Selective and sensitive analysis of pyrrolizidine and tropane alkaloids in food

Pyrrolizidine and tropane alkaloids are natural compounds with alkaloid structures that are produced by plants as a defense against herbivores. Due to their varying degrees of toxicity, regulatory authorities view their presence in food as a serious health concern.

For this reason, food testing labs are looking to implement analytical methods for these compounds in their existing workflows for residues and contaminants.

Methods that use SCIEX instrumentation are available now to help laboratories perform these important analyses and are explained here:

Food and Environmental
An accurate and sensitive method for the quantification of 33 pyrrolizidine and 21 tropane alkaloids in plant-based food matrices

Food and Environmental
Highly selective analysis of pyrrolizidine alkaloids in herbal extracts

- **Highly selective analysis of pyrrolizidine alkaloids in herbal extracts** – technical note
- **An accurate and sensitive method for the quantification of 33 pyrrolizidine and 21 tropane alkaloids in plant-based food matrices** – application summary

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CONCISE REVIEWS & HYPOTHESES IN FOOD SCIENCE

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Impact of cold plasma on the biomolecules and organoleptic properties of foods: A review

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Abstract: Cold plasma is formed by the nonthermal ionization of gas into free electrons, ions, reactive atomic and molecular species, and ultraviolet (UV) radiation. This cold plasma can be used to alter the surface of solid and liquid foods, and it offers multiple advantages over traditional thermal treatments, such as no thermal damage and increased output variation (due to the various input parameters gas, power, plasma type, etc.). Cold plasma appears to have limited impact on the sensory and color properties, at lower power and treatment times, but there has been a statistically significant reduction in pH for most of the cold plasma treatments reviewed ($p < 0.05$). Carbohydrates (cross linking and glycosylation), lipids (oxidation), and proteins (secondary structure) are more significantly impacted due to cold plasma at higher intensities and longer treatment times. Although cold plasma treatments and food matrices can vary considerably, this review has identified the literary evidence of some of the influences and impacts of the vast array of cold plasma treatment parameters on the biomolecular and organoleptic properties of these foods. Due to the rapidly evolving nature of the field, we have also identified that authors prioritize the presentation of different information when publishing from different research areas. Therefore, we have proposed a number of key physical and chemical cold plasma parameters that should be considered for inclusion in all future publications in the field.

KEYWORDS
cold plasma, food, food flavour, surface modification

1 | INTRODUCTION

There is a pressing need to develop nonthermal processing technologies that can provide the same antimicrobial effects as thermal processing technologies without the same loss of quality. Cold plasma (CP), for example has been shown to significantly reduce the presence of *Salmonella Typhimurium*, *S. Enteritidis*, *Escherichia coli*, *Staphylococcus aureus*, *Listeria monocytogenes*, *Campylobacter jejuni*, *Campylobacter coli*, and *Aeromonas hydrophila* (Niemira, 2012; Schnabel et al., 2015; Ziuzina et al., 2014). The advantage of this for the food industry is that it reduces the need for lengthy microbial treatment procedures, and the food will not need time to cool (Misra et al., 2014). Furthermore, the review by Lafarga et al. (2019) demonstrated that with further research, CP could

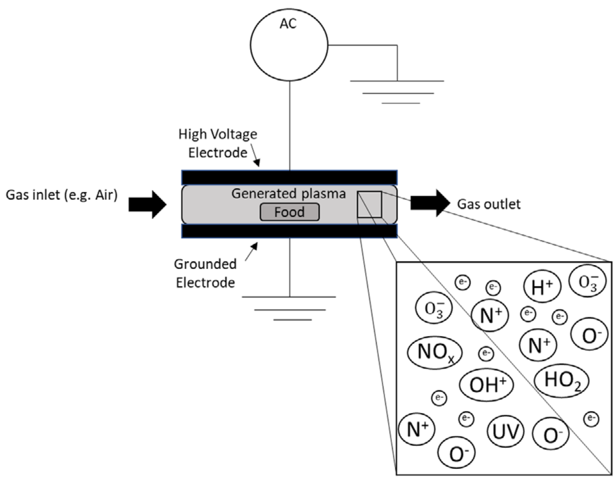

FIGURE 1 An example of an air-based direct dielectric barrier discharge cold plasma (DBD-CP) device and some of its subsequent plasma output including ultraviolet (UV), free electrons, and reactive ions. Output species were obtained from Klämpfl et al. (2012)

be a suitable alternative to chemical disinfection because it does not leave halogen residues behind. As such, CP is an advantageous nonthermal treatment because, with sufficient temperature (~20°C) and pressure (~14 psi) control, it can be used to retain or potentially improve the organoleptic and biomolecular properties, which are typically negatively affected with thermal treatments (López et al., 2019).

Plasma is the ionized form of gas and is formed by either the thermal or nonthermal ionization of molecules and atoms into free electrons, cations, anions, free radicals, exited molecules and atoms, and electromagnetic radiation (e.g., ultraviolet (UV) light) (Figure 1) (Misra, Pankaj, et al., 2016; Pan et al., 2019). Depending on the source of energy applied to the gas, these ionized atoms, molecules, and free electrons will exist in a thermodynamic equilibrium (thermal plasma), a quasi-equilibrium (low temperature plasma in intraspecies thermodynamic equilibrium: ~100–150°C), or a nonequilibrium state (nonthermal or cold plasma <60°C) (Misra, Pankaj, et al., 2016). Inducing this ionization via thermal energy (~20,000 K) will produce thermal plasma (Misra, Pankaj, et al., 2016). Ionization from low temperature energy sources form plasmas in quasi-equilibrium and nonequilibrium states.

CP (also called nonthermal plasma) refers to plasma in a nonequilibrium state. This is where the electrons have been highly energized (10^4–10^5 K) but, due to their distance from the nuclei and their relatively small size, they transfer very little thermal energy to the overall plasma temperature (293 K–423 K) (Misra, Pankaj, et al., 2016; Pan et al., 2019; K. Zhang et al., 2019). CP treatments use this nonthermal energy to generate a plasma that can etch, add, or modify the surface of materials including food (Thirumdas et al., 2014).

CP can be applied in three ways: direct, semidirect, and indirect plasma. Direct plasma is plasma applied directly to the food sample (Figure 1) (Almeida et al., 2015; Pan et al., 2019), semidirect plasma uses a mesh between the source and sample (Schottroff et al., 2018), and indirect plasma uses a postplasma treated vector (e.g., water or air) which the foods are then exposed to (Almeida et al., 2015).

