

EFFECT OF PULSED ELECTRIC FIELD ON STRUCTURAL MODIFICATION AND PHYSICOCHEMICAL PROPERTIES OF STARCH-BASED EDIBLE FILMS

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Abstract:

Starch-based edible film is a novel packaging material that has gained much recognition and attention in the food industry. Its remarkable functional property like microbial deactivation without posing harmful effect on food materials is remarkable. The application of pulsed electric field (PEF) treatment to starch-based edible film is fundamental to ensuring physicochemical changes and structural modifications of films for the purpose of food safety and handling. However, the effectiveness of PEF is affected by process and product parameters, thus information on the effects of PEF treatment on structural modifications and physicochemical properties of starch-based edible film is useful to the food industry.

This review provided an overview of the effects of PEF treatment on structural modifications and physicochemical properties such as density, solubility, water activity (a_w) and stability of starch-based edible films. The significance of starch-based edible films to the food industry with a focus on potato, cassava, yam and taro starch sources is summarized. The PEF treatment and factors related to process and product parameters affecting its performance and its effects on structural and physicochemical properties of starch-based edible films were succinctly discussed. PEF treatment can cause microbial deactivation, influence the structural and physicochemical properties and produce edible films with much functionality. The future trends of PEF treatment on starch-based edible films should mainly focus on combining other physical treatment as an integrated way of improving the potentials of PEF towards development of a wider range of functionalized films.

Keywords: Packaging material, food safety, PEF treatment, edible film, microbial deactivation, physicochemical properties, physical treatment

1.0 Introduction

The increase in the world population and demand for safe and fresh like foods have made pulsed electric field (PEF) treatment most prominent technology for microbial deactivation in biological materials such as edible films (Kramer, 2009; Han *et al.*, 2009; Han *et al.*, 2012; Wihodo & Moraru, 2015; Hong *et al.*, 2016; Gutiérrez, 2017; Gitonga *et al.*, 2018; Zhu, 2018). The introduction of edible films in handling, transportation and packages containing functional food additives rich in polysaccharides is becoming popular. Polysaccharides (such as cellulose, starch, chitosan, and pectin) are essential edible materials contributing to the reduction of food spoilage and posed a great potential in food handling (Lagarón *et al.*, 2016). According to consumer trends nowadays, the concept of handling fresh food is to have the food contained in a package where its physicochemical property and volatile components can be preserved during storage. High percentage of food products are reportedly lost due to microorganisms activities and poor handling during transportation and storage (FAO, 2011; Yu, 2016; Bradford *et al.*, 2018). The infestation by

microorganisms and inefficient storage systems can cause breakdown of active compounds present in food materials (Odunola *et al.*, 2018). In addition, the infested food materials are prone to food poisoning when eaten by humans that could cause outbreak of food borne disease and consequently cause serious health challenges. Food poisoning and its consequential effect on humans' health have therefore made provision of safe food a major concern among food scientists and food engineers. On the other hand, packaging materials such as polyethylene, plastics and nylon made from hydrocarbon-based compounds had been used for handling ready-to-eat foods, but their non-degradable nature, affinity with foods and variability in conditions in-packed foods have limited their application in the food industry. However, starch-based edible film is an alternative novel packaging material used for reducing quality loss through modification of internal atmosphere in-packed foods. Such unique method of packaging explored the potential of edible material from plant products and aids protection for fresh produce by limiting moisture migration, gas exchange and oxidation at varying

