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Review

Cold Plasma Technology in Food Packaging

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Abstract: Cold plasma (CP) is an effective strategy to alter the limitations of biopolymer materials for food packaging applications. Biopolymers such as polysaccharides and proteins are known to be sustainable materials with excellent film-forming properties. Bio-based films can be used as an alternative to traditional plastic packaging. There are limitations to biopolymer packaging materials such as hydrophobicity, poor barrier, and thermos-mechanical properties. For this reason, biopolymers must be modified to create a packaging material with the desired applicability. CP is an effective method to enhance the functionality and interfacial features of biopolymers. It etches the film surface allowing for better adhesion between various polymer layers while also improving ink printability. CP facilitates adhesion between two or more hydrophobic materials, resulting in significantly better water vapour permeability (WVP) properties. The sputtering of ionic species by CP results in cross-linkage reactions which improve the mechanical properties of films (tensile strength (TS) and elongation at break (EAB)). Cross-linkage reactions are reported to be responsible for the improved thermal stability of CP-treated biopolymers. CP treatment is known to decrease oxygen permeability (OP) in protein-based biopolymers. CP can also enable the blending of polymers with specific antimicrobial substances to develop active packaging materials. In this review article, we have presented an overview of the recent advancements of CP in the food packaging application. Furthermore, the influence of CP on the properties of packaging materials, and recent advancements in the modification of polymeric food packaging materials have been discussed.

Keywords: cold plasma; sustainable food packaging; biopolymer modification; decontamination; biodegradable packaging

Citation: Perera, K.Y.; Prendeville, J.; Jaiswal, A.K.; Jaiswal, S. Cold Plasma Technology in Food Packaging. *Coatings* **2022**, *12*, 1896. https://doi.org/10.3390/coatings12121896

Academic Editor: Raquel Sendón

Received: 1 November 2022
Accepted: 29 November 2022
Published: 5 December 2022

1. Introduction

Conventional thermal processing including pasteurisation, sterilisation, canning, steaming, drying, evaporation, frying, cooking, baking, and blanching has remained at the forefront of the food industry for providing safe quality food, inhibiting pathogenic microorganisms, and improving nutrition [1]. Despite being the relied-upon method, conventional thermal processing leads to a significant decline in the quality of food. Thermal processing reduces the nutritional content by decreasing vitamin content, lowering the biological value of proteins, enhancing lipid oxidation, and depleting the sensory attributes of food [2]. Due to these disadvantages and increased consumer awareness around safe food, novel processing methods such as non-thermal processing including ultraviolet light, ultrasound, irradiation, cold plasma (CP), high pressure processing, and pulsed electric field are emerging technologies [3]. These new non-thermal technologies eliminate heat from processing which leads to better retention of nutrients, and better sensory attributes while inhibiting the growth of microorganisms [3]. Non-thermal processing sterilises food by altering the cell membrane or destroying the genetic material of microorganisms [3]. Despite offering great benefits, non-thermal processing has yet to become established within the food industry and remains in the developmental stage. According to a survey

conducted by Khouryieh [4], it was found that high pressure processing was the most established (35.6%) novel processing method within the food industry while atmospheric cold plasma (ACP) only had an applied usage of 2%. The major limitation of implementing these technologies is the high-cost evolvement. In addition, the effectiveness of these emerging technologies is not yet capable of matching that of traditional methods [4].

CP technology is a novel non-thermal processing method that is gaining interest within the food industry and is particularly effective in the reduction of microbial loads within a short period of time, especially in fresh fruits and meats [5]. The method provides additional beneficial outcomes such as decontamination of packaging materials, a more sustainable method, and limits browning in foods [6]. In this review article, the use of CP technology on food packaging will be analysed in terms of its mechanism, sterilisation effects, and impacts on packaging polymers.

2. CP Technology in the Food Industry

The fourth state of matter, known as plasma, can be seen as an arc or discharge of intense fluorescent light [7]. A mixture of reactive species including electrons, photons, positively or negatively charged ions, free radicals, and atoms in their excited and ground states make up plasma, which is a partially or entirely ionised gas [8]. Plasma has a net neutral charge and is referred to as quasineutral. Plasma is created under various temperatures by ionising a neutral gas and can be classified into thermal and non-thermal plasma. The generation of thermal plasma requires a high pressure above 105 Pa and an electrical supply greater than 50 MV. Non-thermal plasma can exist without a localised thermodynamic equilibrium and does not require high pressure or power [8].

Non-thermal plasma can be subdivided into two categories:

- Quasiequilibrium plasma (50–100 °C), where the reactive species are in local thermodynamic equilibrium
- Nonequilibrium plasma (<60 °C) or CP where the heavier species present have a lower temperature than the electrons.

Nonequilibrium plasma or CP is generated at atmospheric pressure upon application of an electric field to a neutral gas. Direct current or alternate current is applied to the neutral gas between two electrode plates at a set frequency (Figure 1). The stages of CP generation involve excitation, ionisation, and dissociation [9]. The reactive species found in plasma are effective against a range of pathogenic microorganisms such as bacteria, fungi, and viruses as well as pesticides and mycotoxins. CP is a promising technology for non-thermal sterilisation and the development of bio-based food packaging materials. CP allows for modification of previously unsuitable biopolymers to create packaging materials with the desired applicability. There have been modifications to the wettability, sealability, printability, adhesion, and mechanical properties [10,11].

CP technology could be beneficial within the food industry if it is applied on an industrial scale as it has recently shown great capabilities such as a reduction in spoilage, degradation of mycotoxins, inactivation of enzymes, increased bioactive compounds, improvement antioxidants, and reduced allergenicity [12]. The technology has been reported to be effective at eliminating mycotoxins commonly found in seeds, grains, and crops due to high oxidation rates [13]. In terms of enzymes, it has shown to be effective at the inactivation of enzymes peroxidase and polyphenol oxidase which are responsible for enzymatic browning in fruits [13]. In relation to bioactive components, CP can reduce germination rates in brown rice leading to an accumulation of the maximum number of bioactive phytochemicals [14]. By means of allergens, it has been reported by Bourke et al. [13] that CP can reduce the immune reactivity of proteins in soybeans through direct and indirect exposure. In the dairy sector, CP is used as a rapid, non-thermal method of pasteurising milk to maintain milk quality while ensuring food safety [15]. It has been proven by the studies of [16,17] that non-thermal CP can be used as a method of milk pasteurisation with effective microbial reduction.