CP can be generated using many methods including AC corona discharge (CD), AC/DC high voltage current, dielectric barrier discharge (DBD), cascaded DBD, thin layer DBD, diffuse coplanar surface barrier discharge, microwaves, radio frequency (RF), AC plasma arc, and gliding arc discharge plasma (Chen et al., 2019; López et al., 2019; Misra, Pankaj, et al., 2016; Misra, Schlüter, et al., 2016; Pankaj et al., 2018). Of these, DBD and RF plasma jets are the most common, followed by AC corona discharge, microwave, and gliding arc discharge plasma (Pan et al., 2019; Thirmundas et al., 2014). DBD cold plasma, like the example in Figure 1, uses the potential voltage difference between a high voltage electrode and a grounded electrode to generate a large capacitance and an oscillating direct or alternate current discharge (Pan et al., 2019). Radiofrequency induced CPs; however, it uses voltages to alternate the charge, and therefore the position, of the anode and cathode (Thirmundas et al., 2014). AC corona discharge uses a high voltage AC to pass via one or more electrode heads through a gas onto a plate (typically aluminum), thereby ionizing the gas in between and forming a plasma (Kasih et al., 2020). A microwave-based plasma uses a magnetron to generate microwaves at ~2.45 GHz, which then typically passes through a waveguide and a tuner to reduce reflectance, and then out through an antenna to focus the microwave through the quartz barrier to ionize the gas surrounding the food (Wiktor et al., 2020). This type of plasma also requires a cooling system as it can reach up to 120°C (Schnabel et al., 2015). Gliding arc discharge plasma uses a pulsed high voltage current to pass between two diverging electrodes, creating the plasma arc, through which the desired gas will pass through and ionize before reaching the samples (Ashtiani et al., 2020).

CP generation systems induce ionization in the chosen gas, which could be one or more of the following: air, argon, helium, nitrogen, oxygen, and carbon dioxide (Pan et al., 2019). Depending on the gas pressure, these CPs can be further defined as low, atmospheric, or high-pressure plasma systems (López et al., 2019). DBDs, RF, fluorescent, and low-pressure DC discharges can all be used at low to standard (0.1–1.0 bar) atmospheric pressure (Thirumdas et al., 2014).

2 | APPLICATIONS OF CP

Apart from considering what the organoleptic and biomolecular impact of CP has on the intended food product, it is also important to identify what CP treatments and parameters can be used. For example, direct DBD plasma limits the sample size that can be used, because there is a gap limit (predominantly dependent on the gas and media permittivity and the voltage output) between the two opposing electrodes in which the sample would be placed (Chen et al., 2019). The distance from the plasma source can subsequently affect the type of ions that interact with the food, for example, reactive mono oxygen species and free hydrogen ions are more likely to be found nearer to the plasma source because of their short half-life and volatility (Klämpf et al., 2012; Pan et al., 2019). It is worth noting, however, that DBD plasmas could be applied to a liquid or gas vector and then applied indirectly to a sample and not be limited by the electrode gap limit. The treatment area for RF plasma jet is small, but it is more adjustable, and the sample does not need to be in between the electrodes (Chen et al., 2019). Microwave plasma generation is more powerful, although this power means that it requires a cooling chamber (Chen et al., 2019).

Many effects of CP have been researched including the organoleptic impact, biomolecular impact, plant cell structure, and more extensively, the microbial impact. There are, however, still many gaps in the research into the mechanisms and impact of CP on the organoleptic and biomolecular properties of food. These gaps should be thoroughly examined before this can be considered as a viable technology for the food industry. This review will aid in bridging these gaps by providing a comprehensive overview of the current (over the last ~10 years) peer-reviewed research aimed at using CP to improve and alter the biomolecular and organoleptic properties of food. A narrative review will be carried out to allow for direct comparison between the different CP sources, their variation in parameters, and what foods have been trialed for each one. The intention of this will be to identify how previous research in CP should be expanded and refined in future studies and to make recommendation on consistency of data presentation to maximize knowledge exchange in the subject field.

3 | REVIEW METHODOLOGY

This review used Web of Knowledge, Google Scholar, NuSearch (University of Nottingham, Nottingham, UK), and Pubmed to identify key peer-reviewed publications in the field. The growth in publications in the area is highlighted in the infographic (Figure 2) that illustrates the number of publications per year recorded in Web of Science that include the term "cold plasma" and "food." It should be noted that the data has been truncated to 2000–2020 to aid interpretation of Figure 2.

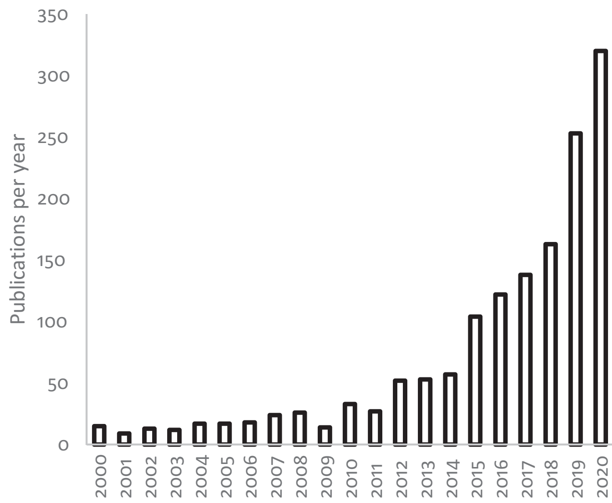

FIGURE 2 Publications per year, data sourced from Web of Knowledge and illustrative of publications including the terms "cold plasma" and "food" during the period, selected data is included (2000 onwards) to highlight research trajectory

4 | COLD PLASMA

Figure 3 shows the application of CP in variety of food and beverage processing such as fruit and vegetables, dairy products, grain and cereals, meat and related products, and beverages (Bourke et al., 2018; J. Lee et al., 2017; Pankaj et al., 2014, 2018; Segat et al., 2015; Silveira et al., 2019). CP can be used as a simple technology to disinfect bacteria and modify food structure in order to enhance its appearance and flavor. Previous studies have shown that CP treatment can impact surface structure and functional groups of amino acids, proteins, amino acids, carbohydrates and other macromolecules (Alves Filho et al., 2020; Mandal et al., 2018; Xu et al., 2017). This technology has also been investigated worldwide for 30 years and has recently been considered as a promising food processing technique in terms of microbial inhibition, enzyme inactivation, and volatile organic compound destabilization (Ekezie et al., 2017). It consumes less energy, has short processing times, and relatively low cost in comparison with the traditional technologies (Alves Filho et al., 2020); however, it only has limited commercial application to date due to slow legislative approval.

