Food and Bioprocess Technology (2018) 11:463–486
https://doi.org/10.1007/s11947-017-2042-9

REVIEW

Understanding the Impact of Nonthermal Plasma on Food Constituents and Microstructure—A Review

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Received: 11 May 2017 / Accepted: 11 December 2017 / Published online: 18 January 2018

Abstract
Nonthermal plasma (NTP) is superior to thermal technologies as a technique that provides a satisfactory microbial safety and maintains reasonable standards in food quality attributes. Currently, the effects of NTP on some food components is regarded as beneficial, such as effects on starch and protein modification. For other food components, such as lipid oxidation, NTP is regarded as an undesirable treatment because it leads to quality deterioration and formation of off-flavor. An overview of the basic principles of NTP and food microstructure in relation to NTP-treated food and the underlying mechanisms are discussed. The review further highlights the latest research on plasma application in food and the related impact on food matrices. Efforts were made to outline the research findings in terms of NTP application on foods with an emphasis on the impacts on the food microstructure and their related qualities. In this review, the industrial capacity of NTP to improve the functional properties of starch, proteins, and lipids as well as provide little or no alteration in food quality compared to other technologies are emphasized. Some oxidative breakdown in relation to starch, proteins, and lipids are discussed and documented in this paper as a review of representative available publications.

Keywords Nonthermal plasma · Food microstructure · pH · Starch · Protein · Lipid

Introduction

The consumer demand for food products that are healthy with minimum quality alteration and sensible cost has become the norm. Processing of food to alter its composition for health concerns (such as fat reduction) presents challenges in satisfying consumer acceptability (Pascua et al. 2013). The presence of scattered elements in food, such as fat globules and air bubbles in food, or the presence of proteins and carbohydrates that are thermodynamically incompatible, can make food products unstable. However, some food products may become denatured as the food macromolecules and quality are impaired via conventional thermal pasteurization and sterilization (Jeantet et al. 2016). Other techniques may even damage the food structure or alter the food quality parameters, such as color, taste, and aroma, thereby decreasing the valuable nutritive contents (Scholtz et al. 2015) or forming toxins within the food products, especially when chemical solutions are used as disinfectants (Thirumdas et al. 2014). Limitations associated with conventional thermal technologies have created much interest in nonthermal technologies (Stoica et al. 2013) such as irradiation (Cuppett et al. 2000), ultrasound (Abid et al. 2013; Ferrario and Guerrero 2016; Li and Farid 2016), pulsed light (Nicorescu et al. 2013), high hydrostatic pressure (Chen et al. 2015; Yu et al. 2013), ozone (Khadre and Yousef 2001; Pandiselvam et al. 2014, Pandiselvam et al. 2017a; Pandiselvam and Thirupathi 2015), electrolyzed water (Ding et al. 2015, 2016; Xuan et al. 2017), and nonthermal plasma (NTP) (Almeida et al. 2015; Bermúdez-Aguirre et al.

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2013; Fernández et al. 2012; Lee et al. 2016; Liao et al. 2017a; Smet et al. 2016; Yu et al. 2013; Zou et al. 2004). Ozone is a - reactive species and is separately regarded as a nonthermal technique for decontamination. Its generation by corona discharge is similar to NTP (Pandiselvam et al. 2017b). These alternative technologies may only remove some of the earlier mentioned shortcomings. NTPs have wide application in food industry, such as food sterilization, modification, or enhancement of food the matrix/structure (Gurol et al. 2012; Misra et al. 2015; Sarangapani et al. 2016). NTP's advantages are connected to its ability to work within low temperature ranges without causing significant changes to heat-sensitive food materials (Surowsky et al. 2013, 2014; Thirumdas et al. 2014; Gurol et al. 2012; Niemira 2012; Laroussi and Leipold 2004), minimizing nutrient and sensory property degradation (Liao et al. 2017b; Stoica et al. 2013). Regardless of its suitability for heat-sensitive food materials, its potent effect on resistant microorganisms has been documented in various articles. Precisely, log reduction of 3.1 in *Bacillus atrophaeus* and 2.4 in *Bacillus subtilis* spores were recorded with pure argon gas plasma (Reineke et al. 2015). Others observed log reductions of 4.1, 2.4, and 2.8 log with *Salmonella enterica*, *B. subtilis* spores and *B. atrophaeus* spores, respectively, after 30 min of plasma treatment (Hertwig et al. 2015). NTP inactivation of pathogens and food microorganism, including spores have been extensively reviewed elsewhere and is not covered here (Surowsky et al. 2014; Thirumdas et al. 2014; Liao et al. 2017b; Niemira 2012; Scholtz et al. 2015; Stoica et al. 2013). NTP and ozone are clean and environment-friendly green technologies. This trait may be due to their ability to improve the shelf life and safety of food products. The latter was given the status of generally recognized as safe (GRAS) in 2001 by the Food and Drug Administration (FDA) of the US government (Kim et al. 2003; Tzortzakis and Chrysargyris 2017; Tzortzakis et al. 2007). Likewise, to date, no research has been published with regard to the possible generation of toxic compounds or by-products after NTP treatment of food materials (Thirumdas et al. 2014). Other non-food industrial applications of plasma include materials processing to modify or treat paper, textiles, glasses, and electronics (Harry 2010; Niemira 2012), sterilization of medical instruments (Deng et al. 2007a, b; Harry 2010), treatment of water and exhaust fumes, material deposition and synthesis (Misra et al. 2016b), and recently, in three-dimensional (3-D) printing for tissue engineering for fabrication at the micron-scale to mimic the microstructure of natural tissues (Wang et al. 2016).

However, fewer or no reviews are available on the impact of NTP on food microstructure, despite recently published book reviews of NTP and general plasma applications. The aim of this paper was to review the recent research progress about the impact of NTP on food constituents and microstructure.

Nonthermal Plasma and its Generation

NTP, the fourth state of matter, is an ionized gas that contains a variety of active electrically charged particles, such as electrons, ions, radicals, metastable excited species, and vacuum ultraviolet radiation, which have sufficient energy to initiate chemical reactions (Rød et al. 2012; Mir et al. 2016; Han et al. 2016, Liao et al. 2017b). NTP can be generated by applying electrical or microwave energy to gases (atmospheric or synthetic air, oxygen, nitrogen, helium, hydrogen, argon) or combinations of gases (Harry 2010; Mir et al. 2016) that are either at low or atmospheric pressures. The discussion will focus on NTP generated at atmospheric pressure due to its advantages for the food industry and because it does not require extreme process conditions (Misra et al. 2011). Upon the application of an electrical field to the gas, several reactive species are generated during the collision of electrons, gas particles, and atoms. On the basis of thermodynamic equilibrium, elastic collisions lead to transfer and redistribution of a fraction of the kinetic energy to other particles. The energy stored in the free electrons and only the electrons temperature (T_e) reaches the higher values of 10^4 K, much higher than neutral ions temperature (T_n) nearly at room temperature ($T_{e>>}T_n$) and whole process gas temperature, T_g ($T_{e>>}T_g$), thus allowing the NTP to maintain a relatively low temperature conditions (Surowsky et al. 2014; Scholtz et al. 2015; Liao et al. 2017b; Harry 2010; Niemira 2012). On the other hand, the inelastic collisions transfer energy of more than 15 eV, thereby allowing various plasma-chemical reactions, such as excitation, dissociation, or ionization to occur (Surowsky et al. 2014). These processes lead to the generation of plasma reactive species (RS) such as: reactive oxygen species (ROS), reactive nitrogen species (RNS), charged particles, electrons, and UV/VUV photons (Surowsky et al. 2014; Liao et al. 2017b; Scholtz et al. 2015). The plasma RS are responsible for all plasma-induced inactivation of microorganisms, functional modifications and degradation in food macromolecules. More detailed plasma composition, generation, and types have been discussed extensively (Mir et al. 2016; Misra et al. 2016b; Harry 2010).

Nonthermal Plasma-Generating Devices

Several NTP-generating devices are in existence. The principles of generating the electrical discharges depend on the equipment configuration and are comprised of plasma jet, corona discharge, microwave discharge, radio frequency discharge, gliding arc discharge, and dielectric barrier discharge. The component configuration differs from one equipment to another, giving rise to different plasma terminologies: dielectric barrier grating discharge (DBGD) (Gallagher Jr et al. 2007), corona discharge (CD) (Joubert et al. 2013; Korachi et al. 2010), dielectric barrier discharges (DBD) (Chiang et al.

2010; Kostov et al. 2010; Pankaj et al. 2015), microwave-cold plasma (MCP) (Kim et al. 2017; Won et al. 2017), radio frequency plasma (RFP) (Hertwig et al. 2015), nanosecond pulse plasma (NPP) (Park et al. 2015), and gliding arc discharge (GAD) (Moreau et al. 2007). Some of the plasma discharges and equipment configurations are briefly discussed.

Corona Discharge

This type of discharge is achieved when an electrical field of high intensity is generated at atmospheric pressure. The increased local electric field forms a local nonuniform discharge only at a high nonuniform field. The energy generated results in corona plasma discharge flumes at the tip of pointed electrode (active electrode) or thin wire, as shown in Fig. 1. The active electrode is called as the active region of the corona discharge (Scholtz et al. 2015).

Dielectric Barrier Discharge

As the name implies, DBD is generated in between two electrodes separated by a dielectric barrier layer of either ceramic, quartz, polymer, or glass. DBD is one of the safest discharge methods because it avoids spark and arc discharges by limiting the current. Unlike corona discharge, DBD can work within a wider range of gas pressures (10^4–10^6 Pa). The DBD gap between the electrodes varies from 0.1 mm to several centimeters. Unlike other plasma discharges, the DBD configuration (Fig. 1) offers the flexibility of in-package sterilization as well as sterilization of food-packaging materials. DBD can handle AC or DC voltage supplies (Misra et al. 2016b).