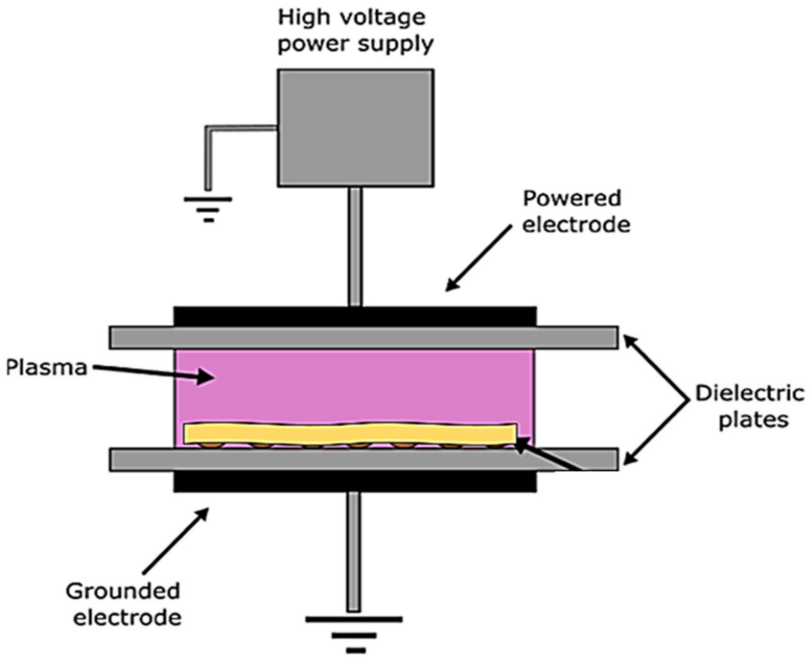

Figure 1. Schematic diagram of Dielectric Barrier Discharge (DBD) for CP treatment [18].

3. CP in Food Packaging

CP technology can also be applied to food packaging during washing through spray form preventing contamination and any unwanted growth of microorganisms [6]. Most packaging materials used in the food industry cannot withstand heat such as polyethylene terephthalate (PET). The possible use of CP is demonstrated by [19] not damaging the structures of the packaging materials as it is operated under low temperatures. It is also highly effective as it eliminates cross-contamination that usually occurs between the application of microbial limiting treatments and the packaging process of the food product [20]. In recent studies, CP treatments applied to food packages containing cabbages slices [21], Korean rice cakes [20], and fish cakes [22] all demonstrated the effectiveness of reducing bacterial loads, especially *Salmonella* spp. by applying in-package treatments. It has also shown its ability to alter the structure of cellulose compounds leading to a better breakdown which may improve the development of edible films in food packaging [23]. The preservation of fruits postharvest is difficult to maintain however ACP can be used as a preventative measure in inhibiting decay and has been seen to be successful in blueberries [24]. Further, CP technology has recently been applied to many food products and shows promising effects on food packaging decontamination which could have the potential to revolutionise traditional conventional methods within the food industry.

3.1. Impact of CP on Food Packaging Surface Sterilisation

Research suggests CP is a promising method of food packaging surface sterilisation as it is quick and induces no negative alterations to the functionality of the packaging materials. The food packaging process is considered a critical control point as part of the Hazard Analysis and Critical Control Points plan [25]. The conventional methods of sterilising food packaging include dry heat, steam, UV, and chemicals (ethylene oxide and hydrogen peroxide). Heat generating methods are a concern for certain materials such as PET, which is a temperature sensitive compound. Chemical methods of sterilisation may present health risks due to toxicity, mishandling, residue, and environmental hazards. To lower costs and avoid thermal approaches and the generation of chemical effluent, CP has been investigated as an alternative approach to packaging sterilisation. CP does not generate residue, is chemical free, safe, and applies to a range of packaging materials [19]. Muranyi et al. [26] used CP to sterilise PET packaging and other multilayer packaging

materials consisting of polyvinylidene chloride (and low density polyethylene (LDPE). They achieved a 2log$_{10}$ reduction in *Aspergillus niger* and *Bacillus subtilis* with an exposure time of 1 s. Lee et al. [27] treated glass, polyethylene (PE), polypropylene (PP), nylon, and paper foil packaging varieties using CP, inactivation of *Escherichia coli* O157:H7, *Salmonella typhimurium*, *Staphylococcus aureus* was observed. Yang et al. [28] used glow discharge plasma to sterilise PET sheets. The treatment induced a germicidal effect, and *Pseudomonas aeruginosa* was completely inactivated within 30 s. Further studies on CP inactivation of microorganisms on food packaging surfaces are depicted in Table 1.

Table 1. CP inactivation of microorganisms on food packaging surfaces.

Number	Food Type	Microorganisms	Plasma Source	Time (min)	Maximum Logarithmic Reduction	References
1	Sushi stored in PET containers	Aerobic bacteria	DBD 70–80 kV	5 min	11.5 log CFU g^{-1}	[29]
2	Modified atmosphere packaging packed ham	*Listeria monocytogenes*	DBD 03 kV at 3.5 kHz	5 min	2 log CFU g^{-1}	[30]
3	Fresh cut apples in PP and PET	SARS-CoV-2	Surface DBD 6 kV at 5 kHz	10 min	Completely Degraded	[31]
4	Processed chicken breast in PET container	*Escherichia coli* O157:H7 *Salmonella* *Listeria monocytogenes*	Atmospheric DBD CP (ADCP) Treatment at 38.7 kV	25 min	3.9 log CFU Cube^{-1} 3.7 log CFU Cube^{-1} 3.5 log CFU Cube^{-1}	[32]
5	Wheat and barley grains in PP container	Bacteria and fungi	DBD 80 kV	20 min	Barley: 2.4 log$_{10}$ CFU g^{-1} Bacteria2.1 log$_{10}$ CFU g^{-1} Fungi Wheat: 1.5 log$_{10}$ CFU g^{-1} Bacteria 2.5 log$_{10}$ CFU g^{-1} Fungi	[33]
6	Spinach in LDPE	*Escherichia coli*	DBD 12 kV/40 W at 60 Hz Air	5 min	3–5 log$_{10}$ CFU Leaf^{-1}	[34]
7	Cherry tomato in PET container	*Salmonella* sp.	DBD Air Voltage: 35 kV at 0–2.4 kHz	3 min	0.75 log CFU g^{-1}	[35]
8	Romaine lettuce in PET container			3 min	0.34 log CFU g^{-1}	
9	Mixed salad in PET container			3 min	0.29 log CFU g^{-1}	
10	Romaine lettuce in PET clamshell container	*Escherichia coli* O157:H7	DBD Air 42.6 kV at 0–2.4 kHz	10 min	0.8 –1.7 log CFU g^{-1}	[36]
11	Egg (in Shell) in rigid PP/ cryovac pouch	*Salmonella* sp.	DBD $O_2/CO_2/N_2$ 85 kV at 60 Hz	15 min	5.5–6.37 log CFU egg^{-1}	[37]
12	Salmon (fish) in ethylene vinyl acetate/polyamide/PE	*Photobacterium phosphoreum*	DBD Ar/CO_2 Frequency: 11 kV at 16 kHz	1 min	3 log CFU g^{-1}	[38]
13	Milk (Whole) in plastic (plastic type not specified)	*Escherichia coli*	Surface DBD Air 250 W at 1.5 kHzVoltage:	10 min	2.43 log CFU mL^{-1}	[39]
		Salmonella typhimurium			2.40 log CFU mL^{-1}	
		Listeria monocytogenes			2.46 log CFU mL^{-1}	
14	Cheese in plastic container (plastic type not specified)	*Escherichia coli*	Surface DBD Air 250 W at 1.5 kHz	15 min	2.88 log CFU g^{-1}	[40]
		Salmonella typhimurium		15 min	3.11 log CFU g^{-1}	
		Listeria monocytogenes		15 min	2.26 log CFU g^{-1}	
15	Beef loin in zipper bag (Polytetrafluoroethylene) with conductive sheet).	*Listeria monocytogenes*	Surface DBD N_2/O_2 100 W at 15 kHz	10 min	1.90 log CFU g^{-1}	[41]
		Escherichia coli			2.57 log CFU g^{-1}	
		Salmonella typhimurium			2.58 log CFU g^{-1}	