The application of CP itself or in combination with other processing technologies might create a breakthrough processing technique for future food processing industries.

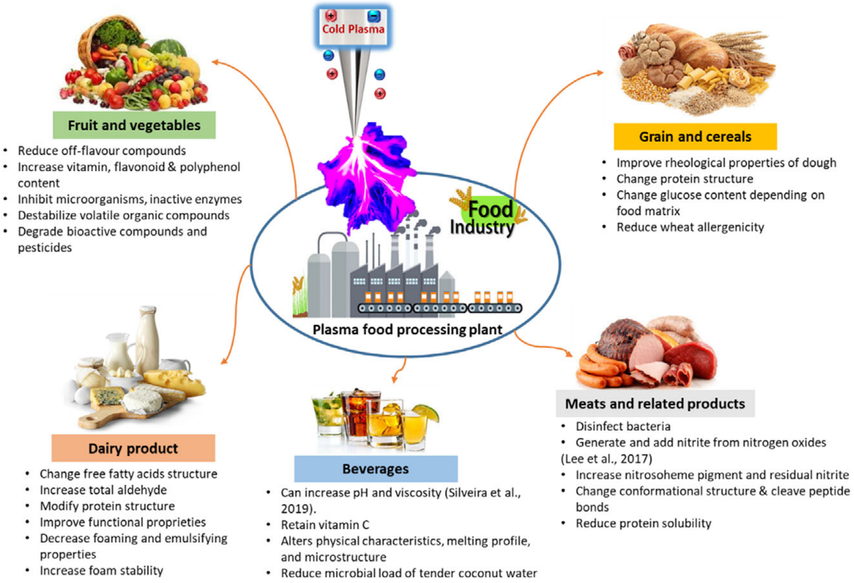

FIGURE 3 Application and effects of cold plasma (CP) on various food and beverage products

The following sections analyze the specific effects of CP treatment on biomolecules and organoleptic properties of foods.

4.1 | Effects of CP

4.1.1 | Organoleptic impact

The impact of CP on the organoleptic properties of foods has been identified through sensory trials and analytical measurements (color, pH, texture, rheology, and moisture content).

During a 12-panel sensory trial, Puligundla et al. (2017) found a significant reduction in appearance, flavor, taste, and overall acceptance of rapeseed sprouts after 3 min of 20 kV air-based corona discharge CP treatment, but this was not significant at 2 min. Song et al. (2015) also found that microwave plasma treatment with high power (0.9 kW at 2.45 GHz) reduces the sensory appeal (flavor, brittleness, and appearance) of lettuce after 10 min. However, this reduction was not statistically significant ($p > 0.05$) (Song et al., 2015). Schnabel et al. (2015) also found no change in texture or appearance of apples, strawberries, lamb's lettuce, and carrots after 10 min with a microwave CP treatment (1.1 kW at 2.45 GHz); although, no statistical tests were carried out in this study. Twenty sensory panelists, from a study by Jahid et al. (2014) stated that the lettuce, treated for 15 min with high energy deep UV-light induced CP, rated 4 on the hedonic scale ("dislike slightly"); however, the original value was not given in the paper and the change was stated in this paper as acceptable. Further details on parameters and quality and sensory changes can be found in Table 1 and Table S2.

The impact of CP treatment on the color of foods has been widely explored, although Baier et al. (2015) suggested that the impact of CP on this color change (ΔE) is heavily dependent on the plasma conditions. Furthermore, Baier et al. (2014) found that the gases used, the distance between source and sample, and the exposure time are all important controls for color change (Figure 4a). Color change is typically measured by the CIELAB scale, where L^* is the luminosity or lightness, a^* is the green (−) to red (+) scale, and b^* is the blue (−) to yellow (+) scale (Almeida et al., 2015; Lacombe et al., 2015).

Kovačević et al. (2016) observed a significant reduction in total color with increased gas flow (0.75–1.25 L/min) in the single electrode AP CP jet (2.5 kV at 25 kHz), and a significant decrease in L^*, a^*, and b^* when CP treated. However, the treatment time and sample volume had no significant impact on this increase (Kovačević et al., 2016). Lacombe et al. (2015) has attributed this decrease in color to the free radical damage of ozone, produced by some CP, on the chromophores.

Lacombe et al. (2015) also reported that the L^* value of blueberries was significantly reduced after 120 s of gliding plasma arc CP (549 W at 47 kHz); this was attributed to the heat melting the cutaneous wax cuticle layer of the blueberries. This was contradictory to the findings by Almeida et al. (2015) (DBD CP using 70 kV at 50 Hz for 60 s) and Chaiwat et al. (2016) (unknown CP at 60 W for 0.3–1.0 s), who observed a significant increase in L^* after CP treatment of orange juice (Figure 4b) and tapioca starch, respectively. These studies, however, had many different variables, gases, pressures, plasma sources (gliding plasma arc, DBD, and "unknown"), etc., which can potentially change the outcome considerably (Table S1). This was also seen by Baier et al. (2014), who found, after an unknown CP

TABLE 1 Shelf-life, pH, titratable acidity, moisture content, firmness, and cooking and drying time changes in foods treated with cold plasma (CP) ($n = 31$) "(n.d)" refers to no data

