dyes with the PPY-CS-LS composite.

4.2. Antifouling Paints and Coatings

Marine biofouling is the undesirable growth of organisms on submerged surfaces [124]. Any submerged, clean, and unprotected substrata will be quickly colonized by microorganisms (bacteria, diatoms, and unicellular eukaryotes) and later by larvae of invertebrates and spores of algae [125,126]. Biofouling has huge economic impacts in maritime industries [127]. Biofouling significantly increases vessel drag, fuel consumption, clogs membranes and pipes, and also interfere with the stability of sensors attached to the vessels. Worldwide, countries spend billions of US dollars in order to manage and prevent this problem [128]. Currently, antifouling paints utilize toxic biocides that pollute the environment [127]. Thus, new low-cost and non-toxic antifouling paints are urgently needed. Chitosan has been proposed as a promising antifouling agent due to its antimicrobial properties [129] (Table 3). Plastic substrates coated with 2.5% chitosan significantly reduced the settlement of bryozoan *Bugula neritina* compared to untreated substrates. Chitosan mixed with a non-toxic paint base and applied to plastic substrates were found to protect the substrates from bacterial fouling over one week of laboratory experiments [130]. Chitosan incorporated into a silicon-polyurethane marine paint was exposed to biofouling for over two months in cold seawater [129]. Experiments demonstrated a short-term antibacterial action of chitosan, while no anti-algal action was observed. In contrast, chitosan paints significantly reduced densities of micro-fouling on the surface of a sea glider exposed to periodical oscillations of environmental conditions in the Sea of Oman. Moreover, it was found that

the structure of microbial communities formed on chitosan paints were similar to unprotected surfaces and were totally different from what is found on surfaces coated with copper-based antifouling paints, suggesting that chitosan-based coatings have minimal negative impact on marine communities [131].

Few researchers have combined chitosan with nanoparticles in order to increase antifouling properties and stability of coatings (Table 3). Chitosan-ZnO hybrid coatings were found to inhibit bacterial and diatom fouling more efficiently than chitosan coatings alone [86]. In another study, nanocomposite films of chitosan mixed with silver nanoparticles were found to be effective against *E. coli* and *Bacillus* bacteria and thus it was proposed that these “green” films can be used as packaging materials [132]. Nitric oxide (NO)-releasing chitosan coatings were found to exhibit dose-dependent behaviour for the degradation of biofilms of *Pseudomonas aeruginosa* [133]. The effect of NO-chitosan coating was better than chitosan alone, suggesting synergistic action of chitosan and NO. Chitosan functionalized with polyelectrolyte brushes were tested against bacteria in laboratory experiments and against biofouling in static field experiments in the Mediterranean sea, showing promising antifouling activities [134].

Chitosan has been applied to create membranes with antifouling properties (Table 3). Chitosan membranes had strong antibacterial properties [135] and growth of gram-positive bacteria was reduced more significantly than gram-negative bacteria. Cellulose membranes with smaller pore sizes were modified with higher molecular weight chitosan in order to improve their activity against gram-positive *Staphylococcus aureus* and gram-negative *E. coli* bacteria [136]. A forward osmosis (FO) membrane was fabricated using layer-by-layer assembly of chitosan and graphene oxide nanosheets on a porous support layer, which was found to be effective in fouling rejection of alginate in laboratory experiments [137]. Similarly, ultrathin antibacterial protective nanocoatings were prepared from ionic derivatives of chitosan using a layer-by-layer deposition methodology [138]. Silicone and glass surfaces coated with this nanocoating were able to prevent the adsorption of proteins and biofilm formation by *S. aureus*. The antifouling effect of chitosan nanocoating was found to depend on its thickness. Chitosan-modified polyacrylonitrile (PAN) hollow fibre membranes were reported to have better antibacterial properties against *E. coli* and *Bacillus subtilis* compared to unmodified PAN membranes [139]. Composite polyethersulfone (PES) nanofiltration membranes synthesized by blending *O*-carboxymethyl chitosan (*O*-CMC) and iron oxide (Fe₃O₄) nanoparticles were found to have considerably higher water flux, permeation, and fouling resistance compared to the unfilled PES membrane [140]. In a similar study, PES membranes prepared using *O*-CMC and silver nanoparticles were found to prevent protein fouling as well as having a good antibacterial property towards *E. coli* and *S. aureus* [141].

Table 3. Chitosan-based antifouling applications.