3.2. Impact of CP on Modification of Food Packaging Polymers

Polymeric packaging materials such as PET, PE, PP, polyvinyl chloride, and polystyrene are the common plastics utilised in the food industry. Polymers are a major contributor to the packaging industry accounting for two third of the total plastics produced [42]. Polymer packaging materials offer many benefits however their hydrophobicity and low surface energy leads to necessary surface alterations needed to obtain desirable packaging features [43]. The conventional chemical methods of modifying food packaging generate waste leading to a negative effect on the environment, however, with novel methods such as CP treatments, there is no waste generated resulting in a more sustainable solution to food packaging modification [43].

CP impacts food packaging by increasing polymer energy, modifying the surface, and increasing adhesiveness for printing and seals [12]. The impact of surface modification can be altered by different plasma configurations and electrode materials [12]. The alteration occurs after the formation of plasma whereby the active species formed lead to physical and chemical interactions on the surface of a polymer resulting in a modification of its properties including roughness, functionality, wettability, barrier function, antimicrobial, and biodegradability.

PE naturally has low surface energy which can be modified through CP treatments to produce higher surface energy, this material is mainly used within films or coatings [44]. In recent literature, PE has been applied as a nano fibrillated cellulose film [45], an LDPE bilayer film with summer savory essential oil [46], and an LDPE film coated with gallic acid [47]. The antimicrobial activity of the material in combination with CP has been proven to be effective against certain bacteria including *Escherichia coli* and *Staphylococcus aureus* [47], however, there is no reduction reported for *Listeria monocytogenes* [44]. In terms of barrier functions, the material in combination with CP has been reported in many studies to improve oxygen barrier function [45] and decrease water vapour permeability (WVP) [46]. The application of CP on LDPE increases surface roughness (SR) effectively which enhances the ability to coat the material with preservatives [47]. The material in combination with CP improves adhesion due to increased production of polar groups and increases tensile strength (TS) [46]. These beneficial outcomes are dependent upon the amount of electrical energy used and the length of the application as certain alternations of these two factors could lead to negative outcomes.

PET can be used in a variety of forms including in-package treatments, films, and foils, in recent literature the material has been used for albumin/gelatin film plates [48], bilayer films with surface CP pretreatments and in-package treatments for cabbage slices [21], chicken breast [32] and fish cakes [22]. In terms of benefits, the material has shown great adherence to microbial inhibition as it has been proven to eliminate numerous bacteria including *Escherichia coli*, *Staphylococcus aureus*, *Salmonella* spp., and *Salmonella typhimurium* [19]. In a study by Kim and Min, cabbage slices stored within PET containers observed a 0.9 log CFU g^{-1} reduction in *Salmonella* after 3 min of CP treatments [21]. However, in another study on fish cakes by Lee et al. [22] *Salmonella* growth reduction was greater between 1.3–1.4 log CFU g^{-1}. The material has also been deemed effective in combination with CP in reducing the fungus *Penicillium digitatum* which generally impacts mandarin oranges [49]. In terms of disadvantages, due to PET being hydrophobic in nature and obtaining low density polar functional groups the material is predisposed to poor dye adhesion, poor particles, and microparticles [50]. The ability to transfer ink onto the material is low creating printing constraints. CP treatments offer a solution to these issues as the application increases water permeability for PET materials improving wettability properties without chemical alteration [51].

In a study conducted by Kim et al. [52] the use of LDPE and PET packaging is compared in terms of microbial inactivation, the use of shaking, positioning, WVP, tensile strength, and opacity. In terms of sterilisation effects, LDPE packaging offered better microbial inhibition for mesophilic aerobic bacteria on grape tomatoes compared to PET packaging [52]. Microbial inhibition depends upon the packaging material's dielectric permittivity as it creates different stages of micro discharges leading to different amounts of reactive species produced during CP treatments [52]. For example, LDPE has a lower dielectric permittivity compared to PET resulting in a higher inactivation rate of bacteria as observed in this study [52]. Other factors influence microbial inhibition such as shaking and positioning of the packages. Shaking of packages before CP treatments eliminates inaccuracies as it develops contact between reactive species improving microbial inhibition [52]. There was a higher microbial inactivation rate for Salmonella at 0.4 log CFU $tomato^{-1}$ observed for grape tomatoes when shaking was applied. In Figure 2, it is clear that there is a higher microbial inactivation rate when shaking of LDPE packaging is applied.