atmospheric conditions (Kramer, 2009; Kumari *et al.*, 2017). The accurate use of starch-based films in food product is controlled by the regulation standards and also meets environmental issues, thereby eliminating the problems associated with disposal of non-biodegradable and non-renewable packaging materials (Atarés & Chiralt, 2016). Starch is very relevant in food packaging industry and in biomedical related field due to their potentials in carrying active compounds (Hassan *et al.*, 2018). Although, its use is relatively new in food industries, research in this field has increased in the last few years and it has been shown that starch-based edible films show a good performance in food protection (Kenny *et al.*, 2016). During its production, several reactions take place within the film matrix that gives rise to microbial reaction which consequently affects the films (Kenny *et al.*, 2016; Gutiérrez, 2017). This unwanted reaction requires heat treatment for microbial deactivation to enhance quality assurance. Traditionally, thermal heat treatment affects food structures and causes detrimental effect on the physicochemical property. Nowadays, non-thermal processing methods capable of microbial deactivation have been developed (Sun *et al.*, 2014; Wihodo & Moraru 2015; Lim *et al.*, 2015; Zeng *et al.*, 2016). Among many others, PEF technique has advantages over thermal processing methods due to its potential in reducing microorganisms activity while maintaining the structural and physicochemical properties of food materials been treated. It involves the application of pulses of high voltage to food materials placed between two electrodes (Wihodo & Moraru, 2015; Gitonga *et al.*, 2018). Several findings have shown that PEF with field strength (up to 50 kV cm⁻¹) and 0 to 50 µs pulses are capable of deactivating microorganisms in starch, lipid and protein (Xiang, 2008; Xiang *et al.*, 2011; Wihodo & Moraru 2015; Hong *et al.*, 2016). Figure 1 shows a typical arrangement of pulsed electric field system.

Meanwhile, the introduction of electric field on the surface of starch-based edible film induced “particle orientation effect” resulting in the absorption of the carbon backbone of the starch molecule, thus affecting the structural and physicochemical properties (reference). This review however, shows the current trends and research findings on the effects of pulsed electric field treatment on structural modification and functional properties of starch-based edible films.

1.1 Significance of starch-based edible films

The significance of starch-based edible films consist not only in their biodegradability, but also in their potentials in preserving food materials from spoilage and simultaneously serving as functional food

supplements (Azeredo, 2009; Olivato *et al.*, 2012). Conventional materials used in food packaging produced from polymers of hydrocarbon posed serious environmental problems such as air and water pollution, resulting into serious health challenges to humans and animals (Marsh & Bugusu, 2007; Sorrentino *et al.*, 2007; Meneely *et al.*, 2018; Poyatos-Racionero *et al.*, 2018; Khan & Malik, 2019). This awareness has inferred a shift towards the development of biodegradable films. A new pedigree to improving the quality and shelf life of fresh food and enhanced functional ingredients employs the use of starch-based edible films. Edible films have thickness in the range of 100-254 µm and can be wrapped on the surface of the food materials. It can be used for different postharvest purposes, depending on areas of application, such as presenting a barrier to moisture migration and gas flow (O₂ and CO₂), inhibition of oxidation process and microbial activity, prevention of diffusion of volatile compounds, thus preserving functional properties (such as antioxidants and antimicrobials) (Lim *et al.*, 2015). Starch-based edible film can serve as packaging or handling material as well as food material (Sorrentino *et al.*, 2007). Also, natural additives with active agents such as ethylene scavengers, CO₂ emitters and O₂ scavengers can be incorporated into starch-based edible films to enhance the concentration of active components required for specific purposes. Raw materials for the production of starch-based edible films can be obtained basically from yam, cassava, potato and other biological materials containing starch concentrate. Among these raw materials, yam and cassava are widely used because they satisfied major aspects of film forming, including excellent barrier, low cost, odorless, colorless, and non-toxic. Starch can be used for the production of edible film for shelf life extension of fruit and vegetables, sea foods and meat packaging, but its low strength, sensitivity to moisture and non-stabilized structure when exposed to varying atmospheric conditions has limited its use in food packaging application (Xie *et al.*, 2013; Dang & Yoksan 2015). In order to overcome these limitations, recent researches had focused on various modifications that can improve the functional and structural properties of starch for films production. The addition of starch to other polysaccharides and biological materials such as lipids, protein, cellulose, algae, chitosan, and pectin had proved to be a unique method towards achieving improved films properties (Al-Naamani *et al.*, 2016; Bonilla & Sobral, 2016; Saberi & Chockchaisawasdee, 2017; Barikloo & Ahmadi, 2018).