Food	Power and gas source	Power (Hz; V; W)	Distance (cm)	Time (min)	Quality changes	Source
Milk	AC corona dischargeplasma jet; air (no flow data)	(n.d); 9 kV; (n.d)	(n.d)	3.0–20	No significant change in the pH of the milk.	Gurol et al. (2012)
Tomato (cherry)	Corona discharge plasma jet; air (1500 L/min)	20 kHz; 8 kV; (n.d.)	6.0	0.0–2.0	Tomato shelf life (based on retained hardness, radical scavenging activity and lycopene content) was significantly increased from the control. Firmness decreased with increasing power, but this was not observed under intermittent mode.	T. Lee et al. (2018)
Rapeseed seeds	AC corona dischargeplasma jet; air (2 L/min)	58 kHz; 20 kV; (n.d.)	2.5	1.0–3.0	There was no significant change in moisture content.	Puligundla et al. (2017)
Prebiotic orange juice	Dielectric barrier discharge (DBD); air (no flow)	50 Hz; 70 kV; (n.d.)	2.0	0.25-1.0	Significant decrease (pH 4.3–3.9) in the pH of orange juice.	Almeida et al. (2015)
Zein powder		(n.d); 75 V; 75 W	0.8	1.0–10.0	Significant reduction in pH (6.05–5.82) with increasing exposure	Dong et al. (2017)
Peanut protein		(n.d); 35 V; (n.d)	0.8	0.0–4.0	Significant reduction in pH (6.92–6.80) with increasing treatment time.	Ji et al. (2018)
Rice flour		50 Hz; 60–70 kV; (n.d.)	(n.d)	5.0–10.0	Significant increase in pH for short (6.52–6.63) and long (6.70–6.77) grain rice after 70 kV and 10 min.	Pal et al. (2016)
Whey protein (isolate)		50 Hz; 70 kV; (n.d.)	(n.d)	1.0–60.0	Observed a significant reduction in pH (from 6.8 to 6.2) of whey isolate.	Segat et al. (2015)
Radish paocai		50 Hz; 60 kV; (n.d.)	4.0	1.0–3.0	No significant change in pH with CP treatment compared to the control and pasteurization.	Zhao et al. (2020)
Shrimp (Pacific white)	DBD; argon/Oxygen (80:20); argon/air (80:20) (no flow)	50 Hz; 120 kV; (n.d.)	2.0	10.0	The pH increased significantly less in all CP treatments compared to the control. As determined by two different CP treatments (Ar/Air and Ar/O_2) and two differently processed shrimp, across storage days 9–15.	Shiekh and Benjakul (2020)
Kiwi fruit (minimally processed)	DC DBD; air (no flow)	(n.d); 15 kV; (n.d)	7.0	0.0–20.0(each side)	Titratable acidity significantly reduced (17.2–14.0) only after 10 min of treatment on both sides on the 4th day of storage. But this was not observed on any other day nor after 20 min either side on any day.	Ramazzina et al. (2015)

(Continues)

TABLE 1 (Continued)

Food	Power and gas source	Power (Hz; V; W)	Distance (cm)	Time (min)	Quality changes	Source
NaCl enriched tryptone soya broth	DBD; helium (4 L/min) and Oxygen (40 ml/min)	15 kHz; 7 kV; 7.45 W	0.8	5.0	No significant reduction in pH.	Smet et al. (2019)
Apple (fresh cut)	DBD; air (0.8 m/s)	12.7 kHz; 15 kV; (n.d.)	9.0	0.0–30.0	No significant change in titratable acidity.	Tappi et al. (2014)
Melon (fresh cut)	DBD; air (7 × 10ŕş mş/s)	12.5 kHz; 15 kV; (n.d.)	7.0	15–30(each side)	No significant change in titratable acidity.	Tappi et al. (2016)
Pasteurized orange juice	DBD; air or modified air (65% O_2, 30% N_2 + 5% CO_2)	60 Hz; 90 kV; (n.d.)	4.4	0.5–2.0	No significant reduction in pH.	Xu et al. (2017)
Water (distilled)	DC high voltage pulsed plasma gas discharge (124 pulse/s); N_2, O_2, CO_2, air (10 L/min)	(n.d); 23.5 kV; (n.d)	(n.d.)	0.0–0.5	Increased in acidity from pH (6.03–3.90)	Rowan et al. (2007)
Pears, cucumber, and carrots	DC high voltage current; air	(n.d); 500 V; 15 W	1.0	0.00–0.06	Reduction in moisture content of 5% for all three samples. However, no statistics were carried out.	R. X. Wang et al. (2012)
Bacon (sliced)	Radio frequency; helium (10 L/min) and He (9.99 L/min) + O2 (10 ml/min)	13.56 MHz; (n.d.); 75–125 W	0.3	0.0–1.5	No change in pH regardless of changing exposure time, power or gas compositions. Moisture evaporation was observed	Kim et al. (2011)
Rice (Chinese milled)	Radio frequency; helium (no flow data)	13.56 MHz; (n.d.); 80–520 W	3.0	0.0–2.0	Significant reduction in cooking time with increasing treatment time, but not with increasing power. No significant change in water absorption ratio with increasing treatment time or power. Significant reduction in moisture content after 80 W and 20 s but further treatment time and power increased moisture content.	Liu et al. (2021)
Lettuce	Cold plasma[Note:]; nitrogen (no flow data)	(n.d); (n.d); 300–400 W	(n.d)	0.5–3.0	No significant differences in texture.	Cui et al. (2016)
Starch (Tapioca)	Plasma jet[Note:]; argon (1 L/min)	600 MHz; (n.d.); 50–100 W	(n.d)	5.0	For granular starch there was a significant reduction in starch clarity at 50 W followed by a significant increase at 100 W. For cooked starch 50 W and 100 W led to a significant increase in and decrease in starch clarity.	Wongsagonsup et al. (2014)

(Continues)

TABLE 1 (Continued)