Microwave Discharge

Microwave discharge is an electrodeless discharge. Electromagnetic waves are generated from a magnetron coupled to a cooling system through the process chamber. The excited discharge is subsequently absorbed by the process gas, resulting in inelastic collisions and ionization reactions. The intensity and density of the microwave generated are 0.25 W m^{-2} and 2.45 GHz, respectively, when operated at a power level of 50–1000 W. The resultant wave is then sent to the gas plasma treatment chamber where the food samples to be treated are placed (Won et al. 2017).

Radio Frequency Discharge

The radio frequency (RF) discharge equipment consists of RF generator, a ceramic nozzle with an RF voltage electrode and a gas supply system. The nozzle is coupled with two electrodes: a needle and grounded ring electrodes. Plasma discharge generation occurs at the tip of this electrode and extends outwards to the target with the aid of flowing gas. The intensity of the plasma plumes depends on the gas flow rate and the power applied (Hertwig et al. 2015).

Gliding Arc Discharge

Gliding arc discharge plasma is also an NTP characterized by a warmer discharge than corona and DBD discharges (Fridman et al. 1999). This plasma uses four electrodes as opposed to the conventional two electrodes used by other plasma discharges. The discharge is generated by two divergent aluminum electrodes connected to a plasma generator working at high voltage (up to 9 kV or higher). The other two electrodes are made from copper and are also connected to the plasma generator. The process gas inlet is via a nozzle at the top of reactor (Fig. 2). A plasma discharge arc is generated between these electrodes where the distance between them is at a minimum. The discharge generated is transported to the target surface by gas flow, and thus, the plasma inactivation is initiated (Moreau et al. 2007).

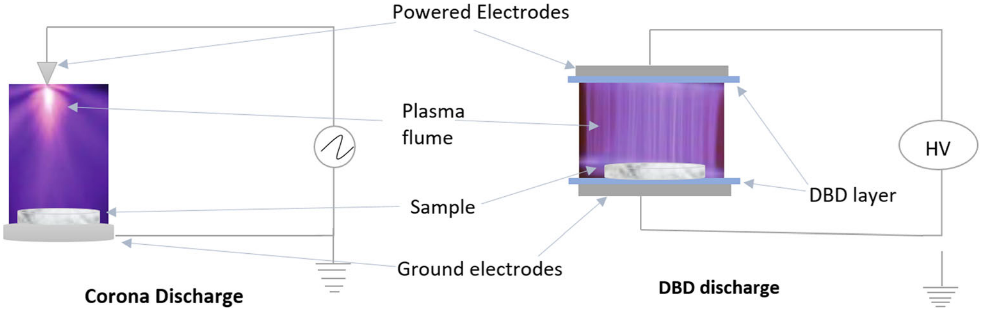
Plasma Jet

Plasma jet devices differ from other plasma devices in their ability to release stable plasma discharge to another surface with a very low electrical field. Here, the target surface need not be confined within the plasma circuit. Consequently, this method allows for treatment of various food samples without size restriction. Unlike other devices, plasma jets have four types of electrode configurations, namely, DBD jets, dielectric-free electrode jets, DBD-like jets, and single-electrode jets. The schematic arrangements of the DBD jet and DBD-like jet, with their respective electric field orientations are illustrated in Fig. 3 (Lu et al. 2012).

NTP Processing Parameters

The potency of NTP technology depends on various processing parameters. They include input power, voltage, frequency, type of process gas and flow rate, treatment time, and mode of exposure (direct and indirect). The distance between the plasma source and the target food surface play an important role in the concentration of the reactive species. As the surrounding air mixes and recombines with the process gas, the ROS concentration increases with increase in the distance from the discharge source to target surface, but the neutral species and the concentration of process gas decline. The neutral species and the concentration of process gas further decrease when most of the plasma species come into contact with the target surface due to their involvement in the plasma-chemical reaction process (Surowsky et al. 2014). The importance of the process gas cannot be overlooked, as it determines the types of reactive species generated (Reineke et al. 2015). For example, only argon-related neutral and active or ionized

Fig. 1 Schematic diagram of corona and DBD discharges. (HV is high voltage supply)



species are derived from pure argon gas plasma (Niemira 2012). On the other hand, the technology becomes cheaper and more affordable if atmospheric air is employed as the process gas compared to the expensive noble gases. Many researchers have worked with various gas combinations in the inactivation of *B. atrophaeus* and *B. subtilis* spores (Reineke et al. 2015); polyphenoloxidase and peroxidase activities (Surowsky et al. 2013); improving the safety of pork loins (Kim et al. 2013); studying the effect of hydrogen, oxygen, and ammonia on starch granules (Lii et al. 2002a); and protein destruction (Deng et al. 2007a, b). Hury et al., in their study on *B. subtilis* spore inactivation, reported oxygen-based plasma to be more efficient than pure argon plasma (Hury et al. 1998).

Regarding the mode of exposure, direct and indirect exposure are two parameters which decide the quanta of heat transmitted to sample. In terms of efficacy, direct exposure is preferable to indirect exposure. In the latter case, the amount of heat transmitted is reduced and charged particles recombine prior to reaching the sample, thus minimizing the potency. Likewise, short-lived neutral reactive species may not reach the sample either (Patil et al. 2014; Laroussi 2005).

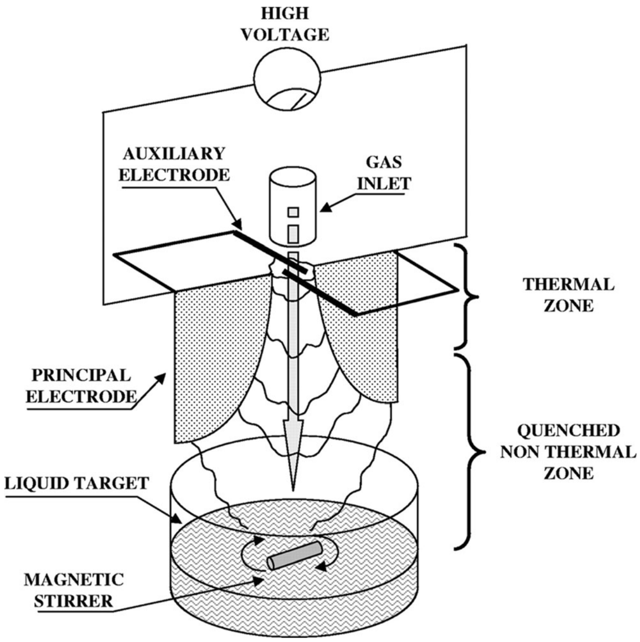


Fig. 2 Gliding arc discharge reactor showing different components Source: (Moreau et al. 2007)

Power is another vital parameter that must be considered in NTP treatments. Wongsagonsup et al. reported that cross-linking in 100-W-cooked starch was more frequent than in 50 W-treated cooked starch. The researchers further stated that the gel structure of the 50-W-treated granular starch was stronger than that of 0-W-treated starch due to increased cross-linking (Wongsagonsup et al. 2014). Higher increments in the surface roughness of corn starch films at higher voltage levels were recorded after DBD treatment (Pankaj et al. 2015). The increment was attributed to the etching effect due to the bombardment of energized plasma RS. Similarly, at higher voltages of 70 and 80 kV, 5-min DBD treatment resulted in additional functional groups.

The longer the exposure time, the more the inactivation of microorganisms. This assertion conforms with some food microstructural research. For example, Bahrami et al. (2016) reported an increase in hydroperoxide and head space n-hexanal with increases in plasma treatment time and voltage. The lipid oxidation marker, 2-thiobarbituric acid reactive substances (TBARS), increases with increases in power, treatment, and storage time (Rød et al. 2012). The tensile strength and surface hydrophilicity of zein films were reinforced after increased plasma treatment time (Dong et al. 2017).

Plasma Mechanism

The mechanisms of plasma interactions with macromolecules is quite complex because of the living tissues and cells, plasma sources (Abd El-Aziz et al. 2014), and the target food matrix (Smet et al. 2017; Surowsky et al. 2014). A possible mechanism for how the process gas generates plasma RS in the substrate is illustrated in Fig. 4. This plasma process phenomenon involves the initial cutting out of the surface, called