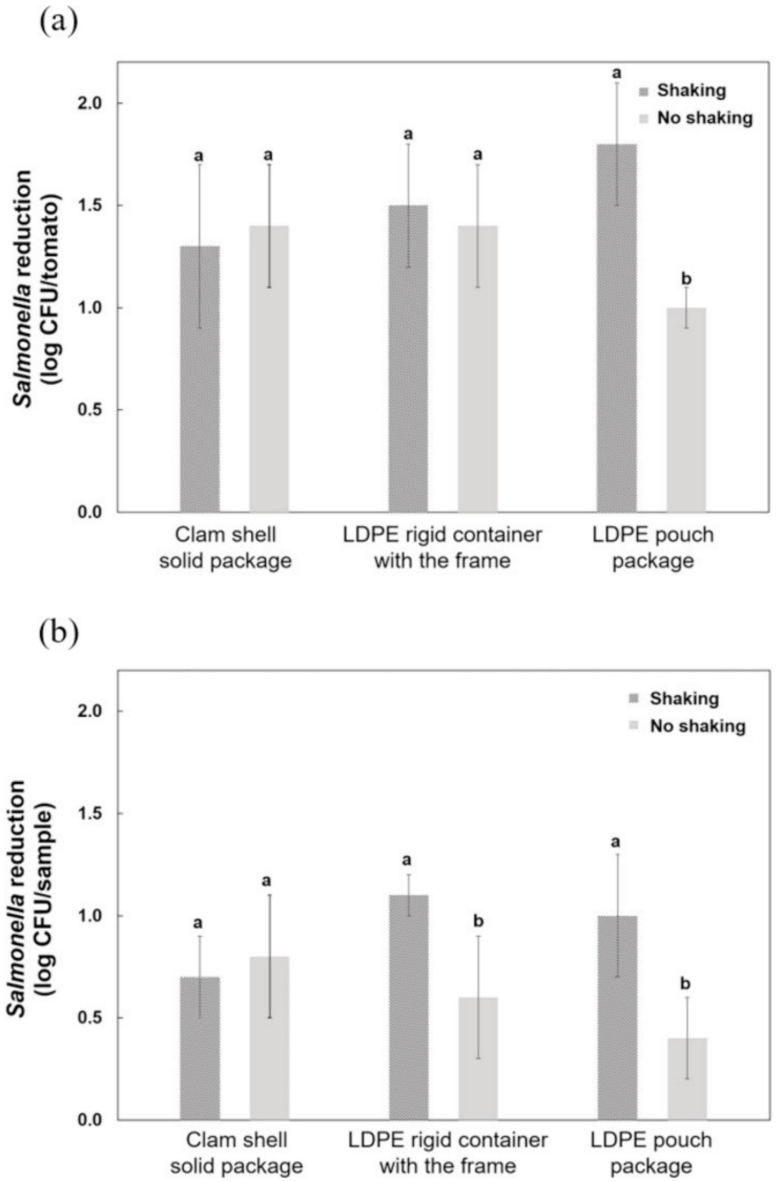

Figure 2. The effect shaking has under DBD ADCP treatment on (**a**) grape tomatoes (**b**) slices of romaine lettuce, red cabbage, and carrot with clamshell solid packages, LDPE rigid containers and the LDPE pouch package. The letters indicate significant differences at $p < 0.05$. Reprinted with permission from ref. [52]. Copyright 2022 Elsevier.

The position of packages in line with the electrodes determines the reactive species formed which also has a direct effect on microbial inhibition [52]. The water vapour barrier (WVP), TS, and opacity increased when PET and LDPE were treated under CP technology. This study shows how the two polymeric packaging materials can be applied to create beneficial effects in combination with CP treatments.

In terms of safety and quality aspects, there must be testing completed on CPs effects on food packaging materials such as PET and PE to ensure it is within migratory limits and to assess permeability associated with water vapour and O_2 [53]. There is limited research (some of these studies can be observed in Table 2) available on PET and PE as the majority of literature focuses on the impact the technology has on specific foods in terms of their sensory attributes. Overall, both polymers show promising effects for the food industry with CP treatments to enhance bacterial reductions and increase adhesiveness.

Table 2. Impact of CP on modification of food packaging polymers.

Number	Packaging Material	CP Source	Treatment	Properties of Packaging Material	References
1	LDPE-gallic acid	Cold radio frequency plasma (13.56 MHz)	Different power (0–90 W) Treatment time (15–150 s)	• Reduce growth of *Escherichia coli* and *Staphylococcus aureus* by 0.5–0.6 log CFU mL^{-1} • Highest TS for the treatment 30 W for 60 s	[47]
2	LDPE-chitosan-savory essential oil	ACP (power: 3 kW)	10 s	• Decreased WVP and oxygen transmission rate (OTR) • Improved TS and elongation at break (EAB) of film • The shelf life of fillets extended up to 13 days at the refrigerator.	[46]
3	LDPE-PET	ADCPDBD	3 min	• Increased WVP • TS, transparency, glass transition temperature, and surface morphology are unchanged	[52]
4	PET-ZnO	High voltage transformer (220 V, 60 Hz)	3 min	• Higher inactivation of *Salmonella* (2.4 log CFU g^{-1}) on ZnO CP treatment than the ZnO only (0.4 log CFU g^{-1}) or CP only (1.7 logs CFU g^{-1}) treatment	[22]
5	PET	ADCP	26 or 27 kV for 1, 2, 3, or 4 min	• Enhanced shelf-life of mandarins (oranges) in ADCP treated plastic packages by inhibiting the growth of *Penicillium digitatum*	[49]

3.3. Impact of CP on Biopolymer-Based Packaging Materials

In comparison to plastics, biopolymers are known to exhibit inferior mechanical properties, low thermal stability, poor surface functionality, poor printability, and low adhesiveness [9]. As demonstrated in Table 3, the application of CP alters the molecular structure of biopolymer films in a variety of ways, including by introducing an active site, crosslinking, rearranging, and improving the structural configuration of the polymeric network [9]. These modifications can positively impact the mechanical, thermal, and barrier properties of biopolymer films.

Polylactic acid (PLA) is well suited as a packaging material due to its biodegradability, affordability, transparency, processability, and plastic like mechanical properties [54]. PLA in its pristine state is brittle and therefore poses challenges as a packaging material. Literature suggests PLA can be reengineered using CP to combine it with other materials such as nisin, metal oxide nanoparticles, and graphite [54]. Hu et al. [54] created an antimicrobial food packaging by coating a PLA film with nisin. CP treatment facilitated adhesion between the two layers (Figure 3) and is effective against an array of Gram-positive bacteria including *Listeria monocytogenes*. Enhanced antimicrobial activity, gas barrier properties, and more suitable surface morphology were observed when CP was employed to combine the two materials.

Zein is hydrophobic and is therefore insoluble in water, it can be dissolved using ethanol or acetone. Zein films exhibit a glossy appearance, low permeability, biodegradability, and grease proof properties. It is challenging to layer a hydrophobic material onto another hydrophobic material due to the lack of polar groups. To overcome this, CP can activate the surface layer of hydrophobic materials. Plasma discharge can create polar containing groups on the surface layer, enabling interface adhesion between two hydrophobic materials [55]. Contact of zein to CP treatment for the 60 s before layering onto PLA film, enhanced hydrophobicity (by 24.1%), TS (by 50.5%), EAB (by 29.7%), and WVP (by 44.3%) in comparison to the untreated film [55]. The multilayer films showed good biodegradability and UV barrier properties. Chen et al. [55] concluded that the approach of layering a PLA coating onto zein film created a packaging material with the desired application for use in the biodegradable packaging industry. Similarly, Dong et al. [56] enhanced the water resistance properties of the zein based film by exposure to CP (Figure 4) which sequentially facilitated grafting to PCL.

Pectin is a biopolymer that is used in food packaging due to its exceptional mechanical properties, biocompatibility, biodegradability, and nontoxicity. The use of CP in combination with other bioactive chemicals has been used to improve the functionality of biopolymeric films. The aim of Jahromi et al. [57] developed pectin films with antioxidant activity by including clove essential oil emulsions stabilised by CP. A DBD device was used for the CP treatment for 5 min at a frequency of 10 kHz and a voltage of 7 kV (28 °C, 34% RH). The films had improved mechanical strength, stretchability, surface hydrophobicity, and clove essential oil retention during storage.

Studies on CP treatment in food packaging for other biopolymers such as chitosan and starch have also been performed by Sheikhi et al. [58]. Chitosan and starch are both nontoxic biopolymers that are biocompatible, and biodegradable. However, they both have poor mechanical qualities, which are significantly improved when both biopolymers are combined. For the further increment of the mechanical properties, CP treatment is carried out. Sheikhi et al. [58] investigated the mechanical and physical properties of a starch-chitosan composite film exposed to low pressure air and argon plasma at 600 mTorr for various time points (4, 8, and 12 min) and assessed the shelf life of chicken breast fillet packaged with plasma treated films. After 12 min of processing, it was observed that argon plasma is more effective at increasing the TS of the films, increased from 10.59 to 22.09 MPa. Nevertheless, the CP treatment did not prolong the shelf-life of the fillets.

Table 3. Application of CP on biopolymer based packaging materials.