Application	Carrier/Additive	Type of the Study	Effective Against	References
Films	No	Laboratory	Bryozoan	[130]
Films	No	Field	Micro-fouling	[131]
Ultra-thin nanocoatings	No	Laboratory	Bacteria	[138]
Films	Polyelectrolyte brushes	Laboratory and field	Pathogens, micro-fouling and macro-fouling	[134]
Paints	Silicon-polyurethane	Field	Micro-fouling and macro-fouling	[129]
ZnO nanocomposites	ZnO nanoparticles	Laboratory	Micro-fouling	[86]
ZnO nanocomposites	ZnO nanoparticles	Laboratory	Pathogenic bacteria and fungi	[85]
Silver nanocomposite films	Silver nanoparticles	Laboratory	Pathogenic bacteria	[132]
Cellulose membranes	No	Laboratory	Pathogenic bacteria	[136]
PAN-chitosan membranes	No	Laboratory	Bacteria	[139]
Chitosan membranes	No	Laboratory	Pathogenic bacteria	[135]
Forward osmosis membranes	Graphene oxide nanosheets	Laboratory	Proteins	[137]
PES membranes	Fe ₃ O ₄ nanoparticles	Laboratory	Proteins	[140]
PES membranes	Silver nanoparticles	Laboratory	Proteins, bacteria	[141]

4.3. Shelf-Life Extension of Fruits and Vegetables

During postharvest transportation and storage, fresh produce (fruits and vegetables) undergo quality deteriorations due to the various physiology reactions and processes, such as postharvest respiration, ripening, ethylene production, and senescence. These physiological changes are directly influenced by the surrounding environment, i.e., temperature, oxygen, humidity, and light, that lead to loss of water, texture, colour, and nutrients of the produce [142,143]. Microbiological spoilage leads to postharvest losses of about 15% to 50% of fruits and vegetables produced worldwide [144]. In developing countries, the percentages of product losses are quite high due to a lack of appropriate technologies for postharvest storage of fruits and vegetables. The physiological, biochemical, and environmental changes promote the growth of postharvest pathogens that are the major causes of loss/damages in the supply chain. However, controlling postharvest destruction of fresh produce involves the application of synthetic antimicrobials, fungicides, and insecticides. Consumer concerns about the adverse effect of synthetic chemical residues on human health and environment, and the chances of the development of pathogen resistance, have led global scientific research towards the development of alternative strategies for the preservation of fresh produce [145].

The application of a natural polymer (biopolymer)-based films and coatings on food surfaces have recently gained interest for the shelf-life extension of fresh produce due to their similar functions to those of conventional protection or synthetic packaging. Out of several natural polymers, the use of chitosan-based treatment at the postharvest stages has been considered to be a suitable alternative treatment to replace the use of synthetic chemicals [146]. Chitosan-based films and coatings have been reported to effectively extend quality and storability of food products in general, and of fresh agricultural produce in particular, due to its excellent natural antioxidant, antibacterial, and antifungal activities [147–150].

4.3.1. Packaging Films

One of the main research areas in food industries has focused on developing new packaging techniques capable of improving the post-harvest life of fresh foods based on their interaction with packaging. Such techniques are well-known as “active packaging”. Active packaging can be defined as the incorporation of an active system/materials into packaging film or a container to maintain the quality or extend the shelf-life of food products. In particular, antimicrobial packaging is one of the most innovative and promising active packaging systems developed over the last decade, and these systems are capable of inhibiting microbial growth and action, leading to the maintenance of food quality with improved shelf-life [148,151,152].