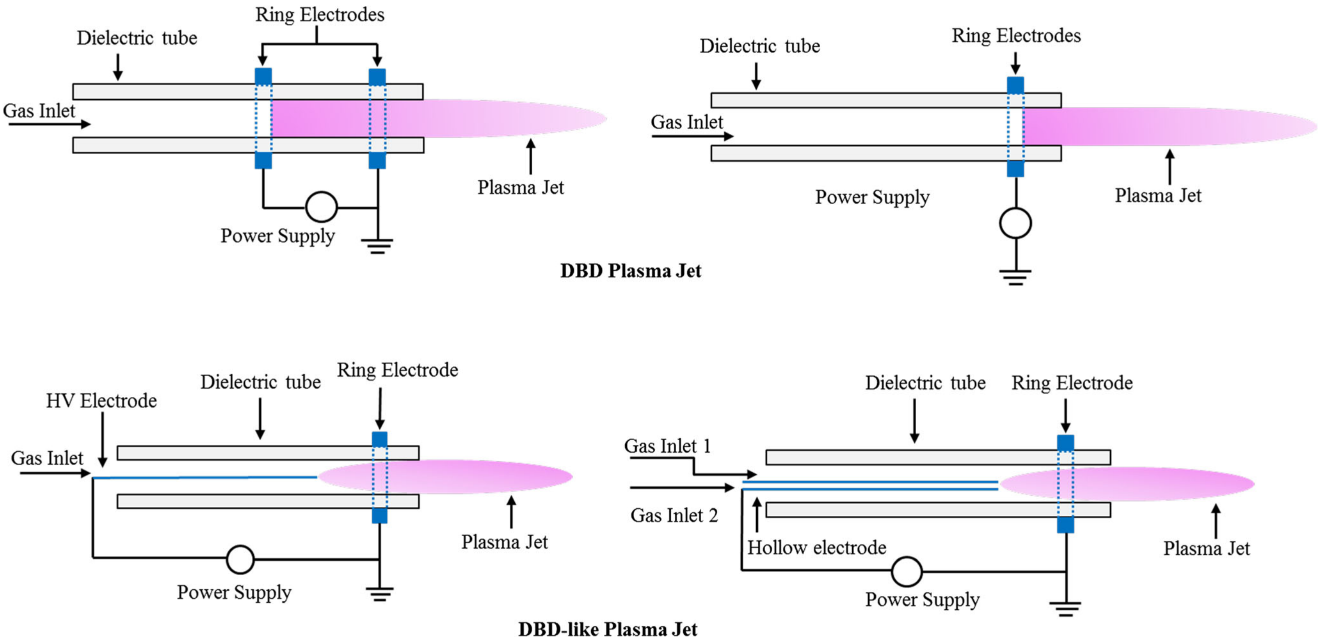
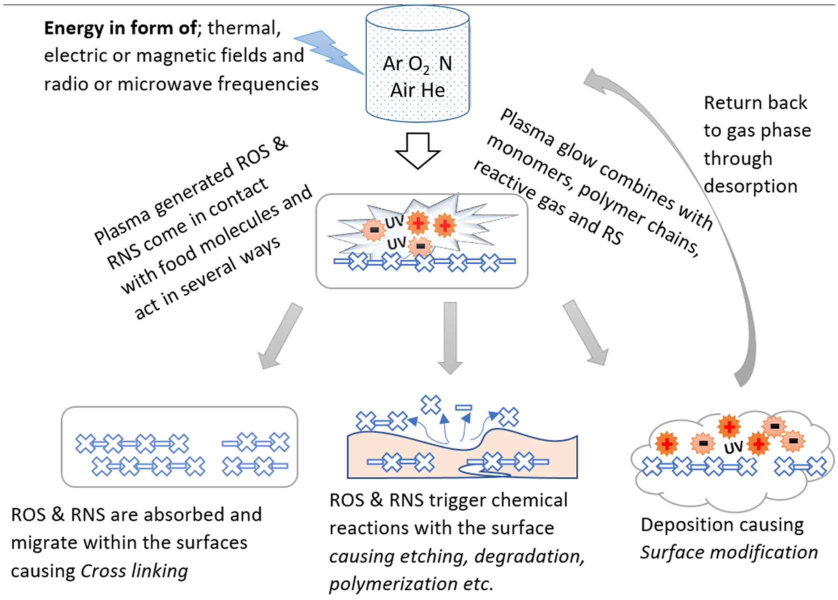


Fig. 3 Schematic view of DBD and DBD-like plasma jets. Source: (Lu et al. 2012)

etching, plasma-enhanced chemical vapor deposition on the solid surface during plasma polymerization and then finally physical and chemical transformation of the food material without any reduction or increase of substrates or by-products (Thirumdas et al. 2014). It is worth noting that the most active components of plasma are ROS and reactive nitrogen species (RNS). These components are responsible for inducing oxidation reactions (Laroussi and Leipold 2004; Surowsky et al. 2014) which are the most important plasma-related reactions in regards to organic compound degradation or microorganism inactivation. ROS, due to their high reactivity, can react with nearly all cell components. The majority of the RS generated, such as the free radicals (like OH and NO), excited O_2 and excited N_2, can cause etching and consequently react with macromolecules within the cells (Abd El-Aziz et al. 2014; Fernández and Thompson 2012; Laroussi et al. 2003; Yang et al. 2016). Prior to the diffusion of macromolecules out of the cells, the cell walls are eroded by ROS, such as O, OH, O_3, and H_2O_2, leading to breakage of chemical bonds and lesions within the cell membrane (Park et al. 2015). This phenomenon is similar to structural starch modification by plasma. The modification requires the cross-linking of OH and C–OH bond to form new C–O–C glycosidic bonds between the starch chains and subsequent release of a water molecule (Wongsagonsup et al. 2014). Zou et al. (2004) in their study of argon plasma-treated starch, proposed that the

Fig. 4 Modifications induced by cold plasma on food substrates



plasma induced a cross-linking mechanism. The researchers reported that cleavage occurs between the reducing ends of two polymeric chains, i.e., C–OH. Thus, forming new C–O–C linkages between the two chains due to cross-linking, with subsequent removal of water molecules. The modification of the starch surface was due to etching and formation of fissures/holes on the surface of the starch granules. This allows the penetration of plasma ions into the molecular structure of the starch, which causes further depolymerization and cross-linking of starch molecules (Lii et al. 2002a; Thirumdas et al. 2016). This could have a major effect on starch rheological properties. The protein oxidation and etching process is strongly triggered by atomic oxygen ($O^{\cdot}$) and OH radicals, which are important components of ROS and oxidizing agents (Surowsky et al. 2014). These RS are responsible for DNA breakage, amino acid side chain and unsaturated fatty acid oxidation, protein-to-protein cross-links, sugar modification, and peptide bond cleavage. The cleavage of OH to form glycosidic bonds in plasma-induce cross-linking mechanism in starch molecules is illustrated in Fig. 5. An increase in the carbonyl content of whey protein isolate was reported after plasma treatment. This was attributed to an increase in the number of amino acid side chain groups, such as NH− or NH_2 or by peptide bond cleavages (Segat et al. 2015). Changing the thiol groups (SH) to disulfide bonds (S–S) in protein structural modification has been linked to free radicals or ozone combining with the newly released SH and the initial SH thus forming the new S–S (Dong et al. 2017).

Effects on Food Constituents and Microstructure

Behavior of Liquid and Solid Products under NTP

The plasma RS penetration and their interaction with the food matrix depend on many factors, including type of plasma exposure (direct or indirect), food state (liquid or solid), water content, and composition. These RS within the plasma flumes play an important role in interaction with food and living organisms. However, the synergetic effect lies with the long-living active RS generated within the plasma and the food surface under treatment (Fridman et al. 2007). Park and co-workers reported in their gas phase DBD and the liquid-state nanosecond pulse plasma (NPP) study that, among the abundant plasma radicals generated, some have short life time and cannot be detected in the solution, even though they have strong influence on microbial inactivation (Park et al. 2015). In such cases, liquid food samples behave like a volume element that comes in contact with the applied NTP. Here, penetration depth might not be that important, but both the surrounding liquid components and microorganisms are impaired, depending on the process parameters. Therefore, neither ions nor electrons directly interact with microorganisms submerged in the liquid. Instead, the ions and electrons are swiftly absorbed by the liquid medium via the gas-liquid interface (Machala et al. 2013). This principle is similar to that used in plasma-activated water, which significantly alters the morphology of microbial cells on fruit and vegetables (Ma et al. 2016). The moist environment provides fertile conditions for microorganisms' growth and thus serves as an additional obstruction to the direct interaction of plasma and microorganisms. Therefore, the plasma RS penetrates the liquid, interacting with the microbial cells through ablation or etching, which leads to many biological reactions within the system (Mittler et al. 2011). Hydroxyl radicals (OH) are the main reactive oxygen species (ROS) formed in a liquid medium. When water molecules and plasma come into contact, dissociation reactions with electrons occur, which greatly depends on the electron energy, water content, and collisions of the water molecules with electrons. OH radicals can further react to yield hydrogen peroxide (H_2O_2), hydroperoxy ($OOH^{\cdot}$) radicals, or superoxide ($O_2^{-\cdot}$), depending on the pH of the liquid. These radicals are thought to be stable and active for a much longer time than the plasma exposure itself (Locke and Shih 2011; Fridman et al. 2007). In molecular dynamics (MD) simulations of the action of ROS on peptidoglycan (PG), ROS such as O, OH, O_3, and H_2O_2 caused breakage in bonds that are structurally important in PG. In particular, O_3, with a strong antimicrobial effect was shown to decompose in water, forming OH radicals as the major secondary oxidant. This leads to structural collapse and subsequent damage to the bacteria cell wall. Furthermore, O radicals are more potent in the breakage and formation of bonds than other plasma species (Pandiselvam et al. 2015; Park et al. 2015; Tzortzakis and Chrysargyris 2017). For plasma-treated methylene blue and methyl orange dyes, OH radicals were responsible for alteration of the dyes' structures by reacting with one of the methyl groups, subsequently removing the hydrogen atom, thus forming a water molecule and a CH_2 radical in the dyes (Attri et al. 2016).

However, in regard to solid food matrices, penetration depth does not matter. This is especially true when the primary aim is to achieve reasonable surface microbial decontamination and retention of the majority of food nutrients and quality; low penetration depth is desirable, as this can be enough to effectively inactivate microorganisms. A penetration depth of 15 μm was reported to be effective in deactivation of *Porphyromonas gingivalis* biofilms (Xiong et al. 2011). A strong bactericidal effect was reported on 25.5-μm-thick layer of *Enterococcus faecalis* biofilm (Pei et al. 2012).