Number	Packaging Material	CP Source	Treatment Times	Properties of Packaging Material	References
1	Zein-chitosan	DBD plasma reactor Treatment power: 100 W	30, 60, 90, 120, 150 s	• A 60 s plasma treatment showed optimum tensile strength, improved water • Vapour barrier and improved thermal stability • Long term plasma exposure (120 s or greater) caused protein oxidation and strengthening of the etching influence on the surface	[55]
2	Fish myofibrillar protein	Alternating current glow discharge plasma Power supply: 0–15 kV, 60 Hz	2 or 5 min	• Films treated for 2 min CP showed improved EAB and decreased tensile strength, • Films treated for 5 min CP showed decreased EAB and increased tensile strength • Water solubility increased with 5 min treatment • WVP improved with 2 min of treatment	[59]
3	Pectin-clove essential oil	DBD system, at a frequency of 10 kHz and a voltage level of 7 kV for	5 min	• High mechanical strength and stretchability • High surface hydrophobicity, mechanical strength, and flexibility • Higher retention of clove essential oil during storage	[57]
4	Starch-chitosan	Radio frequency (20 kHz) plasma cleaner system	4, 8 and 12 min	• TS increased from 10.59 to 22.09 MPa after 12 min of treatment • Solubility and hydrophilicity of the films increased • OP of films improved • CP treatment did not extend the shelf life of the fillets	[58]
5	PCL-zein	ACP	30 s at 35, 50, 65, 80, or 100 V, and 65 V at 5, 15, 30, 45, or 60 s	• ACP aid as an efficient surface grafting pre-treatment approach	[60]
6	Corn starch (28% amylose)	DBD	20 kV, 200 Hz, 10, 15, and 20 min	• Reduction in hydrophilicity and water solubility • Increase in strength and stiffness • No changes in WVP were observed • TS increased • EAB decreased • CA increased • SR increased • Glass transition temperature significantly decreased • T_m significantly decreased • T_{deg} temperature significantly increased	[61]

Table 3. *Cont.*

Number	Packaging Material	CP Source	Treatment Times	Properties of Packaging Material	References
7	Corn Starch-poly (ε-caprolactone Starch	Radio-frequency generated plasma reactor	40 W, 13.56 MHz, 25 min	• CA decreased, 23.01% increase in polarity observed after 5 min exposure to plasma • SR increased by 33.27% after 5 min exposure • Slight increase in WVTR and OTR after 2 min exposure. After 5 min unacceptable barrier properties were observed • An increase for both TS and EAB was reported • Biodegradation increased. A 34% loss in TS and 42% loss in EAB were reported when buried in soil for 56 days • Higher microbial activity was observed on film compared to untreated film	[62]
8	High amylose corn starch	DBD plasma system	80 kV, 5 min	• CA decreased • SR increased • T_{deg} temperature decreased from 242.5 °C to 241.24 °C • Surface hydrophilicity increased; new oxidised species were identified on the surface • No change in WVP	[63]
9	Zein films (pristine)	DBD50 Plasma Reactor Power: 65 V Outer electrodes diameter = 50 mm	65 V, for 5, 15, 30, 45, and 60 s	• After 45 s contact TS was 10.0 ± 1.28 MPa greater than control • EAB increased by 28.57% compared with the untreated (30 s) • CA decreased from 72.3° (untreated) to 48.4° (treatment for 60 s) • SR improved, after 60 s treatment, and values increased up to 84.1 nm, which was 20 times the untreated • WVP decreased by 24.5% after treatment for 15 s (compared to untreated) • T_{deg} slightly increased due to reported crosslinking at surface level	[43]
10	Bovine Gelatin films	DBD plasma system	60, 70, and 80 kV for 1, 2, 3, 4, and 5 min	• CA decreased from 126.3° to 58.1° after 5min 80 kV • SR increased from 2.73 nm to 4.5–12.8 nm after plasma treatment • No significant change in WVTR or OP	[63]
11	Gelatin films	DBD (atmospheric pressure by air, N_2, O_2, Ar, and Ar ethanol gases)	19W (Air), 16.5W (Ar), 30W (N_2), 27W (O_2), 19.5W (ethanolAr) for 15 min	• CA decreased; surface hydrophilicity increased. Owing to the increased population of polar groups on the surface • SR increased significantly • OP decreased, due to crosslinking after plasma treatment, thus preventing oxygen transmission • T_m increased, resulting in higher heat capacity of film • No significant change in mechanical properties of the film after plasma exposure • No significant change in WVP	[64]
12	Polycaprolactone-zein	ACP	65 V for 5, 15, 30, 45 and 60 s	• TS increased from 17.2 MPa to 18.5 MPa • EAB increased • CA increased • T_m decreased • Grafting ability increased from 12.2% (untreated) to 28.9% (treated) when exposed to plasma for 30 s at 65 V	[60]
13	Zein-PLA	DBD CP system	60 V for 60 and 120 s	• TS increased by 50.5% compared to control • EAB increased by 29.7% • SR decreased • Surface hydrophobicity increased by 24.1% • CA decreased from 75.2° to 42.2° • WVP decreased by 44.34% • T_{deg} increased • A higher rate of biodegradation was observed, resulting from the increase in surface layer pores after plasma exposure • UV barrier improved due to successful grafting of two biopolymer layers	[65]

CA Contact Angle; EAB Elongation at Break; OP Oxygen Permeability; SR Surface Roughness; T_{deg} Thermal Degradation; T_m Melting Temperature; TS Tensile Strength; WVP Water Vapour Permeability; WVTR Water Vapour Transmission Rate.

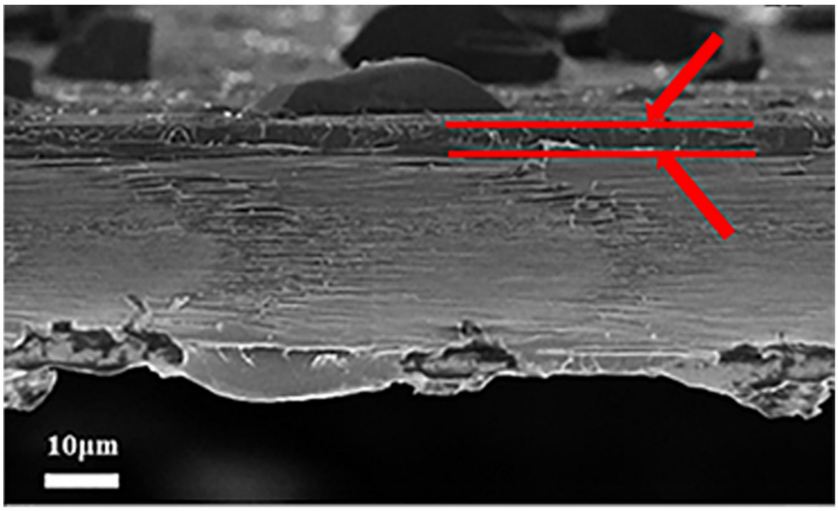

Figure 3. CP treatment effectively facilitated the grafting of nisin onto PLA film. Reprinted with permission from ref. [54]. Copyright John Wiley and Sons 2022.