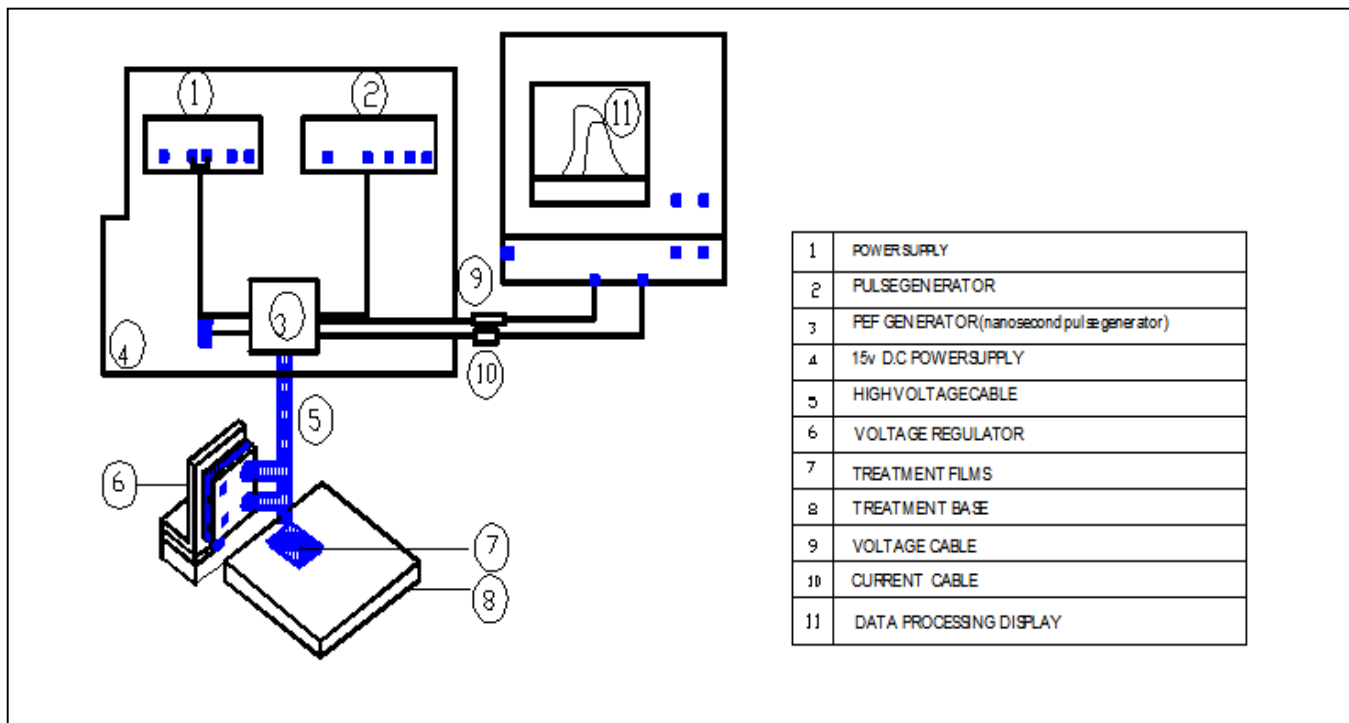


Figure 1: Typical arrangement of pulsed electric field system

1.2 PEF treatment and factors affecting its performance

1.2.1 Factors related to process parameters

PEF treatment is considered as a reliable non-thermal technique used in deactivation of microorganisms without tampering with either the structural or physicochemical properties of foods. During PEF treatment, food items are exposed to ambient or slightly above temperature within microseconds. The effect of PEF treatment on structural and functional properties of starch-based edible films is associated with the flow of electric field through the films matrix, increasing the internal energy and cause the induction of particle orientation effect thereby, resulting in absorption of the carbon backbone of the starch molecule (Hu *et al.*, 2015; Sanyang *et al.*, 2015). This perturbation phenomenon has a great potential in modifying the configuration of the carbon chains present in starch. Meanwhile, the electric field can be applied continuously depending on the treatment time and desired effects. Continuous PEF operational mode, treatment temperature and time and electric field strength (E) are the important factors affecting PEF. Basically, PEF processing technology consists of high voltage power supply, an energy capacitor bank and a charging resistor. During operation, energy derived from high voltage power supply is stored in the capacitor bank which is transferred into the film matrix

to generate the necessary electric field. The level of pulses released into the film matrix depends on the energy and the capacitance of the storage and the two are related through an input voltage as given in eqns. 1 and 2, according to Xiang (2008).

$$C = \frac{\tau}{R} = \frac{\tau \sigma A}{d} \quad (1)$$

Where:

C = Capacitance of the energy storage capacitor (F)

τ = Pulse width (microseconds)

R = Electrical resistance (Ω)

σ = Electrical conductivity of films ($s m^{-1}$)

A = Area of electrode surface (m^2)

d = gap between the electrodes (m)

$$Q = \frac{1}{2} CV^2 \quad (2)$$

Q = Energy stored in a capacitor (Jm^{-3})

V = charging voltage (kV)

Meanwhile, energy stored in the capacitor can be released over the film matrix through a gap between the electrodes at a very high level power. The high voltage can be switched on or off depending on signal received in the treatment chamber. Whenever, the capacitor is fully charged with adequate energy received on the activated signal, the high voltage is switched off to allow the charges in the charged capacitor to flow through the films matrix. Cold water is used to dissipate the heat energy generated by the electrodes in order to prevent undesirable effects in the