Food	Power and gas source	Power (Hz; V; W)	Distance (cm)	Time (min)	Quality changes	Source
Grapes	Gliding plasma AC electrode arc pulses; air (10 L/min)	50 Hz; 27 kV; 300 W	3.5	0.0–1.0	Significant reduction in drying time with increasing exposure. Significant increase in effective moisture diffusivity with increasing exposure up to 50 s. Significant increase in rehydration ratio.	Ashtiani et al. (2020)
Blueberries	Gliding plasma AC electrode arc pulses; air (113 L/min)	47 kHz; (n.d.); 549 W	7.5	0.0–2.0	Significant reductions in firmness after 60 s.	Lacombe et al. (2015)
Saffron	Gliding plasma AC electrode arc pulses; air (1.5 m/s)	50 Hz; 8 kV; 1 kW	(n.d)	0.0–1.0	Significant reduction in drying time (37–20 min) with increasing CP treatment time (0–60 s).	Tabibian et al. (2020)
Tomatoes (juice)	Gliding plasma AC electrode arc discharge	50 Hz; 3.8 kV; 40 W	4.0 (vortex)	0.0–5.0	Significant increase in pH (~4–~4.5) after CP treatment. This was not affected by treatment time or storage time.	Starek et al. (2020)
Apple (freeze dried (FD)) and Potato (FD)	Microwave energy (5/7s on/off Pulsed); air (20L/min)	2.45 GHz; (n.d.); 1.2 kW	1.0	2.5–10.0	Observed a significant decrease in surface pH after 2.5 min of treatment of both fresh apples (3.9–1.8) and potatoes (5.9–1.9) and this decreased with treatment time.	Bußler et al. (2017)
Black pepper seeds, allspice berries, and juniper berries	Microwave plasma; argon (20 L/min)	2.45 GHz; (n.d.); 600 W	1.0	0.0–1.0	Significant reduction in water activity for all spices with increasing treatment time.	Wiktor et al. (2020)
Strawberries (inoculated)	Indirect (PAW) single electrode plasma jet; argon (98%) and oxygen (2%) (5 L/min)	10 kHz; 18kV; (n.d.)	2.0	10.0–20.0	No significant change in pH was observed. No significant difference in firmness with increasing treatment time.	Ma et al. (2015)
Gellan gum	KinPen 09 DC Plasma jet; Argon (99.999%; 99.99%; 99.95%; 99.9%) and oxygen (0; 0.01; 0.05; 0.1). 5 L/min	1.1 MHz; 65kV; (n.d.)	1.0	0.0–6.0	No significant change in pH during treatment.	Surowsky et al. (2013)
Starch (Tapioca)	Semi continuous plasma downer reactor (PDR); argon (99.999%) (no flow rate)	(n.d); (n.d); 60 W	(n.d)	30.0–90.0	Statistically significant reduction in moisture content (~13.5–10.2 MC%)	Chaiwat et al. (2016)
Apples (fresh cut)	Surface barrier discharge; air (no flow data)	9 kHz; 5.0 kV; 50 W	(n.d)	0.0–2.0	Reduction in weight loss and browning over storage time (6 days) for the refrigerated CP treated sample (120 s).	Zhou et al. (2020)

Note: "(n.d)" refers to no data.

[1]Sources that have not been categorized by the authors.

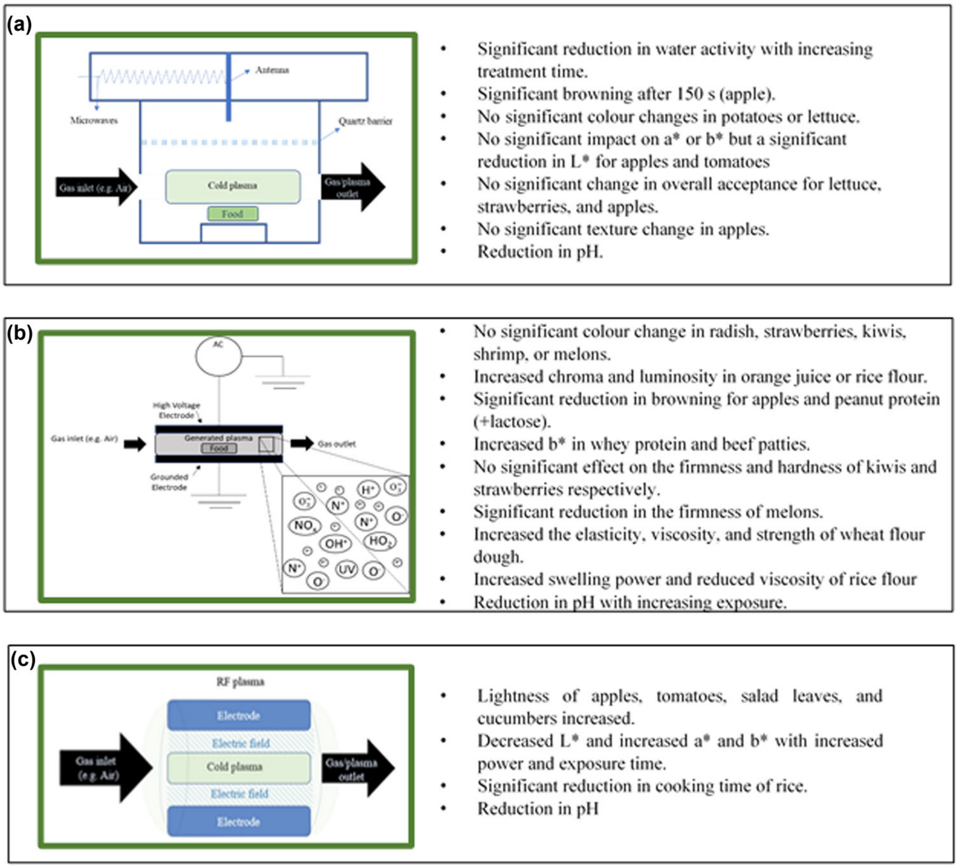

FIGURE 4 Summary of organoleptic changes in foods treated with microwave plasma (A), dielectric barrier discharge (DBD) cold plasma (CP) (B), and radio frequency (RF) plasma (C), from the following studies: Almeida et al. (2015), Baier et al. (2014), Bußler et al. (2017), Butscher et al. (2016), Kim et al. (2011), Liu et al. (2021), Misra et al. (2014, 2015), Pal et al. (2016), Ramazzina et al. (2015), Schnabel et al. (2015), Shiekh and Benjakul (2020); Segat et al. (2015), Smet et al. (2019), Song et al. (2015), Tappi et al. (2014, 2016), X. Wang et al. (2021), Wiktor et al. (2020), Yu et al. (2020), and Zhao et al. (2020)

jet treatment (assumed to be RF based on the stated frequency) of 2–6 kV at 1.1 MHz for 120 s, that the L^* of lettuce leaves increased in 65% O_2, 16% N_2, and 19% CO_2 conditions, but decreased with 90% N_2 and 10% O_2 plasma. (Figure 4c). This difference was also observed for the a^* scale, where Chaiwat et al. (2016) observed a reduction in a^*, and Lacombe et al. (2015) observed an increase in a^*, again for tapioca starch and blueberries, respectively. This difference was not observed for b^*: Chaiwat et al. (2016), Lacombe et al. (2015), and Segat et al. (2015) (who applied DBD to whey protein isolate at 70 kV and 50 Hz for up to 60 min) all observed an increase in b^*. Despite this, more studies stated no significant color change than those that did. Bermúdez-Aguirre et al. (2013), Cui et al. (2016), Gurol et al. (2012), Ma et al. (2015), Misra et al. (2014), Ramazzina et al. (2015), Song et al. (2015), and Tappi et al. (2016) all observed no significant color change (Figure 4). Although, in the study by Cui et al. (2016), the plasma source type, plasma source to sample distance, and the temperature were all omitted.