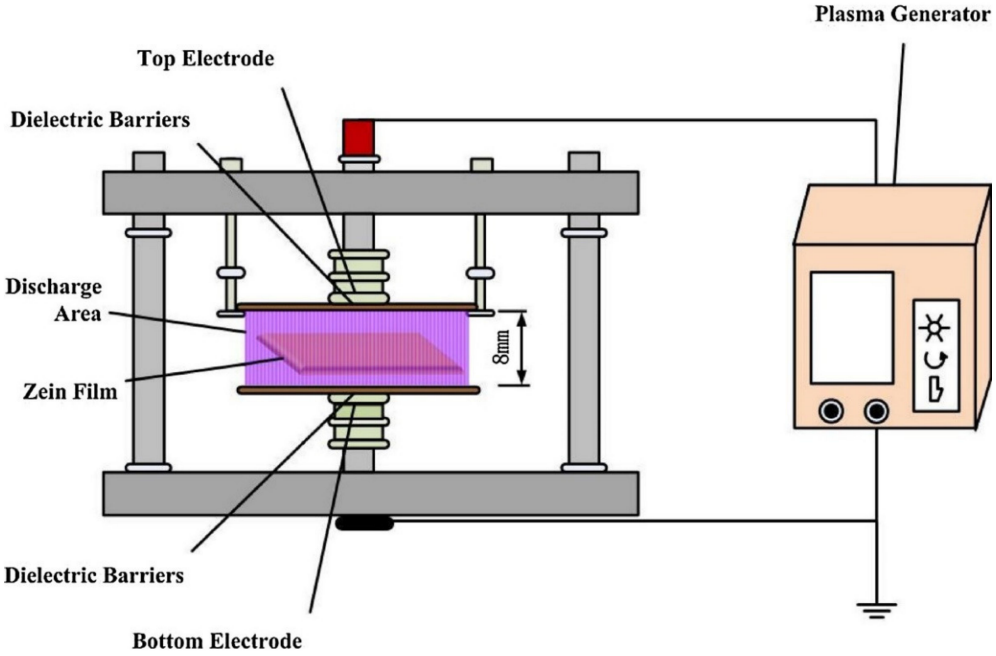

Figure 4. Representation of the experimental setup for treatment of zein films with ACP. Reprinted with permission from ref. [43]. Copyright Elsevier 2022.

4. Effect of CP on Packaging Properties

4.1. Surface Roughness (SR)

CP treatment alters the surface of biopolymer materials by increasing surface roughness. This is due to the bombardment of high energy plasma species i.e., electrons, photons, and charged atoms that etch the film surface [44]. The etching effect from CP treatment promotes adhesion between various surfaces and facilitates the production of multilayer films. Particularly in non-adsorbent biomaterials like PLA, the roughness induced by CP treatment improves ink printability [66]. After CP treatment an increase in SR was observed for biofilms composed of PLA and nisin. The untreated films had a SR value of 4.26 nm, after CP treatment for 30 s, SR increased to 6.24 nm [54]. The degree of roughness caused by CP was dependent upon exposure time and voltage applied (Figure 5). These results agree with the studies of Romani et al. [67] where the SR of the fish protein films with the CP treatment was observed by the SEM. The increase of the SR results in the enhancement

of physicochemical and biodegradable properties of the films. Hu et al. [54] reported that increased CP treatment exposure resulted in increased roughness of nisin coated PLA films. The untreated film had a roughness of 4.3 nm, roughness increased to 5.2 nm and 6.24 nm with 15 s and 30 s exposure, respectively. With exposure times above 45 s, roughness decreased significantly.

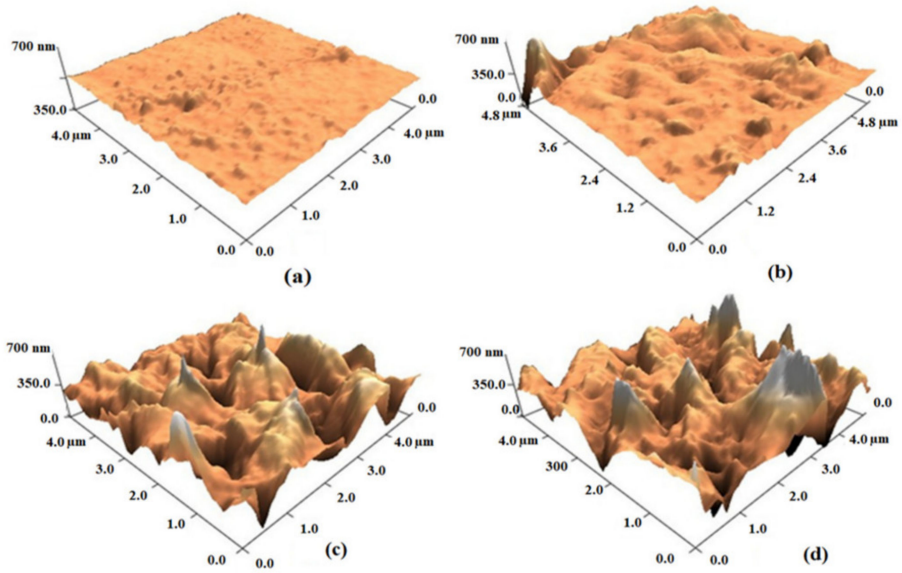

Figure 5. Atomic force microscopy (AFM) showing the measure of the roughness of (**a**) untreated zein-PLA film, (**b**) 60 kV, 5 min, (**c**) 70 kV, 5 min, (**d**) 80 kV, 5 min [9].

4.2. Contact Angle (CA)

CA refers to the interaction and spread of liquid across the surface of materials. Materials with an attraction for water are hydrophilic, water spreads across the surface area, creating a lower CA. Materials which repel water are hydrophobic, water will not spread or adhere to this material, therefore will have a higher CA. By making biopolymer films more hydrophilic and wettable, CP treatment has been proven to reduce the CA. CA [9]. According to Song et al. [67] adding CP to the PLA film surface increased polarity, which in turn boosted the film surface's hydrophilicity and wettability. Sandanuwan et al. [51] found CA decreased with higher exposure times to CP. CA decreased faster when the biofilm was closer to the plasma jet tip. The studies of Jahromi et al. [57] also agree with the above studies where the water CA of pectin clove essential oil packaging films has increased from $74.88 \pm 1.92^{\circ}$ to $86.86 \pm 0.27^{\circ}$ with 5 min CP treatment.

4.3. Mechanical Properties

Mechanical properties such as TS and EAB of packaging films are key parameters in determining packaging effectiveness to extend the shelf-life of the food. The mechanical properties relate directly to the integrity of the film. CP treatment causes etching and crosslinking of the film surface, which can alter the mechanical properties of the films. Ionic species are sputtered onto the film surface, breaking the C-C and C-H bonds and producing free radicals that take part in cross-linkage reactions that improve the mechanical properties [9,46]. Several studies have shown TS to increase with time exposed to plasma treatment. This is due to the longer interface time between plasma generated radicals and the biopolymer surface [9]. Dong et al. [60] studied the combined impact of voltage and plasma treatment time on the mechanical properties of a zein PCL multilayer biopolymer. TS and EAB were significantly enhanced with plasma treatment for 15 s, however, when voltage was increased above 100 V the TS decreased. They concluded that the mechanical properties of the biopolymer depended on the bulk matrix rather than the surface layer. The

mechanical properties of biopolymer films are a function of various factors such as the material type, treatment time, voltage, and the gas type used for plasma generation [9,55,68].