Biopolymer-based packaging films have received considerable interest as an alternative packaging material to plastics. The bioplastics are usually degradable under appropriate conditions of moisture, temperature, and oxygen availability and do not produce any toxic residues. The problems associated with biodegradable polymers are three-fold (3P): performance, processing, and price. Problems due to “performance and processing” are universal to almost all biodegradable polymers irrespective of their origin [153]. In particular, brittleness, low heat-distortion temperature, high gas and vapour barrier properties, and poor resistance to harsh processing operations have strongly limited extensive applications of biopolymers. The application of nanotechnology has opened new options for improving properties of biopolymers. The incorporation of nanoscale materials as a filler into the biopolymer matrix have shown to markedly improve mechanical, thermal, barrier, and other physio-chemical properties, compared to the base polymers and conventional (microscale) composites [154]. Nano-sized fillers can be either inorganic or organic materials, such as clay (e.g., montmorillonite), natural antimicrobial agents (e.g., nisin), metal (e.g., silver, gold), and metal oxides (e.g., zinc oxide (ZnO), titanium dioxide (TiO₂)), to be the material of choice due to its antimicrobial activity, thermal stability and ability to improve mechanical properties of biopolymer films [147,149,153,155–157]. Chitosan-ZnO hybrid coatings on polyethylene films have been reported to reduce growth of pathogenic bacteria and fungi [85], as well as increase the shelf life of okra (*Abelmoschus esculentus*) vegetables [158]. Zhang et al. developed a chitosan-TiO₂ composite film for the packaging of red grapes and evaluated antimicrobial activity against food-borne pathogenic microbes, namely *E. coli*, *S. aureus*, *Candida albicans*, and *Aspergillus niger* [147]. Results showed that the chitosan-TiO₂ composite films were effective against all four tested microbes, with 100% sterilization within 12 h. The composite film could successfully protect red grapes from microbial infection thus enhancing their shelf-life (Figure 5). Recently, several other researchers have studied the fabrication, characterization, and application of chitosan-based nanocomposites films with antimicrobial properties for food packaging applications. A summary of recent work done on antimicrobial nanocomposite films and their applications in extending the shelf-life of fruits and vegetables are given in Table 4.



Figure 5. Preservation of red grapes wrapped with (a) polyethylene film, (b) pure chitosan film, and (c) chitosan-TiO₂ film, stored at 37 °C for six days. (Reproduced with permission from [147], Copyright © 2017, Elsevier).

Table 4. Chitosan-based antimicrobial films and their properties for the packaging of fruits and vegetables.

CS/CS-Composite Film	CS Concentration in FFS (% <i>, w/v</i>)	Nano-Filler Concentration (% <i> w/w</i> of Solid Matter)	Food Systems/ Applications	Antimicrobial Activity (Microorganism Tested)	Effect of Films	Thickness, Mechanical and Permeability Properties				References
						Thickness (μm)	TS (MPa)	EAB (%)	WVP or WVTR	
CS/TiO ₂	2.5	TiO ₂ -10	Red grapes	<i>E. coli</i> , <i>S. aureus</i> , <i>C. albicans</i> , <i>A. niger</i>	Red grapes were preserved for 22 days with composite film compared to 15 days with bare chitosan film before mildew occurred	70	46.33 ± 1.88	25.77 ± 2.91	No data	[147]
CS/TiO ₂	2.0	TiO ₂ -1.0	Tomato fruit	No data	Composite film delayed the ripening process of tomato fruits and loss in quality	No data	16.43 ± 0.46	53.06 ± 2.15	19.14 ± 1.13 (g ^m -2d ⁻¹)	[159]
CS/nano ZnO	2.0	Zinc acetate-100	No data	<i>E. coli</i> , <i>S. aureus</i>	Composite films showed good antimicrobial activity against both Gram-negative and Gram-positive bacteria	No data	41.73 ± 0.48	32.44 ± 0.52	22.53 ± 0.31 (g/h m ²)	[160]
CS/Gelatin/AgNPs	1.8	AgNPs-0.1	Red grapes	No data	Shelf-life of red grapes were extended by 14 days compared to control	87.60 ± 2.19	21.19 ± 1.01	27.23 ± 0.76	No data	[155]
CS/Bacterial cellulose nanocrystals (BCNC)/AgNPs	1.0	AgNPs-1.0	No data	<i>S. aureus</i> , <i>B. cereus</i> , <i>E. Coli</i> , <i>Ps. Aeruginosa</i> , <i>C. albicans</i>	Addition of BCNC and/or AgNPs in chitosan film improved its physical, mechanical, and antimicrobial properties	140	42.89 ± 1.76	22.50 ± 2.07	2.16 ± 0.06 (×10 ⁻¹⁰ g/smPa)	[151]
CS/Polyurethane (PU)/nano ZnO	CS/PU ratio 0.25:0.75	ZnONPs-1.0, 3.0 and 5.0	Carrot pieces	<i>E. coli</i> , <i>S. aureus</i>	Composite films efficiently enhanced the shelf-life of carrot pieces by 9 days compared to control	100	8.1	2.156	163.0 (g/m ² /day)	[161]
CS/Sulfur nanoparticles (SNP)	2.67	SNP-2.0	No data	<i>E. coli</i> , <i>L. monocytogenes</i>	Composite films showed high antibacterial activity against foodborne pathogens and could be potentially used in antimicrobial food packaging	55.6 ± 4.7	39.4 ± 4.4	8.3 ± 3.1	0.96 ± 0.17 (×10 ⁻⁹ g.m/m ² .Pa.s)	[148]