CP appears to reduce the pH of foods when treated with DBD (in orange juice, zein powder, peanut protein, whey protein isolate, and shrimp), microwave plasma (in apples and potatoes), and DC high voltage CP (water) (Table 1) (Almeida et al., 2015; Bußler et al., 2017; Dong et al., 2017; Ji et al., 2018; Rowan et al., 2007; Segat et al., 2015; Shiekh & Benjakul, 2020; Smet et al., 2019; Tappi et al., 2016). However, no significant changes in pH and titratable acidity have also been found in foods treated with corona discharge (milk) (Gurol et al., 2012), DBD (radish, soya broth, apple, melon, and pasteurized orange juice) (Zhao et al., 2020; Smet et al., 2019; Tappi et al., 2014, 2016; Xu et al., 2017), RF (bacon) (Kim et al., 2011), plasma activated water (strawberries) (Ma et al., 2015), and the kINPen 09 plasma jet (Gellan gum) (Surowsky et al., 2013). Furthermore, only the study by Starek et al. (2020) and Pal et al. (2016) was found to have seen a significant increase in pH following CP treatments. This indicates that the effects of CP on the pH of foods are dependent on the foods, considering the similarities in the CP treatments by Pal et al. (2016)

TABLE 2 Effects of varying some of the cold plasma (CP) treatment parameters on different food

Plasma source	Food	Parameter varied	Impact	Source
Corona discharge plasma jet (CD)	Tomatoes	Increased Amps (2–4 Amp)	Reduced firmness	T. Lee et al. (2018)
	Rapeseed	Increased time (1–3 min)	Significant reduction in sensory characteristics after 3 min	Puligundla et al. (2017)
Dielectric barrier discharge (DBD)	Corn	Increased relative humidity of input gas	Reduced O_1 in plasma (which could change the impact on the food)	Shi et al. (2017)
	Shrimp	Gas composition (Ar/O_2 to Ar/air)	Reduction in overall sensory acceptability from Ar/O_2 plasma, but not from Ar/Air plasma.	Shiekh and Benjakul (2020)
	Peanut protein	Increased treatment time	Significant reduction in pH	Ji et al. (2018)
Radio frequency (RF)	Sliced bacon	Increased power (75, 100, and125 W) and increased time (0, 60, and 90 s)	Reduced lightness and increased the a* and b* values	Kim et al. (2011)
	Rice (Chinese milled)	Increased treatment time (0–2 min)	Significant reduction in cooking time	Liu et al. (2021)
Gliding arc plasma	Blueberries	Increased time	Significant reduction in firmness, lightness, and anthocyanins	Lacombe et al. (2015)
Unknown*	Lettuce	Smaller plasma diameter	Increased output intensity	Baier et al. (2014)
		Smaller plasma source to sample distance (1–1.7 cm)	Significant damage to plant cells (at 1 cm)	

Note: *Based on the use of 1.1 MHz frequency, this was assumed to be an RF based CP.

and Segat et al. (2015) and their opposing effects on rice flour and whey protein, respectively. However, Pan et al. (2019) suggested that variation in reactive species output between CP technologies could explain changes in the pH, conductivity, and redox potential of foods. Further details on parameters and pH and color changes can be found in Table 1 and Table S3, respectively.

Furthermore, CP appears to have also changed textural properties of foods. Tappi et al. (2016) and Lacombe et al. (2015) found a significant reduction in the firmness of tomatoes (using DBD plasma at 15 kV and 12.5 kHz for 30 min each side) (Figure 4b) and blueberries (using gliding arc plasma), respectively. Misra et al. (2014) used DBD plasma for a much shorter duration (10 min) than Tappi et al. (2016) at a higher power (70 kV) and a considerably lower frequency (50 Hz) found no significant effect on the firmness of the strawberries (Figure 4b). Furthermore, it is reasonable to expect that the reduction in DBD exposure influenced this change in firmness, since Lacombe et al. (2015) found that increased duration of gliding plasma arc exposure led to a reduction in firmness of blueberries (Table 2). However, a more controlled study is needed to identify any changes in firmness with changing exposure time of CPs from DBD, corona discharge, radio frequency, and so on.

Different CP treatments, including an RF surface barrier discharge CP (20 V at 9 kHz for 120 s) (Bahrami et al., 2016), semicontinuous plasma downer reactor (60 W for 1 hr 30 min) (Chaiwat et al., 2016), and a DBD CP (70 kV at 50 Hz for 10 min) (Misra et al., 2015) have all been found to increase the gel strength of different dough. Based on Fourier transform infrared (FTIR) and rheological measurements, Chaiwat et al. (2016) suggested this could be caused by starch cross-linking. Pal et al. (2016) found a significant increase in wheat dough swelling power with increasing time (0, 5, and 10 min) and power (60 kV to 70 kV) of CP, meaning that these parameters are also likely to have a big impact on the final food product. It would therefore be interesting to explore the other impacts from parameters such as gas composition and flow rate.

Baier et al. (2015) was the only study found to have looked at the effects of plasma treatment on aroma, and they found a reduction in carrot aroma after indirect microwave plasma treatment. However, based on the substantially variable input parameters for any CP treatment, it is unlikely this study would be representative of all CP treatments.