4.4. Thermal Properties

Heat sealing is a crucial step in the packaging process, hence thermal characteristics are essential in assessing the efficacy of biopolymers [9]. Dong et al. [43] found CP pretreatment for 60 s improved the denaturation temperature of a zein-PCL film. They stated this was due to the crosslinking of polar compounds on the film surface. Chen et al. [55] reported similar results for zein-PLA films where plasma treatment led to increased thermal stability, they specified this was due to the hydrogen bonding between zein and PLA molecules. However, Chen et al. [55] found exposure to plasma for longer than 120 s to result in inferior thermal properties to that of untreated films. Thus, overexposure to CP can negatively impact the thermal properties of films.

4.5. Water Barrier Properties

The WVP determines the effectiveness of the moisture barrier properties of packaging material. It is directly related to the shelf life and quality of the food. Biopolymer films are known to possess poor moisture barrier properties. Several studies have employed CP to modify WVP properties. Chen et al. [55] found treating starch and PCL films with plasma improved the WVP properties by 94%. Similarly, Dong et al. [43] found CP improved the WVP properties of zein-PCL films by 24% compared to the untreated control. Other studies have found plasma treatment to have no impact on WVP properties [58,64]. According to Hoque et al. [9] WVP is influenced by the surface characteristics, thermodynamic characteristics, vapour pressure, and concentration gradient across the films. Irrespective of CP treatment, the biopolymer matrix, and materials selected to construct the film can impact moisture barrier properties. LDPE packaging properties before and after plasma treatment were examined by [52]. They stated no significant changes to have occurred, however WVP increased after CP treatment.

4.6. Oxygen Permeability (OP)

OP relates to the rate of oxygen transport across the film, which determines the shelf life and quality of the packaged food item. Materials with a low OP are desired for food packaging applications. Biopolymers have poor OP properties compared to conventional plastic. Several studies found OP significantly improved after CP treatment of biopolymers constructed of gelatin, whey, gluten, and starch [58,64,68]. In contrast, Pankaj et al. [63,69] reported CP treatment to result in no significant change to the OP of gelatin and chitosan-based films. In the studies of Sheikhi et al. [58] OP of films improved after CP treatment. The oxygen transmission rate of the untreated film was 0.062 ± 0.02 cm^3 mm m^{-2} day^{-1}, nevertheless, when treated with air or argon plasma for 12 min, it increased to 0.024 ± 0.015 and 0.015 ± 0.013 cm^3 mm m^{-2} day^{-1}, respectively.

4.7. Antimicrobial Properties of CP Treated Films

The in-package plasma treatment of food depends upon the packaging material as a dielectric layer. Exposure of the packaging surface to plasma ensures the internal package surface is decontaminated [53]. Films containing essential oils and antimicrobial peptides have shown higher activity after plasma treatment. CP treatment has no direct impact on antimicrobial activity, however, the increased adhesion between biopolymer layers protects the antimicrobial components thereby increasing antimicrobial activity [9]. Hu et al. [54] reported CP treated films consisting of nisin and PLA to exhibit a log reduction of 3.23 against *Listeria monocytogenes*. They acknowledged that the film composed of purely PLA did not inhibit *Listeria monocytogenes*. However, the introduction of nisin to the film matrix was facilitated by CP which increased the antimicrobial properties.

Loke et al. [70] loaded cinnamaldehyde into a packaging film composed of carboxymethyl cellulose and LDPE. They reported enhanced attachment between layers

and high antimicrobial activity against *Staphylococcus aureus* and *Escherichia coli*. Antimicrobial efficacy was also tested against *Vibrio parahaemolyticus* in packed fish fillets stored at refrigeration temperature, a decrease of 1.27–1.92 log CFU g^{-1} was observed on the CP treated film containing cinnamaldehyde. Similarly, Wong et al. [47] prepared CP treated films composed of gallic acid and PE. The film was effective at reducing *Escherichia coli* and *Staphylococcus aureus* by 0.5–1.1 log CFU g^{-1}. Therefore, research suggests that CP can enable the blending of polymers with specific antimicrobial substances to develop active packaging types. On chicken breast meat packaged in air, Zhuang et al. [3] showed that in-package DBD plasma therapy considerably decreased the populations of both food-borne pathogens (*Campylobacter* and *Salmonella*) and spoilage microorganisms (*psychrophiles*) by as much as 1 log. However, CP treatment was noted to modify the way raw meat appeared by lightening the surface colour. *Salmonella* was more successfully inactivated on grape tomato surfaces when the packaged food was placed closer to the electrodes, according to Kim et al. [52]. Therefore, treatment was more effective when the gap between the food and electrodes was minimised.

4.8. Biodegradability

The enzymes produced by bacteria, yeast, and fungi are responsible for degradation action. However, these enzymes are ineffective against traditional plastics, hence the reason for plastic pollution. CP treatment is reported to enhance the rate of biodegradability of biopolymer films [55,62,71,72]. Arolkar et al. [62] found the biodegradability of films composed of corn starch and poly(ε-caprolactone) increased after exposure of the films to plasma treatment. The films were buried in soil and the biodegradability was measured by changes in TS and EAB. Biodegradability was reported to be a function of plasma exposure time. Song et al. [67] found CP treated packaging consisting of zein and PLA to degrade faster than the untreated control when left in the soil for 35 days. They concluded that plasma treatment increased the surface area of porous structures which facilitated swift microbial degradation (Figure 6). Oh et al. [72] reported similar results for films composed of defatted soya bean meal, faster degradation by microorganisms was attributed to the development of porous structures induced by CP treatment.

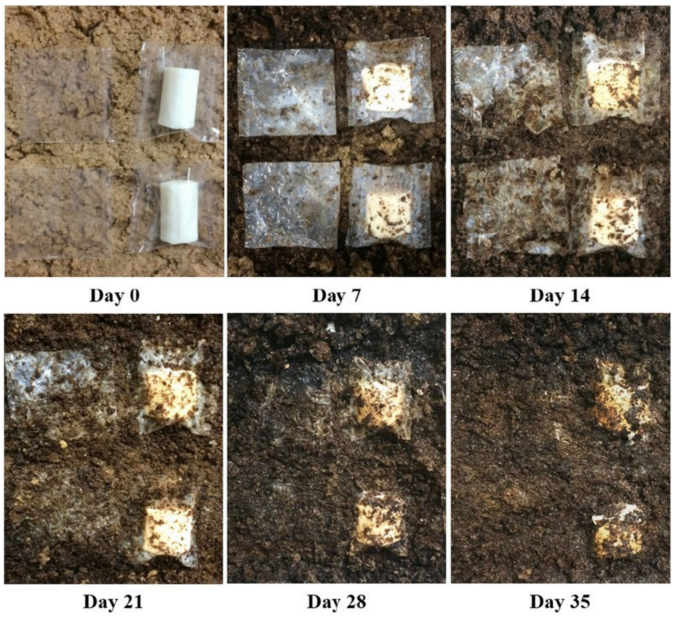

Figure 6. Effect of CP treatment on biodegradability of PLA film when composted at 90% relative humidity at 37 °C for 35 days. Reprinted with permission from ref. [67]. Copyright John Wiley and Sons 2022.