4.3.2. Coatings of Fruits and Vegetables

Coatings provide a thin layer of materials on food surfaces to maintain or control the ingress of gases, moisture, and solutes from the environment. Additionally, coatings can also act as a carrier for functional antimicrobial and antioxidant substances to additionally enhance its functionality for ensuring food quality and food safety of fresh produce (Figure 6). Coatings on the surface of fruits and vegetables could provide the following functions:

- i. Offer barrier properties against moisture and oxygen
- ii. Help to deliver antimicrobial activity to inhibit or delay the microbial growth
- iii. Deliver antioxidant effects that help to reduce the oxidation process, loss of colour, vitamins, etc.
- iv. Help to maintain the loss of volatile components and stop acquiring foreign odours

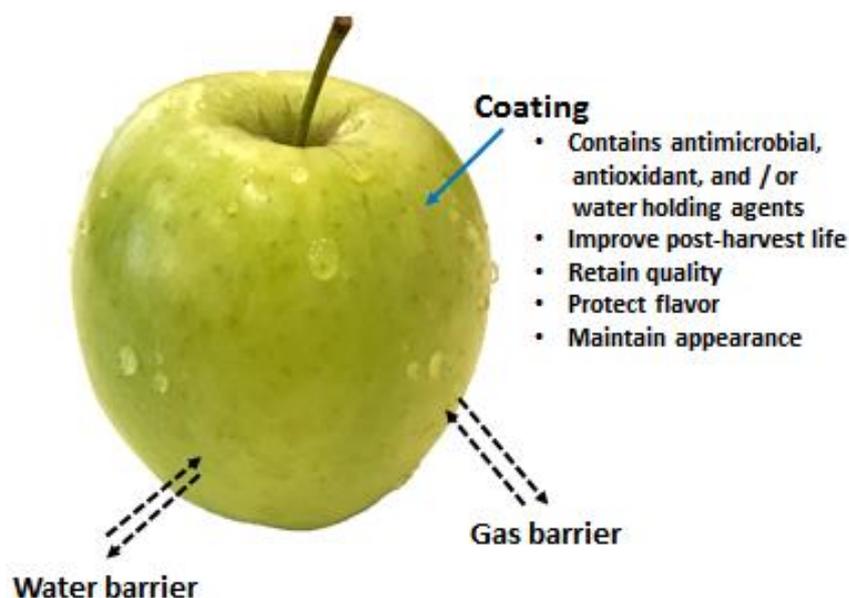


Figure 6. Illustration of coatings for improving the shelf-life of fresh produce.

Several researchers have reported the development or fabrication of chitosan-based nanocomposite coating formulations to improve post-harvest storage life by studying moisture loss, reduced respiration process, deliverability of antimicrobial and antioxidant agent, etc. A brief summary of the major coating formulations and their applications on fresh and minimally processed fruits and vegetables are given below in Table 5.

Table 5. Chitosan/chitosan-based composite coatings formulations for fruits and vegetables.

Chitosan Based Coating Formulations	Chitosan % (w/v)	Fruits and Vegetables Type	Effects of Coating	References
CS only	1.0–3.0	Rose or Pitaya fruit (<i>Hylocercus undatus</i>)	Coating containing 3% chitosan played the beneficial role for slowing down fruit withering, and maintained the fresh appearance and extended the maximal storability at ambient temperature	[162]
CS only	1.0	Litchi fruit	Coating effectively reduced the respiration rate as well as transpiration rate of litchi fruits during storage	[163]
CS only	1.0	Red kiwifruit (<i>Actinidia melanandra</i>)	Chitosan coating was found to enhance the shelf-life of kiwifruit	[164]
CS only	1.0–3.0	Mango fruit (<i>Mangifera indica</i>)	Coating delayed the climacteric peak, water loss, firmness, and ultimately prolonged the quality attributes of fruit	[150]
CS only	1.0–3.0	Guava fruit (<i>Psidium guajava</i>)	Coating effectively maintained the quality of guava fruits by increasing antioxidant value, and delaying ripening of fruits during storage at room temperature	[165]
CS only (Low, med, and high molecular weight)	1.0	Kiwifruit (<i>Actinidia kolomikta</i>)	High-molecular-weight chitosan showed more pronounced improvements on the shelf-life of kiwifruit	[166]
CS-nanoparticles	1.0	Table grapes	Edible coatings of chitosan nanoparticle efficiently delayed the ripening process and led to reduced weight loss, soluble solids, and sugar contents	[167]
CS coating with modified atmosphere packaging (MAP)	1.0	Pomegranate fruit	Chitosan coating with MAP treatment led to maintenance of visual quality and initial red aril colour intensity of fruits for up to 6 months under cold storage at 6 °C.	[168]
CS/Gum Arabic	1.0	Banana fruit	Coating delayed ripening by decreasing the rate of respiration of fruits and enhanced shelf-life for up to 33 days	[169]
CS/Ascorbic acid	1.0–2.0	Pomegranate arils	Shelf-life of arils could be extended to 21 days at 5 °C from 10 days for uncoated arils (control)	[170]
CS/g-Salicylic acid	1.0	Table grapes	Composite coatings on grapes improved the post-harvest life by decreasing the rate of respiration, decay incidence, and weight loss, and by maintaining/improving levels of total soluble solids, titratable acidity, and sensory attributes during storage	[171]
CS/beeswax	1.5–2.0	Mango fruit (<i>M. indica</i>)	Coating on fruits ultimately maintained firmness and improved shelf-life up to 3 weeks compared to control (uncoated)	[172]