Additionally, T. Lee et al. (2018) found that corona discharge (CD) CP treatment (8 kV at 20 kHz for 120 s with 10 s on/off plasma) significantly increased the shelf-life (based on less reduction in firmness, DPPH radical scavenging activity, and lycopene content) of tomatoes. Shiekh and Benjakul (2020) found that DBD CP (120 kV at 50 Hz for 10 min) reduces the increase in pH with increasing storage time, which could have a positive impact on the shelf-life quality of shrimp. They also found that the argon and air (80:20) combination significantly lessened the reduction in color, flavor, taste, texture, and overall acceptability compared to the argon and oxygen (80:20) combination over 15 days. Zhou et al. (2020) have found a reduction in the weight loss and browning over 6 days of storage after 2 min of surface barrier discharge plasma (5 kV at 9 kHz), thereby also improving shelf life quality. Furthermore, Starek et al. (2020) found a significant reduction in carotenoids and ascorbic acid in tomato juice after 7 days storage, which was not observed with the Gliding arc discharge plasma treatment (3.8 kV at 50 Hz for 5 mi). However, Zhao et al. (2020) found that there was a significant increase in nitrile content after 0 and 7 days of storage with CP treatment compared to pasteurization and the control, which the authors suggested may have a negative impact on the sensorial attributes.

4.1.2 | Biomolecular modification

Effects on functional food components are also highly dependent on the plasma equipment and configuration (Muhammad, Liao, et al., 2018). The effects of CP could generate many changes in surface proteins, carbohydrates, and lipids.

Bahrami et al. (2016) found no significant change in total protein content, although there was a significant increase in high molecular weight proteins with increased power (from 40 to 90 W at 9 kHz for 120 s) on the RF surface barrier discharge CP. This was also observed by Takai et al. (2012) who CP (unknown source) treated (–3.5–5 kV at 13.9 kHz) lysozyme. A few possible explanations for increased molecular weight proteins have been the following: Increased protein carbonyl groups due to protein oxidation from the CP (Segat et al., 2015); unfolding of the protein structure (due to SH bond breaking) exposed aromatic amino acids to sugar's carbonyl groups, hence increasing the weight through glycosylation (Yu et al., 2020). Moreover, the secondary structures of proteins have been observed to be modified by CP (Muhammad, Xiang, et al., 2018). After 5 min of DBD CP treatment (70 kV at 50 Hz), Misra et al. (2015) found a reduced amount of apparent α-sheets and increased α-turns and α-helices; however, apparent α-sheets appeared to increase with extended plasma exposure (from 5 to 10 min). This could explain why Surowsky et al. (2013) also found that the kINPen 09 DC CP jet treatments (65 kV at 1.1 MHz for 6 min) increased α-sheet content, although they found that it reduced α-helical content.

It is likely that these changes are caused indirectly by changes in the primary structure and the amino acid composition. For example, Dong et al. (2017) observed an increase in free SH groups with DBD CP at 75 V (and 75 W for 10 min). However, Segat et al. (2015) and Ji et al. (2018) observed a reduction in SH groups with DBD CP (at 75 kV and 50 Hz for 60 min, and 0–3 min of DBD at 35 V, respectively); this effect was found to increase with treatment time (Ji et al., 2018). Segat et al. (2015) also found that there were significantly more protein-based carbonyl groups after cold plasma treatment. Furthermore, Pal et al. (2016) found a statistically significant reduction in both basic and acidic amino acid contents in rice flour after 10 min of DBD CP treatment (70 kV at 50 Hz). The effect of which was greater with both increased time and power. The acidic and aromatic amino acid contents of some rice flours were also observed to increase with increasing power and exposure time (Pal et al., 2016). Takai et al (2012) found that the formation of HOO, O_2^-, and NO from 30 min of CP (of an undeclared source) treatment (–3 – +5 kV AC at 13.9 kHz) increased the molecular weight of lysozyme and reduced the apparent fluorescence of a tryptophan side chain, which suggested it could alter the chemistry of side chain amino acids. Hence, more research should be carried out to determine interaction reactions of different amino acids and CP. Furthermore, these changes have been attributed to an increase in the oxidation of proteins (Segat et al., 2015; Zhao et al., 2020). Zhou et al. (2020) observed an increase in C–O and C=O groups and a decrease in C–C and C–H groups on the surface of apples after surface barrier discharge treatment (5 kV at 9 kHz for 2 min).

Although this does provide evidence for some secondary structure modifications with the CP treatment, there is limited evidence of the mechanisms and chemical interactions of CP with proteins (Misra, Pankaj, et al., 2016). More research is still required on the mechanisms behind the effects of CP on protein and lipid oxidation as well as the moderate effects on lipolysis, protein denaturation, and protein aggregation (Pérez-Andrés et al., 2018). Further details on parameters and protein changes can be found in Table S5.

Almeida et al. (2015) also found a reduction in the number of oligosaccharides, with four to seven glycosidically bound saccharide monomers (four to seven degrees of polymerization (DP)), with increasing treatment time (15–60 s) of direct and indirect DBD CP (70 kV at 50 Hz). The same was true for CP treatment of fructose. However, CP teatment increased the concentration of sucrose as well as carbohydrates with a DP of 3. There was also a large reduction in oligosaccharides after indirect CP treatment compared to both direct CP treatment and no CP treatment (Almeida et al., 2015). It was suggested that this could be caused by the breakage of glycosidic bonds through the interaction with the reactive oxygen species produced by the plasma (Almeida et al., 2015). However, Rashid et al. (2020) found a reduction in carboxylic acid groups on saccharides after 30 min of DBD CP treatment (80 kV), which was claimed to indicate a lack of CP-induced oxidation. It is therefore important that the mechanisms behind this decrease in oligosaccharides should be analyzed further (Almeida et al., 2015).

A significant increase in soluble solids content (°Brix) was observed by Tappi et al. (2014), after 10 min of DBD CP treatment (15 kV at 12.7 kHz) of a cut apple. However, Tappi et al. (2016) found no significant difference in the soluble solids content of melon after 30 min of DBD CP treatment (15 kV at 12.5 kHz). Puligundla et al. (2017) observed no significant differences in the reducing sugar content of rapeseeds after 3 min of AC corona discharge CP jet (20 kV at 58 kHz), and Almeida et al. (2015) found glucose content to be unaffected by 60 s of DBD CP treatment (70 kV at 50 Hz 2 cm away from the sample). The similarities in the CP processing methods of Tappi et al. (2014) and Tappi et al. (2016) suggest that the availability of the sugars and carbohydrates on the surface of the food are likely to impact the effects of DBD CP.