treatment chamber. Some processing factors that could possibly affect the operation of PEF include; electric field intensity, number of pulses and pulses waveforms, pulse width and treatment time (Han *et al.*, 2009). Han *et al.* (2012) showed that increase in electric field strength and treatment time supported the transformation of starch crystalline structure to amorphous structure with lowest peak viscosity (560 BU) observed at 50 kV cm⁻¹.

The structural modification of starch was initiated at high field intensity through a progression at enhanced PEF treatment which demonstrated the ability of starch granule to swell freely before breaking down. Whereas, a field strength of 2 kV cm⁻¹ ($Q = 1.7 \times 10^8$ Jm⁻³, $t_{exp} = 74.5$ s) was used to modify the degree of substitution and pasting temperature (Hong *et al.*, 2016). Similarly, PEF effect on potato starch showed a shorter time of exposure (t_{exp} 806 μ s) and higher field strength (40 kV cm⁻¹) and enhanced structural modifications with many surface pits on starch granules (Han *et al.*, 2009). More studies are needed on the intra-granular molecular arrangement of the starch granules under different PEF treatment. Regarding temperature effect, heating is linked with order-disorder transitions in the structural elements due to the influence on melting of the structural units and formation of entanglements in carbon chain backbone (Sun *et al.*, 2014). The order-disorder effect is significantly depended on the quantity of moisture present in starch. For example, application of high temperature modified phase change (e.g. emulsification and gelatinization) properties of starch at different moisture levels (Sun *et al.*, 2014). The process of heating starch in the presence of moisture allows hydroxyl group of amylose (linear in structure) and amylopectin (branched in structure) to react with water and thus formed a weak crystalline structure (Chung *et al.*, 2009). However, this influences chain separation and enhances mobility of molecular chain and helical structures, resulting in structural alteration of amorphous and crystalline region in starch granules (Bemiller & Huber, 2015). Meanwhile, there exists a link between temperature change and energy input. Han *et al.* (2012) demonstrated that the energy responsible for the changes in physicochemical properties of tapioca starch was positively related to temperature change. Also, Hong *et al.* (2016) demonstrated the effect of PEF assisted acetylation of potato starch. The findings showed that a progression in the electric field intensity from 2 to 5.5 kV cm⁻¹ resulted in more significant heating of potato starch from 30 to 39°C. The high field strength (higher thermal heating condition) resulted in the highest level of substitution of -OH by the acetyl group. In soy protein treated with PEF technique, field intensity of 35 kV cm⁻¹ was capable of changing the structure of