Impact of NTP on pH

The pH of a solution is an important factor in determining the effectiveness of NTP decontamination in the medium (Gurol et al. 2012; Liao et al. 2017b). Our aim was to determine the impact of NTP on the pH of food matrices related to

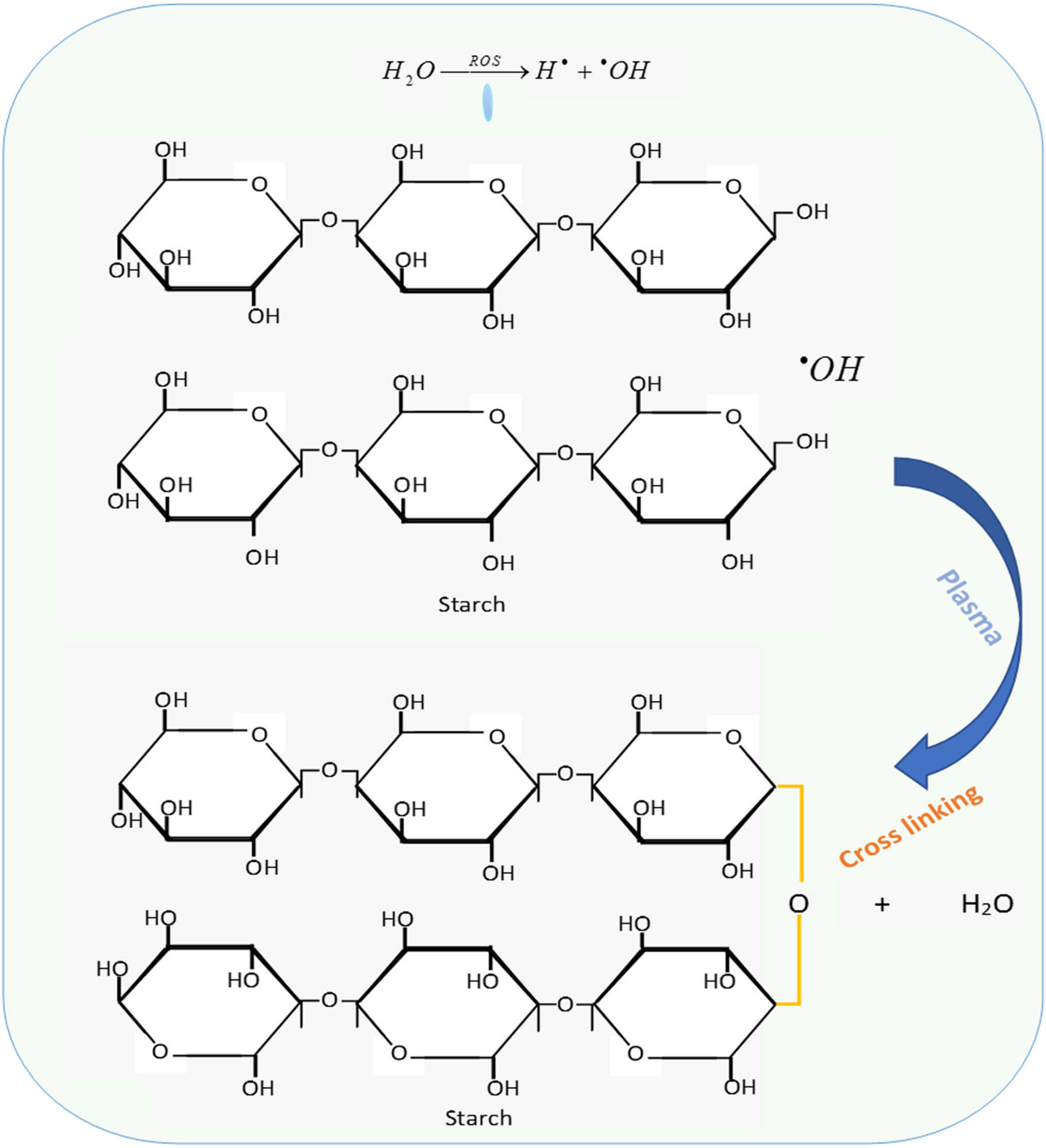


Fig. 5 Proposed NTP cross-linking mechanism caused by OH generated by thermal dissociation of water molecules. The cross-linking is highlighted in yellow

microstructure. Although no available literature reports regarding NTP impact on pH in dry solid food matrix were found, a slight pH reduction in aqueous starch was reported after NTP treatment (Lii et al. 2002a, c). This reduction was linked to the addition of a new acidic group, such as a carboxyl or carbonyl group, possibly by ozone or nitrogen oxide oxidation. Lii et al. reported a decrease of between 1.4 and 2.8 in the pH of starches from different botanical origins after exposure to low-glow plasma (Lii et al. 2002a). This decrease was due to oxidation by ozone or nitrogen oxide, which led to additional carboxylic acids. Similarly, after 20 min of NTP treatment of milk, no remarkable changes were reported in the pH values of the milk (Gurol et al. 2012). However, in pea protein extract, a pronounced decline in pH was observed. The decline was from 8.4 to 7.6 for the control compared to a 10-min treatment, and the pH value reduced further to 7.2 after storage. The increase in acidity may be due to plasma ROS, which might have caused amino acid degradation, with the degradation products diffusing into the solution and thereby causing acidity or further formation of acidic compounds (Bußler et al. 2015). Another result revealed a significant decrease (6.7–2.5) in the pH of soy protein isolate from the first minute up to 10 min of direct CAPP exposure. The alteration of the pH might be a result of interaction of water molecules and ions that led to formation of

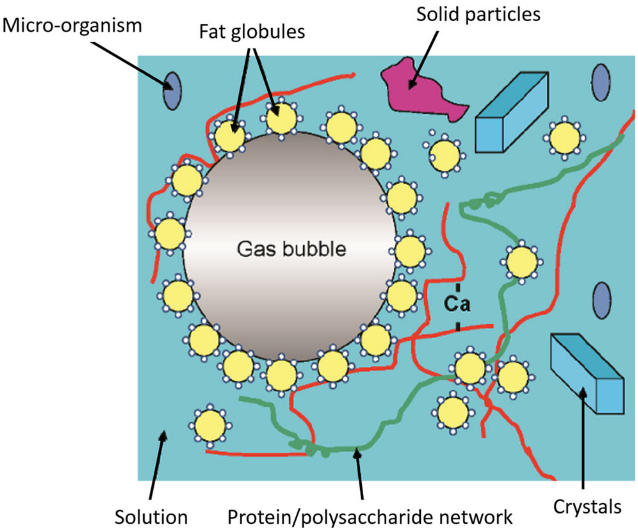


Fig. 6 Food matrix structure. Source: (Jeantet et al. 2016)

hydronium ions. This result is consistent with pulsed ultraviolet (PUV) treatment of the same products in which a significant decrease in pH from 6.7 to 5.5 was recorded after 6 min PUV treatment. In gamma irradiation, no statistical significance was reported (Meinlschmidt et al. 2016). An increase in acidity can also be interpreted by the increase in peak intensity of NTP-treated samples at the 1710 cm^{-1} wave number, which corresponds to carboxylic acid wave number conferred by the C=O bond (Thirumdas et al. 2017).

Food Microstructure

Food microstructure is the arrangement of structural elements within the food and the forces that bind them together (Aguilera et al. 2000). These structural components include starch, proteins, lipids, water, and air (Fig. 6). The nature of how these components are assembled into structures determines the overall properties of foods (Jeantet et al. 2016; Morris and Groves 2013). For instance, starches are employed in food thickening (gels) to stabilize emulsions and foams and to generate texture. When used as gels, their shape, molecular weight, and ionic strength determine the strength and viscosity of the gel. If the starches are used as emulsifiers or foam stabilizers, they are enhanced through their molecular structures at air-water or oil-water interfaces when these components are bound to the starch present in the aqueous medium (Jeantet et al. 2016; McClements 2007). On the other hand, there are food-related structures which contain complex multicomponent structures such as starch-protein-lipid combinations. These structures gelatinize at different stages of processing to produce the final structure and texture of foods (Berk 2013; Jeantet et al. 2016; McClements 2007; Parada and Santos 2016). Application of NTP modifies the surface structures of these biopolymers (Pankaj et al. 2015; Wongsagonsup et al. 2014). In this regard, NTP has several advantages in regards to interaction with polymeric biomaterials. Apart from the absence of thermal damage, it offers uniformity in surface treatment and does not involve the use of hazardous solvents (Desmet et al. 2009; Misra et al. 2015). Another aspect in which biopolymers can be modified by NTP is surface functionalization. Thirumdas et al. detected some additional functional groups in plasma-treated starch through Fourier transform infrared (FTIR) spectroscopy (Thirumdas et al. 2016).

Like starches, proteins play an important role in food microstructure formation. The spatial structures of proteins determine their biological function, and a structural modification might occur as result of processing. For example, globular proteins, due to their various functional behaviors in food applications, are considered to be important ingredients. Any change in these molecules as microstructuring agents in food processing might lead to significant alterations in the quality characteristics of the end products. A significant functional aspect of proteins is their ability to generate reactive particles with a lower drive to aggregate by reducing exposed hydrophobicity (McClements 2007). This functional aspect is responsible for the formation of protein aggregates and bulk networks. Coincidentally, Firoozmand and Rousseau (2015) have also demonstrated that plant-based proteins can be used as structural enhancers to yield protein-starch gel structures with diverse rheological properties and food applications. In view of the high demands on minimally processed food products, NTP-driven microstructural modification is highly anticipated.

NTP Interaction with Food Microstructures

The way food microstructures and constituents interact and the state of food will definitely affect the effectiveness of NTP. For example, in granular starch, the behavior under NTP depends on their botanical origin as well as the structure of the starch (Lii et al. 2002c). Although some components will thrive at the expense of others during oxidation, some will work concurrently with plasma RS in such a way to improve the oxidation reaction. Upon NTP treatment, various transformations occur, ranging from degradation and by-product transformation to multistep chain reactions, and formation of cross-links between different biomolecules particularly proteins (Misra et al. 2016a, b). We will discuss the plasma-related impacts on food macromolecules and food constituents.

Starch

Starch, being semi-crystalline in nature, is a carbohydrate and comprises two polysaccharides, namely, amylose and amylopectin (Kizil et al. 2002). Amylose is a linear molecule composed of anhydroglucose units which are interconnected via α-1,4 linkages with few branched networks. The other component, amylopectin, is a larger branched polymer with anhydroglucose linkages of α-1,4 and α-1,6 which serve as branching points (Kizil et al. 2002; Pankaj et al. 2015). Native starch is an important ingredient in many food products. The majority of starch is derived from corn, followed by wheat, potato, cassava, banana, amaranth, and rice (Thirumdas et al. 2017). These commodities undergo several processing operations such as milling, storage, and cooking before consumption. As a result, the starch granules are subjected to changes such as gelatinization and retrogradation (Patindol et al. 2008). These changes are peculiar to each particular starch product, and as such, hinder their applicability in food industries. Thus, there is a need for functional enhancement by various methods including physical and chemical means. NTP treatment, on the other hand, equally modifies some properties of these starches. Several NTP starch modifications have been reported, ranging from alteration of rheological properties to changes in surface morphology and the addition of functional groups. The impact of NTP on starch-related foods and starch granules of different botanical origins are presented in Table 1.