Table 5. Cont.

Chitosan Based Coating Formulations	Chitosan % (w/v)	Fruits and Vegetables Type	Effects of Coating	References
CS/ PVP/ Salicylic acid (SA)	1.0	Guava fruit (<i>P. guajava</i>)	Fruits coated with CS/PVP-SA showed minimum loss in water and browning of skin that helped in maintaining fruit colour and firmness	[173]
CS/Alginate with Pomegranate peel extract (PPE)	1.0	Guava fruit (<i>P. guajava</i>)	Coatings proved efficient in maintaining the quality of guava for up to 20 days at low temperature storage	[174]
CS/CMC/ Moringa leaf extract	0.5–1.0	Avocado fruit (<i>Persea americana</i>)	Coating on fruits efficiently reduced the respiration rate, moisture loss, and firmness that ultimately helps in improving fruit quality and shelf-life	[175]
CS/PVA blended with ascorbic acid (AA)	N/A	'Superior seedless' grapes	Coating treatment significantly reduced water loss, shattering of berry, and delayed the colour change and titratable acidity	[176]
CS/Pullulan, CS/Linseed, CS/Nopal cactus, CS/Aloe mucilage	1.5	Fresh-cut pineapple (<i>Ananas comosus</i>)	Layer-by-layer edible coatings were beneficial to maintain the quality and extension of the shelf-life of cut pineapple	[177]
CS/MMT	1.5	Tangerine fruits	Coated fruit showed reduced decay rate, loss in weight, and improvements in total soluble solids and titratable acidity	[178]
CS/nano-silica	2.0	Longan fruit	Coating shown to improve the quality of longan fruits for the period of extended storage	[179]
CS-AgNPs	1.0	Fresh-cut melon	Coatings decreased the rate of respiration and rate of production of ethylene compared to uncoated samples and thus had better sensory quality up to 13 days at 5 °C	[84]

5. Conclusions and Future Perspectives

The present review reported on the current and most important topics regarding the development of chitosan-based nanocomposite coating formulation for food preservation, water treatment, and anti-corrosive and self-healing paint applications. Over the past several decades, chitosan has emerged as a promising biomaterial for various industrial applications including food, pharma, paint, cosmetic, and textile industries, due to their natural origin, structural similarity to glycosaminoglycan's (naturally presented in native tissues), biocompatibility, biodegradability, low toxicity, antimicrobial, and antifouling activities. Biopolymer-based edible films and coatings play a significant role in prolonging the post-harvest life and improving the quality of fresh fruits and vegetables. It protects the fresh fruits and vegetables from damages or deteriorations during storage after harvesting by reducing the rate of moisture and gas transfer, by delaying respiration, senescence, etc., and by maintaining structural integrity or firmness and sensory attributes. Technology advancement enabling the preparation of multifunctional chitosan-based coatings or films could be promising to be used as packaging materials for food applications. Chitosan-based coatings containing active anticorrosive agent with self-healing ability have great potential for application in paint industries due to its good film-forming properties, superior adhesion abilities to substrates, antifouling properties, and the ability to reversibly form complexes with potential corrosion inhibitors. Chitosan and its nanocomposites have been used for removing dyes from textile wastewater. It has also found application in the prevention of biofouling from submerged surfaces to membranes. More practical applications of chitosan nanocomposites with metal oxides and clay will emerge in the near future as the world is grappling to reduce synthetic polymer waste in our ecosystems.

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