amino acid and β sheet chain through a phenomenon that leads to polarization of dipole moments between carbon-oxygen atom and disulfide bonding (Li, 2012; Yamasaki *et al.*, 2011).

1.3 Factors related to product parameters

Starch has different crystalline structures based on their botanical origin and possesses different amylose and amylopectin contents which have the capacity to influence its physical and chemical properties. The amylose and amylopectin in starch matrix have great potentials of protecting some chemical species against PEF treatment. For example, the crystalline structure of starch has a potential to alternate high or low density lamella of applied field to a notable distance (like 9-10 nm) (Vermeulen *et al.*, 2006). This effect was due to higher degree of substitution during PEF assisted acetylation of cassava starch in the presence of 10% (w/w) acetic anhydride after PEF treatment at 2 kV cm⁻¹ for 51.0 μ m pulses (Hong *et al.*, 2016). In the same vein, amylose and amylopectin contents in starch-based films can influence properties like electrical conductivity (Ec), pH and ionic strength.

The Ec of a starch is an important parameter that determines the capability of the film to allow the passage of electric current. Starch from different botanical origins has different Ec. Typically, film with high Ec allows passage of low electric field across the treatment chamber. This is not ideal for PEF treatment because Ec has a linear relationship with ionic strength of a material. For instance, high ionic strength of cassava starch resulted in decreased activation rate of microorganisms such as *Listeria monocytogenes* and *Salmonella* spp and caused weakening of the membrane structure.

Several studies had reported on the effect of PEF treatment on the molecular size of starch from different sources (Han *et al.*, 2012; Hong *et al.*, 2016; Zeng *et al.*, 2016). Han *et al.* (2012) demonstrated that PEF treatment of 50 kV cm⁻¹ significantly reduced the molecular weight of maize starch from 102 x 10⁶ to 13 x 10⁶ g mol⁻¹, whereas the molecular weight of waxy rice was slightly affected at the same field intensity (Zeng *et al.*, 2016). These observations were due to the differences in the Ec. However, more studies are required on different starch sources for robust data and information. Notwithstanding, whenever starch crystallinity is considered, the increase or decrease the degree of starch crystallinity depends on starch type and PEF conditions. Increase in electric field intensity gave a decrease in starch crystallinity, thus induced partial gelatinization of the molecular chain (Hong *et al.*, 2015; 2016).

2.0 Starch-based edible films and their structural and physicochemical properties

2.1 Effect of PEF treatment on structural properties

Effect of PEF treatment on structural properties of starch has been investigated by many researchers (Han *et al.*, 2009, 2012; Hong *et al.*, 2015; Xiang *et al.*, 2011; Zeng *et al.*, 2016). The stability of properties such as viscosity, gelatinization, crystallinity and texture are directly related to electric field strength (up to 50 kV cm⁻¹). This high electric field strength effect tends to decrease the viscosity of starch during pasting. For example, Han *et al.* (2009) treated maize starch with PEF at 50 kV cm⁻¹ and reported a reduction in viscosity from 335 to 250 BU during pasting. However, the Wide-angle X-ray diffraction analysis (WAXD) showed that PEF treatment had no significant effect on polymorph typed starch while decreasing the degree of crystallinity (Han *et al.*, 2012). In addition, PEF treatment posed some modifications on gel resulting from starch during pasting. For instance, Differential Scanning Calorimeter (DSC) had showed that PEF (at 50 kV cm⁻¹) tends to reduce the enthalpy and gelatinization temperature (73.5 to 71.5 °C) of maize starch (Han *et al.*, 2009). The reduction in gelatinization temperature depicts the capability of PEF to differentially disrupt the crystalline structure. Also, Zeng *et al.* (2016) performed a small angle X-ray scattering (SAXS) analysis on rice starch and reported that PEF treatment decreased the lamellar thickness of starch, though the exact changes and values were not stated.