Molecular Structure Structural starch modifications can enhance their functional values and diversify their importance in food applications and other processing industries such as paper, textile, and chemical industries. Starch modification is attributed to cross-linking, likely through the following mechanism (Zou et al. 2004):

$$Starch\text{–OH} + \text{HO–}Starch \overset{\text{NTP}}{\rightarrow} Starch\text{–O–}Starch.$$

The NTP-induced chemical changes to starch include cross-linking, depolymerization and formation, and addition of new functional groups. Additionally, a decrease in molecular size and radius of gyration of starches based on the type of plasma and starch have been reported (Bie et al. 2016; Lii et al. 2002a, b, c; Zhang et al. 2014). For example, a study of the effects of oxygen glow plasma on supramolecular structures and molecular characteristics and their related mechanisms on corn and potato starches was done by Zhang and co-workers. Potato starch appeared to be more prone to NTP degradation than corn starch and this effect was linked to more water trapped in the molecules of potato starch (Zhang et al. 2014). Lii et al. opined that larger molecules have a greater tendency to be degraded by NTP than smaller ones (Lii et al. 2002a). Pankaj et al. reported a slight decrease in the maximum degradation temperature of DBD-treated corn starch films, which they attributed to etching and random chain scission of the starch polymer after treatment (Pankaj et al. 2015). In contrast, Zhang et al. stated that the molecular weight and radius of gyration of potato starch was increased after nitrogen and helium glow plasma treatments. For a helium glow plasma treatment of 60 min, the increase in averaged molecular weight of potato starch was from 6.114×10^7 to 1.042×10^8 g/mol (Zhang et al. 2015). In ammonia and hydrogen plasma treatments, the molecular weight of cassava starch was reduced from 9.35×10^7 g/mol to 1.59×10^7, whereas that of hydrogen plasma was reduced to 5.79×10^7 g/mol (Lii et al. 2002a). In line with the aforementioned trend, similar molecular weight reductions in potato starch were reported with increased ozonation times (Castanha et al. 2017).

Zou et al., in their study of commercial starch modification, suggest that a plasma energy-charge-transfer function is an ideal plasma mechanism. The control and argon plasma-modified starch did not differ. Analysis using C-NMR spectra showed that C=O bonds did not exist in the plasma-modified starch, signifying the loss of an OH group due to cross-linking in α-D-glucose units (Zou et al. 2004). In high-amylose corn starch film, the collision of plasma-generated RS causes surface roughness via etching, and the starch film surface suddenly shows the appearance of new O=C–O groups after NTP treatment. Through x-ray diffraction analysis, an A-type crystal structure was apparent (Pankaj et al. 2015). Correspondingly, increased ozonation time resulted in the addition of carbonyl and carboxylic groups in potato starch (Castanha et al. 2017). This might be related to the oxidizing power of ozone, which is also a component of plasma RS. In low-pressure ethylene glow plasma treatment of starches, Lii et al. opined that grafting was likely to occur between rice and potato starch molecules and ethylene, whereas this assertion was not made for cassava, potato, and normal and waxy corn starches (Lii et al. 2002b). These studies indicate that NTP, as an emerging technology, is capable of modifying the molecular structure of starches. However, the changes mostly depend on plasma type, the botanical origin of the starch and length of treatment time.

Rheological Properties The deformation, pasting and flow behaviors of food materials under applied stress are termed "rheological properties." Starch viscosity is an important parameter when using it as food thickener (Ai and Jane 2015). The impact of NTP treatment on starch granules viscosity may occur through depolymerization of amylose and amylopectin chains and degradation of the shear resistance of swollen granules. A reduction in viscosity due to cross-linking as a result of swollen granules was reported in highly cross-linked starch (Ai and Jane 2015). Thirumdas et al. recorded an altered surface morphology of rice starch granules due to fissures formation. They opined this could affect starch rheology, especially when the starch granules are subjected to cooking; the leaching of the amylose molecules into the surroundings through these fissures is possible due to higher solubility and syneresis (Thirumdas et al. 2016). Zhang et al. reported a reduction in the breakdown, trough, peak, and final viscosities of potato starch due to nitrogen and helium glow plasma treatments at 245 V for 30 min. The maximum decrease in viscosity was 18% with the nitrogen glow plasma and 23% with the helium plasma treated for 60 min. Regarding the breakdown viscosity, the decrease was 23% after treatment with helium glow plasma (Zhang et al. 2015). Starches with

Table 1 NTP impact on some plant-derived starches

Plasma type	Starch and starch-related source	Treatment parameters	Observed results after treatment	References
DBD plasma	High-amylose corn starch film	1–5 min; 60, 70, and 80 kV and 48% relative humidity	- Significant increase in surface roughness - No change in A-type crystal structure - Increase in surface hydrophilicity	Pankaj et al. (2015)
Low-pressure glow plasma	Starches of cassava, corn, high amylose corn, potato, rice *Indica*, rive *Japonica*, sweet potato, waxy corn, oat, wheat	Ammonia, hydrogen, oxygen; 1–2 kV; 10, 20, and 30 min; 13.33 Pa 1.33 Pa pressure	- Starch polysaccharides depolymerization - Higher molecular weight starch suffer higher depolymerization depending on plasma gas - Only in oxygen gas was subtle oxidation observed - No waste produce in the dextrin	Lii et al. (2002a)
Low-pressure cold plasma	Parboiled rice	30, 40, and 50 W power; 5-, 10-, and 15-min time; 3-cm electrode distance	- Increase in water absorption - Reduction in cooking time (up to 8 min) - Improvement in textural properties with increase power and time - Gelatinization temperature increment - Surface energy increment and contact angle reduction	Sarangapani et al. (2016)
Oxygen glow plasma	Potato and corn starches	Gas pressure 2000 Pa; 30, 45, and 60 min, 10 mm discharge distance	- Higher degree of destruction at the initial stage in both starches - Potato starch suffers higher destruction in molecular properties - Upon comparison, potato starch has more compact scattering objects thus displayed a weaker resistance to plasma treatment	Zhang et al. (2014)
Low-pressure plasma	Brown rice (*Indica*)	Air as process gas; 30-min treatment time; 1, 2, and 3 kV voltages	- Surface etching of the rice kernel - Reduction in cooking time, width expansion, cooking loss, elongation ratio, water absorption, and viscosity	Chen (2014)

Table 1 (continued)

Plasma type	Starch and starch-related source	Treatment parameters	Observed results after treatment	References
Cold plasma	Brown rice	250 W; 15 kHz; ambient air; 5, 10, and 20 min	- Slight decrease in pH - Significant increase in water uptake rate and α-amylase activity - Significant decrease in hardness	Lee et al. (2016)
Glow discharge plasma	Commercial soluble starch	Argon gas at 200 Pa; 45 min treatment; 700 V	- Significant hydroxyl group protons reduction	Zou et al. (2004)
Plasma jet discharge	Cooked and granular tapioca starch slurry	Argon; 50 and 100 W; 5 min	- Both treated starches at 50 and 100 W have stronger structures - Highly cross-linked clusters of undissolved starch molecules were visible - Increase of glucose bonds in 50 W granulated treated starch and 100 W treated cooked starch	Wongsagonsup et al. (2014)
DBD plasma	Corn starch	Air as gas: 1, 5, and 10 min; 6.5 mm discharge distance; 50 V; 75 W power; 40% humidity	- Increase in pinhole diameter of internal starch structure - Decrease in degree of crystallinity - Molecular chain oxidation - Molecular weight reduction - Paste viscosity decrease	Bie et al. (2016)
Cold plasma	Rice starch	Atmospheric air as gas; 15 Pa; 40 & 60 W power; 3 cm electrode distance;	- Decrease in pH, turbidity, and relative crystallinity - Increase in leaching of amylose molecules, pasting, and in final viscosities - Tendency of starch paste to retrograde was decreased - Formation of fissures on starch granules	Thirumdas et al. (2016)

such properties are said to have high thermal stability and lesser tendency for retrogradation (Li et al. 2011). Bie et al. in their DBD plasma-treated corn starch, reported a significant decrease in starch viscosity. This finding was attributed to the shift in rheological behavior from pseudo-plastic to Newtonian. The researchers further used the power law ($\tau = K\gamma n$) to describe the fitted curves of the treated corn starch. It was shown that the coefficient (K) decreases, whereas the flow coefficient (*n*) increases with increased treatment time (Bie et al. 2016). The dynamic viscoelastic properties of argon jet plasma-treated tapioca starch were examined by Wongsagonsup et al. The findings revealed that 50-W plasma-treated starch formed a stronger gel structure (lower tan δ) than the native starch due to cross-linking. In contrast, a decrease in gel structure (higher tan δ) was recorded for 100-W plasma-treated starch due to the depolymerization effect (Wongsagonsup et al. 2014).

Surface Morphology NTP is similar to any other nonthermal technology, such as ozone, ultrasound, and gamma irradiation (Bashir and Aggarwal 2017; Castanha et al. 2017; Sujka 2017) in regards to starch modification. The exposure of starch granules to plasma treatment causes formation of pores, cavities, fissures, or pinholes on the surface of the starch granules possibly due to etching or surface corrosion (Bie et al. 2016; Lii et al. 2002c; Thirumdas et al. 2016). With low-pressure glow plasma, different starch granule responses are observed after exposure. Electron paramagnetic resonance spectra shows that potato and cassava starches undergo a 28 and 23% decrease in bands, respectively, in order of susceptibility to plasma. Oat and high-amylose corn starch have been reported to be more resistant to plasma treatment. Visible damage was observed in wheat starch granules and KSS7 *Indica* starch, with the latter suffering the most damage. For rice starch, no pores were detected in SEM images. After plasma treatment, the pH decreases in the starch solution, suggesting oxidation of granule surfaces, which subsequently form carboxylic acids (Lii et al. 2002c). Bie et al. (2016) reported after 5 min of plasma treatment, changes in the internal structures of corn starch granules had extended to the surface (Fig. 7). The researchers highlighted the plasma RS entering the interior of corn starch granules was the cause of the stress concentration in the internal structure, particularly in the hilum. Zhang et al. used a polarized light microscope (PLM) to study the surface of potato starch granules after exposure to helium glow plasma. The researchers reported destruction of the starch granules due to surface corrosion (Zhang et al. 2015). Similar assertions were made by Zhang et al. when they studied the effect of glow plasma on microstructural, mesoscopic, and molecular structures of potato starch (Zhang et al. 2014).