Glycosidic bonds can be formed by cross linking hydroxyl groups with aldehyde groups of sugars (Wongsagonsup et al., 2014). This process was found by Wongsagonsup et al. (2014) to increase after 5 min of an argon-based CP treatment (100 W at 600 MHz). This agrees with Muhammad, Xiang, et al. (2018), who suggested surface etching CP can modify starches by depolymerization and crosslinking. Moreover, Pankaj et al. (2015) found an increase in starch surface hydrophilicity and roughness after 5 min of DBD CP (80 kV at 50 Hz). The impacts of CP treatment on carbohydrates, therefore, may be even more complex than just removing glycosidic bonds. Further details on parameters and carbohydrate changes can be found in Table S6.

Yu et al. (2020) found that the use of DBD CP treatment (90 W for 3 min) as a co-glycosylation mechanism for lactose and peanut proteins, improved the solubility of the protein (from 1.01 to 1.34 mg/ml) isolate by increasing the glycosylation reaction. Delaux et al. (2016) has also found that DBD CP treatment (15 W for 7–30 min) can induce polymerization of carbohydrates mostly through glycosylation.

CP has affected starch and protein modification, but it has deteriorated the quality of lipid-based foods because of lipid oxidation (Muhammad, Xiang, et al., 2018). Furthermore, RF CP (300 W at 13.56 MHz for 13 min 20 s) has also been found to partially oxidize the wax cuticle (lipid based) layer of plants, causing the formation of carboxylic acid and aldehyde groups on the surface (M. Zhang et al., 2013). This oxidation by CP has been observed in numerous foods, and therefore should be avoided in highly lipidic food products (Gavahian et al., 2018). Although, excluding oxygen from the gas as well as reducing power and exposure time could reduce this oxidation (Gavahian et al., 2018). Bahrami et al. (2016) also found a reduction of fatty acids and phospholipid contents. However, the effectiveness of this was proportionally dependent on treatment time and power. Further details on parameters and carbohydrate and lipid changes can be found in Table S6and S7, respectively. Other variations of the CP treatment can include equilibration time (plasma running time prior to sample treatment), gas composition, gas flow, gas pressure, pulse or wavelength frequency, voltage, wattage, sample to source distance, exposure time, and treatment container (Jahid et al., 2014; López et al., 2019). These multiple variations have led to a plethora of different possible CP treatments. However, a substantial number of these treatments have not been appropriately labelled or described in many of the reviewed studies, but instead are ambiguous, for example, “plasma jet,” “pulsed plasma,” and “atmospheric cold plasma.” with little to no further information given (Table S1) (Pankaj et al., 2018; Pan et al., 2019).

The cases where the study has recorded the energy inputs, it could be possible to identify what CP treatment it was likely to be. For instance, in the RF plasmas are generated with a frequency of ~10 MHz to 25 MHz and the RF KinPen is around 1 MHz; it is likely, therefore, that the treatment described as “atmospheric pressure plasma” with a frequency of 1.1 MHz, used RF as its plasma source.

However, further research should be as descriptive as possible with these treatments when the variables are so vast, to avoid this type of ambiguity. The same is true for the distance between plasma source and sample, and plasma temperature. Exposure time was the only parameter that was consistently recorded throughout all treatments (Table S1). The variable increase in temperature of CP, shown in Table S1, indicates that although CP is relatively cold, there should still be more monitoring and control to ensure it is kept as a nonthermal treatment.

Expanding on the suggestions by López et al. (2019), further research should include the following to improve clarity and understanding: type of equipment, plasma source to sample distance, gas composition, plasma composition, exposure time, sample temperature change, and detailed food description. There are substantial and significant impacts of varying some of these CP treatment parameters on the food and the plasma output (Table 2). It is, therefore, of vital importance that these are recorded and reported clearly and accurately. This will avoid ambiguity and allow for the easy combination of multiple studies.

Further details on parameters, quality, and biomolecular changes from Table 2 can be found in Tables S1-S7.

We therefore recommend that the following parameters are always recorded when presenting data of biomaterial treatment using plasma:

- Gas composition/humidity/temperature/flow rate.
- Electrode configuration / composition / voltage / power/ reactor design.
- Equipment model number, supplier, and any modifications.
- Plasma composition and delivery top sample mechanism.
- Sample: sample shape and surface topology, pH (where applicable), storage conditions, and moisture content.
- Sample tray material, distance to sample and headspace of exposed vessel and containment method (e.g., positive pressure air valve).
- Duration of exposure, initial temperature, and temperature change over exposure.

5 | CONCLUSION

This review has shown that CP can be used to increase quality-based shelf life, gel strength, and that using lower power and shorter periods of exposure of the CP is key in minimizing negative organoleptic impacts. Furthermore, regardless of parameters and treatments types cold, plasma has been observed to significantly reduce the pH of the treated foods. This is, therefore, an important impact to consider when using this technology. CP does not significantly impact color, and although some have found that it reduces L^* and increases b^*, these were heavily dependent on parameters such as gas flow.

Increased time and power of CP exposure appear to increase acidic, aromatic, and basic amino acids. The CP treatments also appear to have increased the proportion of α-pleated sheets. These would therefore significantly affect foods dependent on active enzymes. CP reduces larger carbohydrates and increases cross linking and glycosylation reactions. Although, only one study has been found to have researched CP for co-glycosylation of proteins, there would be numerous benefits to being able to control the glycosylation mechanisms. It was also found from this review that the majority of CP treatments show an increase in lipid oxidation and subsequent rancidity of highly lipidic foods (e.g., oil, butter, and margarine). It would therefore be prudent that CP treatments are avoided on these highly lipidic foods.

This review has identified substantial gaps for more original research into the organoleptic impact of CP (e.g., quality-based shelf life studies, analytical flavor analysis, and statistically backed sensory studies), and its biomolecular impact (e.g., co-glycosylation research) as well as the interaction mechanisms between the reactive oxygen species output, the biomolecular modification and its subsequent effects on the organoleptic properties. We have therefore proposed key physical and chemical parameters that should be considered for inclusion in all future publications in the field.

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AUTHOR CONTRIBUTIONS

George Warne: Investigation; Methodology; Writing-original draft. Philip Williams: Conceptualization; Supervision; Writing-review & editing. Hue Pho: Investigation; Methodology; Writing-original draft. Nam Tran: Investigation; Methodology; Writing-original draft. Volker Hessel: Supervision; Writing-review & editing. Ian Fisk: Conceptualization; Supervision; Writing-review & editing.

CONFLICTS OF INTEREST

The authors declare no conflict of interest.

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SUPPORTING INFORMATION

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