In essence, the change in structure induced by PEF could cause changes in physiochemical properties of starch-based edible films.

2.2 Effect of PEF treatment on physicochemical properties

2.2.1 Density

Density is an important property of starch-based edible films that can be affected by PEF processing. Based on origin, starch differs in density depending on the composition and molecular weight (Pelissari *et al.*, 2013). The degree of amylose content in a starch determines its density; such that the higher the amylose content, the denser the films. Among numerous starch sources, cassava starch is known to have the higher amylose content with more compact structures. Jose *et al.* (2012) argued that more compact structures lead to less dense films. However, the effect of PEF treatment differs among starches. PEF treatment decreased the density of cassava starch-based films through substitution of carbon back-bone chain which led to the decrease in their molecular weight (Gutiérrez & González, 2016). Whereas, the density of taro starch-based films is significantly high due to a decrease in

availability of hydrogen group associated with electric field effect (Gutiérrez, 2017).

2.2.2 Solubility

Solubility is another parameter of starch-based edible films influenced by PEF treatment. It shows the integrity of films to water. For instance, the more soluble films are, the lesser their resistant to water and vice-versa (Morales *et al.*, 2014; Soledad *et al.*, 2015). Meanwhile, the molecular weight and hydrogen bond of the hydroxyl group of starch has a correlation to its solubility, such that, the stronger the hydrogen bond, the lesser its molecular weight, and the higher its resistance to water (Gutiérrez, 2017). Henceforth, taro starch treated with PEF induced polymerization and gave a less soluble film because of their lower amylose content. Whereas, the water solubility of cassava starch films treated with PEF was significantly higher ($p \leq 0.05$). This was possibly due to the field effect, which generated the fragmentation of the starch chains, thus reducing film molecular weight and increasing solubility (Gutiérrez & González, 2016; Hong *et al.*, 2015).

2.2.3 Water activity (a_w)

Water activity (a_w) is another important parameter influenced by PEF treatment and it is directly related to the amount of moisture present in starch-based edible films. Edible films made from taro starch is said to have greater a_w due to its low amylose content (Gutiérrez, 2017; Gutiérrez & González, 2016). PEF treated taro starch-based films resulted in a significant decrease in a_w attributed to the rearrangement of the dipoles, thus preventing water adsorption. Similar results were reported for native and cross-linked edible films made from native and modified yam and cassava starch (Soledad *et al.*, 2015). Little wonder, cassava starch films with electric field treatment gave a slightly insignificance ($p \geq 0.05$) increase in a_w . However, for safety and quality control, more data should be generated to justify that any increase in a_w value will not support microbial growth.

2.2.4 Film stability

The stability of edible films to either acidic or alkaline medium indicates its tendency to be used as packaging or handling material for foods without having affinity with them. PEF treated films made from potato, cassava and yam starches were stable against changes in pH for more than 24 days (Morales *et al.*, 2014; Soledad *et al.*, 2015). This showed that treated films can be used for packaging acidic and citrus fruits without harmful effect on the products. More so, the susceptibility of treated starches to alkaline medium demonstrates their stability in the medium. Starch with high amylose contents such as cassava starch and yam starch can withstand alkaline solution; therefore, they are more stable and serve as good polymeric materials

for food packaging (Gutiérrez *et al.*, 2015a, 2015b). Similarly, the stability of treated edible films to pH range of 1 to 8 made them suitable for transporting probiotic bacteria into the small intestines without causing any health challenge (Piermaria *et al.*, 2015). Of course, less stable films occur as a result of the disintegration of the starch molecules in an alkaline solution caused by thermolysis reaction between the electric field, sodium hydroxide and the hydroxyl groups of starch molecules, therefore weakening the hydrogen bonds between the starch macromolecules, and thus facilitating swelling (Hu *et al.*, 2009).