Proteins

Proteins are part of food constituents that can be enhanced during food processing to improve the shelf life, organoleptic properties, and functionality (de Jongh and Broersen 2012). This enhancement can diversify and increase the commercial value of protein-rich foods in the food industry. In NTP processing, polymer ablation is among the processes through which these biopolymers are enhanced, possibly via etching (Dong et al. 2017; Fricke et al. 2011). Changes in secondary structure and decreases in enzymatic activity of plasma-treated protein have been investigated using circular dichroism (CD) and fluorescence spectroscopy. Kylián et al. (2008) employed ellipsometric measurements to detect the level of protein removal after the plasma treatment. These changes were linked to plasma RS (Takai et al. 2012; Hayashi et al. 2009). Deng et al. (2007b) and Hayashi et al., (Hayashi et al. 2009) showed that atomic oxygen and nitride oxide play important roles in protein degradation during plasma treatment through their synergistic effects. Possible effects include alteration of the secondary and tertiary structures of the enzymes and oxidase. Plasma-protein interactions are multifaceted and to date, little research has been conducted on this aspect. Hence, there is an urgent need to explore more of this area to elucidate plasma modification mechanisms. Nevertheless, some protein-related studies are summarized in Table 2.

Molecular Structure There is no doubt that NTP causes structural modification to protein structures due to the various chemical changes involved. Saget et al. reported a pronounced increase in protein carbonyl content after 30 min of plasma treatment. However, upon extending the exposure time beyond 30 and 60 min, no further significant increase was observed. The researchers attributed the carbonyl formation to the modification of amino acid side chain groups such as NH– or peptide bond cleavage. Abstraction of –SH groups from the amino acid cysteine was further noticed in the protein structure (Segat et al. 2015). In contrast, Jiang et al. reported a decrease in sulfhydryl groups and carbonyl content of silver carp myosin, which the researchers found to correlate with an increase in ozone treatment time (Jiang et al. 2017). In another research effort, the potent effect of atmospheric pressure cold plasma (ACP) on 20 naturally occurring amino acids solution was reported by Takai et al. (2014). After the treatment, 14 of the amino acids were modified, for which the aromatic and sulfur-containing amino acids were decreased by the plasma treatment. Fluorescence emission spectra measurements in a study by Bußler et al. (2015), showed plasma-induced structural modifications on pea protein isolate (PPI). This finding was attributed to the oxidation of tryptophan and quenching phenomena. Misra et al. used FTIR spectroscopy analysis to show alterations in the secondary structure of ACP-treated wheat flour gluten proteins. A decrease in β-sheets and an increase in α-helix and β-turns for both strong and weak wheat flour gluten were reported. These changes indicate a re-arrangement of protein molecules and an improvement in the strength of the hydrogen bonds in the weak flour, which are possibly caused by plasma ROS, such as atomic oxygen and hydroxyl radicals, attacking the tryptophan (Misra et al. 2015). Equivalently, CD analysis showed a remarkable decrease in the α-helical content and β-turns and β-sheets of myosin, while the random coil content grew proportionally with ozone treatment time (Jiang et al. 2017). These observations illustrate the conformational changes of β-structure in myosin. In ACP treatment of zein films, x-ray diffraction (XRD) analysis showed breakage in the inter-helix molecular

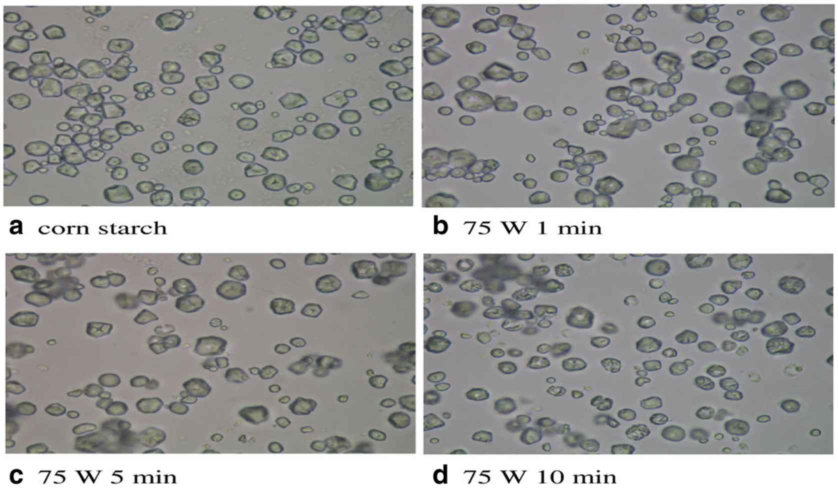


Fig. 7 Optical micrographs of corn starch granules treated with plasma for different times Source: (Bie et al. 2016)

aggregates of the zein film after 60 and 70 kV plasma exposure (Pankaj et al. 2014). Bahrami et al. reported insignificant changes in the total protein levels of plasma-treated wheat flour. However, flour treated with highest voltage of 20 V for 120 s had significant alterations in the distribution of the protein fraction (Bahrami et al. 2016). Another aspect of importance is surface hydrophilicity, as it is an indicator of protein structural unfolding. The surface hydrophilicity and tensile strength of zein films were reinforced by an increase in DBD plasma treatment time (Dong et al. 2017). Significant increases in surface hydrophobicity after 15-min ACP treatment were observed. However, upon increasing the treatment time to 30 and 60 min, a more significant increase was seen. This corresponds to minor structural changes within the initial treatment time and remarkable subsequent changes after that (Segat et al. 2015).

Rheological Properties Understanding the interactions of protein molecules with other molecules such as water, polysaccharides or other proteins that lead to the formation of gels of new structural properties is of paramount importance in food applications. Properties such elasticity, viscoelasticity, and gelation are important to consider for protein modification. The rheological properties of wheat flour were improved, as reflected by dough strength and mixing time, after plasma treatments. This improvement was linked to the oxidation of protein sulfhydryl groups and the subsequent formation of disulfide bonds between cysteine moieties. Increases in moduli and a decrease in tan δ also resulted in improved viscoelasticity of the wheat flour (Misra et al. 2015). The coarsening of the gel structure of globular proteins and reduction in storage modulus via addition of gelatin have been reported (Ersch et al. 2016). The water- and fat-binding capacities of pea protein isolates were increased to 113 and 116%, respectively, after plasma treatment. These results were due to plasma-induced surface modifications that were more pronounced for high-protein and high-fiber matrices than for starch-rich fraction matrices (Bußler et al. 2015).

Surface Morphology As mentioned in the previous sections, NTP induces surface roughness in biopolymers through etching the surface. The etching effects caused by NTP might be a result of a physical process such as the physical removal of lower molecular weight polymers or chemical processes such as bond breakage, chain scission or chemical degradation (Dong et al. 2017; Hayashi et al. 2009; Pankaj et al. 2014). Dong et al. reported significant changes in the surface morphology of zein protein molecules after 7- and 10-min treatments. The researchers linked these surface ruptures to bombardment and etching caused by energized plasma RS that cleave the C–C and C–H bonds (Dong et al. 2017). SEM images of the surface etching are shown in Fig. 8. An increase in surface roughness was reported by Pankaj et al. after DBD plasma application on zein film. The researchers observed that the roughness increase with increases treatment time and applied voltage (Pankaj et al. 2014).

Impact of NTP on Enzymes

Most enzymes are proteins and so their reactions are equivalent to those described for proteins. These compounds could be either desired or undesired in food, for example, catalytic reactions might reduce food quality attributes. Examples of such activities include, peroxidase (POD), polyphenol oxidase (PPO), and tyrosinase. These enzymes are known to cause nutritional losses via enzymatic browning reactions and off-flavor formation through lipid decomposition by lipases (Misra et al. 2016b). Most authors attributed the changes in secondary and tertiary structures of proteins/enzymes by RS produced from plasma discharge. In addition, breakage of C–H, C–N, and N–H bonds by the same RS have also been

Table 2 NTP impact on protein

Plasma type	Type of protein/ enzyme	Treatment parameters	Observed results after treatment	References
Low-pressure glow plasma	Amylose starch protein	Ammonia, hydrogen, oxygen; 1–2 kV; 10, 20, and 30 min; 13.33 Pa & 1.33 Pa pressure	- Increase in formation of complexes with iodine - Had slightly longer chain	Lii et al. (2002a)
Atmospheric pressure cold plasma (ACP)	Whey protein isolate (WPI)	Atmospheric air as gas; 1, 5, 10, 15, 30, and 60 min; 44-mm electrodes distance	- Increase in yellowing color in WPI - Minor pH reduction - Protein oxidation and thiol (-SH) groups reduction. - Increase in foam stability and surface hydrophobicity - Decrease in emulsifying ability	Segat et al. (2015)
Surface dielectric barrier air discharge (SDBD)	Pea protein isolate from grain pea flour	Ambient air; 10-min exposure; 8.8 kV; 3.0 kHz; 12-mm plasma distance	- Increase in water and fat-binding capacities - Increase in solubility - Oxidation of tryptophan and subsequent changes in protein structure - No color change in protein isolate	Bußler et al. (2015)
Cold plasma	Wheat flour protein	Air; 60 and 120 s; 15 and 20 V	- Increase in molecular weight fractions - Improvement in dough strength - No significant change in total proteins	Bahrami et al. (2016)
DBD	Zein powder	Atmospheric air; 1, 3, 5, 7, and 10 min; 75 V; 1 ± 0.2 A; 8-mm plasma discharge distance	- Increase in (SH) group - Change in secondary structure Decrease in pH and mean diameter of zein micelles - Improve solubility in neutral and acidic water solution	Dong et al. (2017)