5. Safety of CP for Food Packaging Applications

The application of CP in food processing and packaging applications is a novel technique that still requires approval. The Food and Drug Administration (FDA) has not approved the use of plasma technology in food processing or packaging applications. Plasma generates reactive species which come into direct contact with food surfaces. The interaction between these reactive species and the food matrix is not fully elucidated. The possible harm that plasma-generated reactive species may cause to human health has only been briefly investigated [10,73]. According to research by Heslin et al. [74] plasma treatment of lettuce resulted in a minimal in vitro cytotoxic effect and spontaneous CHO-K1 (mammalian cells) mutations. Therefore, a risk assessment should be conducted to assess the potential toxicity of plasma treated food or food packaging for both in vitro and in vivo environments [9]. Plasma treatment may cause alteration to certain food constituents. The creation of unfavorable metabolites like short-chain aldehydes, keto acids, hydroxyl acids, and short-chain fatty acids has been documented as a result of modifications to amino acids, the oxidation of high molecular weight molecules to organic acids, and the peroxidation of lipids [9,73,75,76]. With fruit, plasma has been reported to decrease firmness, promote discoloration, and raise acidity levels [10].

Kim et al. [77] used plasma treated water as a nitrite source for sausage meat. As part of the study, the immunological toxicity and mutagenicity of sausage made utilising plasma-treated water were assessed. No negative toxicological response was reported with mice fed plasma treated sausage meat. Jo et al. [78] reported similar results in a study in which rat models were fed plasma treated mushroom powder. The rat models were fed a single dosage of winter mushroom powder (5000 mg/kg body weight) before an acute toxicity test was conducted on them. The results showed that non-thermal plasma therapy had no mutagenicity or immediate damage. Han et al. [79] also investigated the acute and subacute oral toxicity in rat models to explore the safety of edible films treated with CP. After 14 days of monitoring, rats given 5000 mg/kg of the edible film did not exhibit any symptoms of acute toxicity or die. However, changes in the levels of blood components were reported. Alterations in bilirubin, hematocrit, hemoglobin, creatinine, and aspartate aminotransferase were described by the authors. However, because the levels were within the normal physiological ranges, it was decided that these alterations were toxicologically inconsequential. Han et al. [79] found that CP treatment produced very little toxicity in edible films, indicating that CP therapy has no negative side effects. Lee et al. [80] reported similar results for chicken breast treated in-package with CP. Hematological and blood biochemical indicators were used in acute and subacute toxicity testing, and the results demonstrated the chicken breast treated with CP was toxicologically safe. Further, the study by Pogorzelska-Nowicka et al. [81] demonstrated that the sage extracts obtained using cold plasma improves the quality of ground beef. The Sage extracted obtained from CP was able to inhibit lipid oxidation and a high amount of n-3 fatty acids at the end of storage in grounded beef. Misra et al. [53] emphasised the need to analyse how the CP treatment of the packaging affects the migration of additives, monomers, oligomers, and low molecular weight compounds into the food for safety reasons.

6. Disadvantages of CP Technology in Food Packaging Applications

It is important to conduct further research on the possibility that a plasma-produced gas mixture could develop dangerous reactive species in food substances that could react with various food items. According to reports, the plasma process generates reactive oxygen species (ROS) such as hydroxyl radicals, hydrogen peroxide, and superoxide anions that assist in microbial cleansing and can start the oxidation of lipids by removing hydrogen atoms from them [5]. Pork, beef, poultry, seafood, sushi, white rice, brown rice, and wheat flour were all observed to have these negative effects, including an increase in the peroxide value and reactive 2-thiobarbituric acid compounds [5]. To treat any food products with a high lipid content, all of these CP technology applications must be optimised. Additionally, non-optimised CP procedures can cause product quality losses such as lipid oxidation,

protein aggregation, and off-flavor even while they are successful in achieving significant microbial reduction [15]. Another drawback is that endogenous enzymes like polyphenol oxidases and peroxidases, which produce enzymatic browning and surface degradation, cannot be completely inactivated by CP [82]. Therefore, the CP used in applications involving food and food packaging should be optimised based on treatment times, gas compositions, and mode of treatment before use.

7. Conclusions and Future Perspectives

CP is a novel and highly dependable method for decontaminating, preserving, and sterilising food packaging surfaces. CP is an effective method of modifying and creating ecofriendly biodegradable packaging materials. The etching effect along with the surface deposition of polar functional groups promotes adhesion of previously nonbindable biopolymer materials to create new packaging materials with the desired applicability. Many studies have found CP to improve the mechanical, gas barrier, thermal characteristics of polymer films for the development of new packaging materials. Further, CP also enhances the antimicrobial activity of biopolymer films by increasing the retention of antimicrobial compounds within the film matrix. However, limited studies have been carried out on the toxicity, lethality, allergenicity, and biosecurity of the CP treatment on food packaging materials. As a result, further research is needed to comprehend the interaction between plasma generated reactive species and the dietary matrix. The composition of the biopolymer (packaging) structure, the type of plasma gas, and processing conditions (treatment time and voltage) can impact the effectiveness of in-package plasma treatment. CP is a sustainable process that generates no waste or chemical residue. In industry, CP can allow food business operator's to decontaminate packaging and food together rather than separately. Therefore simplifying the production line. Plasma processes can be scaled up for continuous production lines by moving food in closed containers through DBD electrodes on a moving conveyor belt. Pilot scale examples have been built by [83]. At present, adoption is constrained due to the lack of appropriate equipment that can match the output of existing technologies.

Author Contributions: Conceptualisation, S.J. and A.K.J.; writing—original draft preparation, K.Y.P. and J.P. writing—review and editing, K.Y.P., S.J. and A.K.J.; supervision, S.J. and A.K.J. All authors have read and agreed to the published version of the manuscript.

Funding: The present work was supported by Technological University Dublin-City Campus, Ireland under the TU Dublin Researcher Award 2021.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

Abbreviation

ACP	Atmospheric-pressure cold plasma
ADCP	Atmospheric dielectric barrier discharge cold plasma
CA	Contact Angle
CP	Cold plasma
DBD	Dielectric barrier discharge
EAB	Elongation at break
LDPE	Low density polyethylene
OP	Oxygen Permeability
OTR	oxygen transmission rate
PCL	Polycaprolactone
PE	polyethylene

PET	Polyethylene terephthalate
PLA	Polylactic acid
PP	polypropylene
SR	Surface Roughness
T_{deg}	Thermal Degradation
T_m	Melting Temperature
TS	Tensile strength
WVP	Water vapour permeability
WVTR	Water Vapour Transmission Rate

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