Cassava starch films treated with pulsed light demonstrated low stability due to depolymerisation effect on starch which exposes the starch chains, and made them react with the hydroxyl ions in the medium, thus resulting into swollen films. On the other hand, taro starch films treated with pulsed light showed high stability due to cross linking of starch chains making the intermolecular forces stronger, resulting in less reactive in alkaline medium, and thus become less swollen.

Table 1: Effect of PEF treatment on some physicochemical properties of different thermo plastic starch films

Properties Sample	Moisture (%)	Solubility (%)	Crystallinity (%)	a_w	Density (kg/m ³)	Stability
C	20 ± 1 ^b	38 ± 1 ^a	19 ± 0.2 ^c	0.453 ± 0.002 ^a	1.11 ± 0.02 ^d	
C + PEF	17 ± 1 ^a	41 ± 1 ^b	21 ± 0.2 ^d	0.455 ± 0.001 ^a	1.05 ± 0.03 ^c	Less stable
T	29 ± 1 ^d	52 ± 1 ^d	10 ± 0.1 ^a	0.488 ± 0.001 ^c	0.94 ± 0.03 ^a	
T + PEF	26 ± 1 ^c	49 ± 1 ^c	11 ± 0.1 ^b	0.484 ± 0.001 ^b	0.99 ± 0.01 ^b	More stable

Adapted from Gutierrez *et al.*, (2015a and 2015b). Same letters in the same row indicate no significant difference ($p \leq 0.05$). Thermoplastic starch films: Cassava control (C), cassava treated with pulsed light (C + PEF), Taro control (T) and taro treated with pulsed light (T + PEF).

3.0 Challenges and Future Work

PEF is a non-thermal technology that can influence structural and physicochemical properties of starch-based edible films. Specifically, PEF treatment has a great potential to deactivate microbes, modify structure and positively affect the density, solubility, a_w , and stability of starch-based edible films. However, the pulsed light phenomenon that occur during PEF treatment proved efficient in improving structural modifications while deactivating microbes during the preparation of starch-based films. In addition, the introduction of active components such as lipids, protein, cellulose, chitosan and pectin can greatly influence the structural and physicochemical properties and produce films with much functionality depending on experimental conditions and film source. Although, PEF-treatment has great impacts on starch-based edible films for food safety and handling, challenging issues as highlighted below still remain and should be addressed in the future studies.

(1) In order to enhance PEF treatment, effort should be made to understand the electric field flow through the films matrix, thus gaining better insight on the internal energy, energy loss as well as

induction of particle orientation effect. A technique that can be explored is the use of flow meter. In addition, effort should be made to ensure stability of starch with polysaccharides and biological materials such as lipids, protein and cellulose because these bioactive materials favorably add value to the films.

(2) Application of PEF treatment on edible films is relatively new and has only been realized in small scale, majorly in the laboratory. No industrial settings based on the PEF treatment on edible films modifications have been reported yet. More studies on transforming batch process to continuous and pilot scale process are needed. Also, cost estimation (e.g. energy consumption and setting up the facility on industrial scale) and technical feasibility needs careful attention.

(3) In conclusion, PEF (up to 50 kV/cm field strength and 0 - 50 microseconds pulse width) had significant effect on structural modification and physicochemical properties of starch-based edible films. Some other physical treatments such as ultrasound, gamma irradiation or electro-magnetic field treatments should also be tested on edible films and checked if the same functionality can be obtained. However, for process development and optimization,

PEF can be combined with any of the physical techniques to create edible films with a wider range of functionalities.

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