Table 2 (continued)

Plasma type	Type of protein/enzyme	Treatment parameters	Observed results after treatment	References
DBD plasma	Zein film	Atmospheric air; 60, 70, and 80 kV; 50 Hz; 48% RH; 1, 2, 3, 4, and 5 min; 22-mm plasma discharge distance	- Increase in surface roughness and equilibrium moisture content - Change in the protein conformation - Breakage of the molecular aggregate	Pankaj et al. (2014)
Atmospheric DBD-tube jet	Bovine serum albumin (BSA) as model protein	Helium and helium/oxygen as gas; 4, 60, 180, and 300 s; 8 kV	- Removal of protein from surface - Protein degradation	Deng et al. (2007a)
ACP	Whey protein isolate model solution	Ambient air, 70 kV; 1, 5, 10, 15, 30, and 60 min exposure time; 44-mm distance between electrode	- Mild protein oxidation - Increase in carbonyl groups and surface hydrophobicity - Decrease in free SH groups - Increase in foaming and emulsifying capacity	Segat et al. (2015)

highlighted (Hayashi et al. 2009). Others have reported the effects of H_2O_2 (Ke and Huang 2013) and UV (Falguera et al. 2011; Ke and Huang 2013) on protein degradation and enzymatic inactivation. In the inactivation of PPO and POD, Surowsky et al. (2013) reported a reduction in activity and α-helix content after plasma exposure. This reduction was accompanied by reduced and red-shifted tryptophan fluorescence intensities. A 30-min POD inactivation using arc discharge plasma resulted in heightened fluorescence intensities at 450 nm with excitation at 330 nm. This increase is linked to the degradation of heme, a compound responsible for POD activity. The plasma ROS associated with such potent effects were H_2O_2, which reduces the heme into fluorescent products, OH radicals, which destroy the enzyme's structure, and a UV component that was responsible for the acceleration of inactivation (Ke and Huang 2013). This finding was similar to observations in fresh-cut apples treated with low-frequency DBD plasma, where the occurrence of enzymatic browning dropped drastically. The residual activity of PPO was reduced by 42%, with a corresponding increase in treatment time after 30 min. The reduced activity was linked to the chemical effects of OH and NO radicals on the amino acid structures (Tappi et al. 2014). Several articles have been published describing enzymatic inactivation by nonthermal technologies, for example, a study on the effects of HPP on the shelf life of apple juice (Juarez-Enriquez et al. 2015), inactivation kinetics of pectin methyl esterase (PME) using HPP (Riahi and Ramaswamy 2003) and UV irradiation (Falguera et al. 2011). Table 3 presents a summary of literature related to the impact of NTP on enzymes.

Impact of NTP on Lipids

Lipids are mostly found in animal- and plant-based foods as fats. Lipid oxidation is a chemical phenomenon that is accompanied by off-flavor formation, causing a huge impact on the sensory characteristics of foods. However, with NTP, lipid oxidation is initiated by ROS such as atomic oxygen and hydroxyl radicals (Van Durme et al. 2014). Joshi et al. (2011) stated that the ROS generated due to plasma exposure produces oxidative stress, which causes membrane fractions to undergo lipid peroxidation in *Escherichia coli* in proportion to the amount of plasma energy applied. Furthermore, in plasma-triggered lipids oxidation, reactions are actuated by OH radicals (Surowsky et al. 2013), singlet oxygen, hydrogen peroxide-like species, and ROS scavengers such as α-tocopherol (Joshi et al. 2011), or with non-radical and radical oxygen species (such as $^1O_2$, O_3, H_2O_2, ROOH,. $O-_2$,. OH, $RO^\cdot$, $ROO^\cdot$), as in the case of a non-plasma oxidation technique (Colakoglu 2007) whose action on parts of the cell

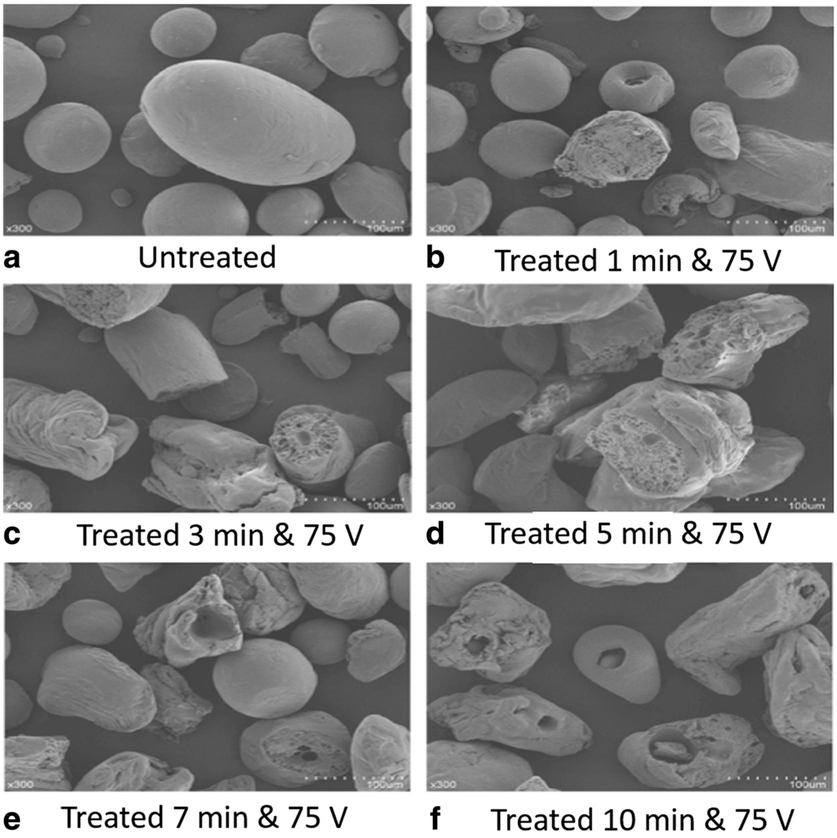


Fig. 8 SEM images of surface morphology of untreated and plasma-treated zein powder. Source: (Dong et al. 2017)

membrane causes modification and disintegration of unsaturated fatty acids into lipid peroxides. To effectively study the chemical phenomena of lipids in real food systems, experimental temperatures are kept at nearly ambient conditions to prevent checkmate additional aromatic formation via mechanisms such as the Maillard reaction, Strecker degradation or the volatile compound formation. However, these processes are very slow (Gunstone 2006; Krichene et al. 2010). To fast-track the oxidation process, chemical catalysts in combination with light were used (Colakoglu 2007). To avoid the use of chemicals and to overcome the tedious nature of the aforementioned techniques, electron beam irradiation (Cuppett et al. 2000) and recently, NTP (Van Durme et al. 2014; Yepez and Keener 2016), were harnessed. Table 4 presents a summary of NTP-induced lipid oxidation with different types of food.

A sizeable number of articles have reported lipid oxidation in NTP-treated samples computed by measurement of 2-thiobarbituric acid reactive substances (TBARS; the detection of malondialdehyde, MDA; Cuppett et al. 2000; Kim et al. 2015; Oh et al. 2016), or measurement of peroxide values that focuses on hydrogen peroxide formation (Anwar et al. 2007; Bahrami et al. 2016; Capuano et al. 2010). All these are formed as primary oxidation by-products. In contrast, measurement of the para-anisidine value (Anwar et al. 2007); 2-propenal, 2- pentenal, and heptanal (Vandamme et al. 2015); head space n-hexanal (Bahrami et al. 2016); aldehydes; and 2-pentyl furan (Van Durme et al. 2014) form as secondary volatile oxidation products signifying lipid oxidation. The measurement of the latter is a more realistic approach (Ahn et al. 2012).

In encapsulated DBD plasma treatment of milk at 5 and 10 min, the TBARS slightly increased in treated milk after 10-min exposure, which did not cause noticeable deterioration. This was linked to plasma RS such as ozone, which may accelerate peroxide formation during lipid oxidation (Kim et al. 2015). Plasma corona discharge was employed to treat raw milk at 35 °C, for up to 20 min with 90 mA current supply. Biochemical analysis showed that the plasma treatment did not impose significant changes to lipid composition. Pronounced changes in the content of other organic compounds such as 1-octanol, 2-heptanone, 2-hexenal, 2-octenal, nonanal, benzaldehyde, and aldehydes were observed. Additionally, the amount of hexadecanoic acid (C16:0) decreased after 3 min of plasma exposure. Subjecting it to longer treatment times resulted in increased amounts. These changes were attributed to dehydrogenation caused by oxygen radicals (Korachi et al. 2015). The safety of pork loins was investigated using DBD plasma with helium and He + O_2 as process gas. In addition to the appreciable reductions in *E. coli* achieved at 5- and 10-min exposure, the TBARS values of He + O_2 plasma-treated samples were higher compared with

Table 3 NTP impact on enzymes

Plasma type	Type of Enzyme	Treatment parameters	Observed results after treatment	References
Cold plasma	Polyphenoloxidase (PPO) and peroxidase (POD)	Argon and oxygen; 0–360 s; 65 V; 10-mm plasma discharge distance	- 90% reduction of PPO activity at 180 s - 85% reduction of POD activity at 240 s - Change in secondary structure	Surowsky et al. (2013)
Plasma discharge	Horseradish peroxidase (HRP)	Argon gas; 1200 V; 20 mA; 3–5 mm plasma discharge distance	- Reduced enzymatic activity - Protein degradation - Degradation of heme and reduced iron contents	Ke and Huang (2013)
ACP-DBD	Tomato POD	Air at 42% RH; 30, 40, and 50 kV; 26-mm distance	- Reduction in enzymatic activity	Pankaj et al. (2013)
Plasma processed air (PPA)	PPO and POD from freshly cut apple and potato slices	Ambient air; 2.5, 5, 7, or 10-min exposure time; 1.2 kW	- Reduction in PPO activity by 62% in fresh-cut apple and 77% in potato tissues - POD activity was reduced by 65 and 89% in fresh-cut apple and potato tissue, respectively	Bußler et al. (Bußler et al. 2015)
DBD and gliding arc plasma	POD from tomato	Air and helium; 1, 2, 3, 4, 5, and 6 min.	- Decrease in enzyme activity from 100 to 3.71% at 7 min - No nutrient destruction was reported	Khani, et al. (2017)
Low-temperature ACP	Lysozyme from egg white	Ambient air, He, O_2, He-O_2; 0, 5, 10, 15, 20, 25, and 30 min exposure time	- Decrease in enzymatic activity - Increase in molecular weight - Change in secondary structure	Takai et al. (2012)
Low frequency DBD	Fresh-cut apples	Ambient air; 10, 20, and 30 min exposure time	- Linear decrease in residual activity with increase in treatment time - Significant decrease in enzymatic browning	Tappi et al. (2014)
RF-APGD plasma	Lipase from *candida*	Helium gas; 0, 10, 20, 30, 40, and 50 s exposure time	- Increase lipase activity after 1 min - Change in secondary structure	Li et al. (2011)

other samples, which is an indication of lipid oxidation (Kim et al. 2013). These observed increases were attributed to free radicals, which are the precursors of lipid hydroperoxides produced as a result of plasma treatment. Similarly, bacon samples exposed to helium/oxygen plasma showed higher TBARS values after 7 days of storage than untreated samples. This result was possibly due to variations in the mixture of gas used, which were linked to different fat content and fatty acid composition within samples purchased from various market sources (Kim et al. 2011). Jayasena et al. (2015) had the same opinion; their pork butt samples showed lower TBARS values compared to a beef loin sample after 10 min of flexible thin-layer DBD exposure. In ready-to-eat meat (Bresaola), lipid oxidation was reported to increase significantly with power, treatments, and storage time. Upon comparison with the control samples, the increase was most pronounced at 5 °C storage temperature after 1 and 14 days. For the control, a slight increase was observed, which was linked to high oxygen concentration (stored in 30% oxygen), which might have instigated some degree of lipid oxidation (Rød et al. 2012).

Van Durme et al. studied the acceleration of lipid oxidation using an RF-plasma jet as opposed to a thermal-based treatment. The researchers employed gas chromatography-mass spectrometry (GC-MS) to measure volatile compounds after plasma exposure. After plasma treatment of vegetable oil, aldehydes and 2-pentyl furan were formed via the action of

Table 4 NTP impact on lipids

Plasma type	Lipid source	Treatment parameters	Observed results after treatment	References
Encapsulated DBD plasma	Milk	Atmospheric air; 250 W; 15 kHz; 5 and 10 min	- Slight increase in TBARS	Kim et al. (2015)
Plasma corona discharge		Atmospheric air; 90 mA; 3, 6, 9, 12, 15, and 20 min; 35 °C treatment temp	- Slight increase lipid composition - Significant changes in other organic compounds	Korachi et al. (2015)
DBD plasma	Pork loin	99.9% He, 0.3% O_2; 3 kV; 30 Hz;	- Higher TBARS values in He + O_2 treated samples than in O_2 treated samples	Kim et al. (2013)
Atmospheric pressure plasma (APP)	Bacon	He, He + O_2; 60 or 90 s exposure time; 75, 100, and 125 W; 3-mm electrode distance	- Higher TBARS values in He + O_2 treatment combination was reported after 7-daystorage.	Kim et al. (2011)
Flexible thin-layer DBD	Pork butt and beef loin	Atmospheric air containing N_2 and O_2; 0, 2.5, 5, 7.5, and 10 min	- The pork butt samples recorded lower TBARS values than the beef loin samples after 10-min exposure	Jayasena et al. (2015)
DBD plasma	Meat	70% Ar and 30% O_2; 27.8 kHz; 27 kV; 15.5, 31, and 62 W power; 0, 2, 5, 10, 20, and 60 s exposure time	- Significant increase in TBARS was observed in treated samples than the control after 1 and 14 days of storage at 5 °C - TBARS increases with increase in power, treatments and storage time	Rød et al. (2012)
RF plasma jet	Vegetable oil	O_2, Ar gas; 13.56 MHz RF power; 25 W power	- Formation aldehydes and 2-pentyl furan as secondary lipid oxidation product	Van Durme et al. (2014)
Cold plasma	Wheat flour	Atmospheric air; 15 and 20 V; 9 kHz; 40 ± 1 and 90 ± 1 W; 60 and 120 s treatment time	- Increase in hydroperoxide value and head space n-hexanal with increased treatment time and voltage	Bahrami et al. (2016)
DBD plasma jet	Fish oil	Ar + 0.6% O_2 gas; 6 kV; 50 50 kHz; 128 mA	- 2-propenal, 2- pentenal, and heptanal were formed as lipid oxidation by-products	Vandamme et al. (2015)

atomic oxygen and singlet oxygen, respectively (Van Durme et al. 2014). In a related study, a DBD plasma jet (Ar/0.6% O_2) was used on fish oil instead to fast-track lipid oxidation. HS-SPME-GC-MS analysis after plasma treatment showed that compounds such as 2-propenal, 2-pentenal, and heptanal were the secondary lipid oxidation products (Vandamme et al. 2015).

The modification of biological chemistry and physical surface properties of wheat flour were reported by Bahrami et al. (2016). In addition to a reduction in total free fatty acids and phospholipids after plasma treatments at high voltage, the hydroperoxide value and head space n-hexanal, which are lipid oxidation by-products, increased with increasing voltage and treatment times (Bahrami et al. 2016). Soybean oil was subjected to high-voltage atmospheric cold plasma for 12 h, using hydrogen and nitrogen as the process gas to produce partially hydrogenated soybean oil free of trans-fatty acids. The increase in saturated fatty acids and monounsaturated fatty acids was 12 and 4.6%, respectively. A decrease in polyunsaturated fatty acids of 16.2% was recorded after plasma treatment. Optical emission spectroscopy shows that atomic hydrogen species were likely responsible for the plasma-induced hydrogenation. The absence of trans-fatty acids in the end product was attributed to atomic hydrogen species attaching to the unsaturated fatty acids thereby changing the chemical structure of the soybean oil by converting the C=C bonds to single bonds (Yepez and Keener 2016). NTP has proven to be an emerging technology with immense potential. Apart from its effects in microbial inactivation, it has also shown promise in a variety of food industry applications.

Limitations and Challenges of NTP Technology

One of the limitations of NTP processing is the increase in lipid oxidation. Reports of heightened peroxide values in some food materials such as wheat flour, walnuts, peanuts, bresaola, and oil after long treatment times at high power is of major concern and needs to be elucidated in future works (Bahrami et al. 2016; Rød et al. 2012; Thirumdas et al. 2014). This finding might be due to plasma RS such as ozone and oxygen that oxidize the lipid molecules. Other concerns include color and pH changes in encapsulated DBD-treated milk after 10 min (Kim et al. 2015). Reduction in firmness and discoloration of fruits and vegetables were also highlighted.

The herculean task of obtaining GRAS status from authorities impedes the industrialization of NTP. This is due to nonuniform optimized food product treatment processes. Achieving such a feat may have a long way to go as different food matrices require unique optimized process parameters due to plasma reaction chemistry related to the food under treatment. The final product must be benign and satisfy established regulations.

NTP is still at the novice stage, designing industrial-scale equipment for the treatment of food requires the remaining issues are ironed out. For example, NTP chemistry with air plasma (nitrogen, oxygen, water vapor), involves 500 simultaneous chemical reactions at different stages of nanoseconds, microseconds, milliseconds, and seconds, thus leading to the generation of more than 75 unique plasma-chemical species (Misra et al. 2016b). The need to harmonize and standardize the plasma analytics is another issue to be considered in accelerating the industrial development of plasma equipment.

The scarcity of literature about the economic analysis of NTP technology is another field of future research. Notwithstanding, the technology has proven to be cheap, especially when atmospheric air is used as the process gas rather than the more expensive noble gases. Atmospheric air has proven to be a universal gas for NTP processing in a majority of fruits, vegetables, cereals, and starch and starchy foods, as well as proteins. Noble gases could be restricted to processing high-value foods and functionalized ingredients due to their expensive nature. NTP designed for the industrial scale should be capable of ionizing air in larger gaps at lower energy consumption, while maintaining the integrity of food product safety and profit margins. The energy costs derived from the implementation of NTP at the industrial scale require thorough evaluation. The additional power consumption might be challenging when replacing conventional food processing with NTP, although additional energy costs might be overcome by other benefits resulting from NTP application. From this viewpoint, the decision to choose from NTP or existing sterilization devices will be left to food processors selecting the method that is less expensive, simple to operate, and uses better equipment. This is a challenging task that all stakeholders should vie for in order for NTP to be commercially accepted.

Conclusion

In this study, the influence of NTP on food microstructure and constituents was reviewed. The review shows that NTP can modify starches via surface etching due to the corrosion effect of plasma RS. Depolymerization, cross-linking, and addition or abstraction of functionality in the molecular chains results in alteration of the rheological behavior of starch biopolymers. Protein modification occurs through changes in secondary structures such as oxidation and ablation of the biopolymer, cross-linking, and addition of new functional groups. Other changes in rheological behavior occur, such as gel structure coarsening, increases or decreases of moduli and increases in protein binding ability among others. All of these properties were linked to chemical reactions with plasma RS. Paradoxically, among the various methods documented for computing lipid oxidation, the measurement of secondary volatile oxidation products was the more realistic.

Nevertheless, some aspects are still lacking details as to how plasma chemistry causes some of the alterations to food macromolecules. Thus, there is a need to unify standard protocols for plasma analytics in various food matrices due to their complexity. In particular, optimization of plasma-factor driven processes in various food systems remain a key challenge. Achieving such a feat will open up a long awaited industrial demand for NTP applications in various food systems.

Acknowledgements This study was supported by the National Key R & D Program of China (2017YFD0400103). The graduate study was funded by the China Scholarship Council under the Ministry of Education of the People's Republic of China.

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