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Experimental Thesis

**Effect of Edible Packaging on the Quality of Healthier Fruit Juice
Powder**

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Master Degree in Nutrition and Food Technology

**Effect of Edible Packaging on the Quality of Healthier Fruit Juice
Powder**

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III

Dedication

To all my family, friends and colleagues who are so dear to me.

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أنا الموقع أدناه، مقدّم الرسالة التي تحمل العنوان:

Effect of Edible Packaging on the Quality of Healthier Fruit Juice Powder

أقر بأن ما اشتملت عليه هذه الأطروحة إنما هو نتاج جهدي الخاص، باستثناء ما تمت الإشارة إليه حيثما ورد. وأن هذه الرسالة كاملة، أو أي جزء منها لم يقدم من قبل لنيل أي درجة أو لقب علمي أو بحثي لدى أي مؤسسة تعليمية أو بحثية أخرى.

Declaration

The work provided in this thesis, unless otherwise referenced, is the researcher's own work, and has not been submitted elsewhere for any other degree or qualification.

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التوقيع:

Date: 22-07-2020

التاريخ:

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XI
**Effect of Edible Packaging on the Quality of Healthier
Fruit Juice Powder**

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Abstract

Rapid technology world driven, environment sustainability, human health and safety issues, food industry innovation and cost reduction plans; all of them form a matrix of challenges for food and nutrition technologist.

Proposed idea of using edible film, can contribute clearly in industry polices by providing a part of solution for many related issues to food industry.

Fruit juice powder, easy, soluble and ready to use packaged with edible films has successfully produced; this product introduces multifunction food for both human (consumer), food industry sector and the environment. The results showed that wrapping the fruit juice powder with pectin film are completely solubilized in only 30 S at 25 °C, while 57 S when cold water 4 °C was used.

Healthier fruit juice powder will be modified in future by integration bioactive compounds in edible film that improve the characterizations of the product by increasing the shelf life as well as reduce the utilization of food additives which will contribute positively in human health.

Chapter One

Introduction

The world main concern oriented towards sustainability when performing different activities along supply chain. So that; it is a must to create efficient food packaging that meets global economic and environmental challenges.

Nowadays packaging represents a crucial role in food preservation and protection from the external environment such as air, water ...etc. and pathogens that cause food spoilage and health hazard for human. The packaging trimline consciously changed to attempt consumer requirement, different materials have been used during early history,19th century until now (Figure 1). Most of plastic molded that contain our foods are not readily recyclable which create a serious waste and ‘over- packaged’ items challenge that everyone is familiar with this issue. (<https://www.recyclesmart.org/packaging>)

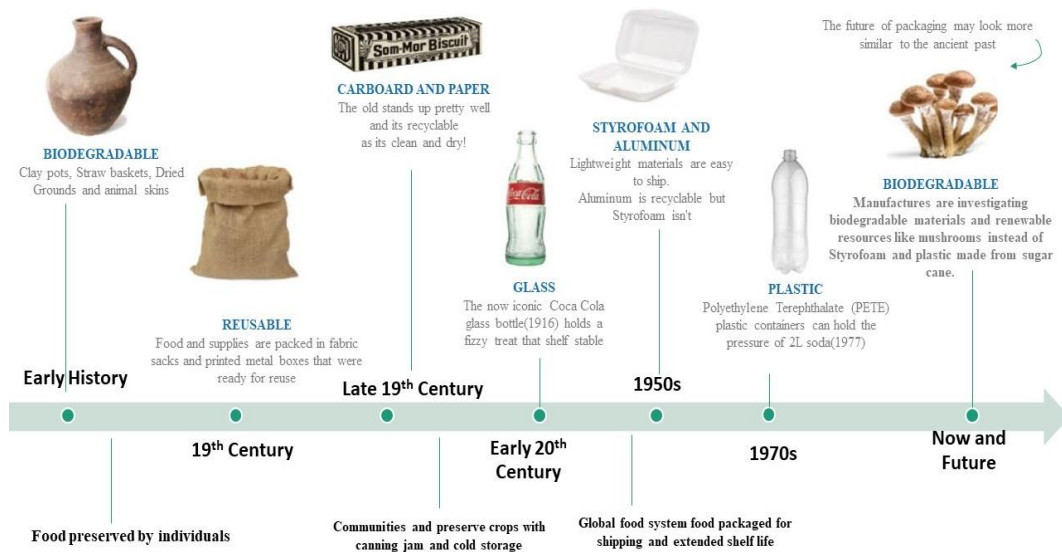


Figure 1: Packaging TimeLine. <https://www.recyclesmart.org/packaging>.

Environmental pollution due to plastic polymers is a significant challenge; whereas it's represented through disposed of plastic waste in landfills, toxins affect ecosystem of marine, air and soil (Khan and Ghouri, 2011). Landfills and oceans are the main destination of the plastic waste, whereas only less than 10% of plastics produced every year is recycled (Porta, 2019). Plastic packaging waste represent more than 15% and 25% of total solid wastes in the USA and Europe, respectively (Valdés et al., 2015).

A study published in 2015, manifests that around 49 million tons of plastic wastes were collected every year out of which 39.9% were food packaging materials (Wróblewska-Krepsztul et al., 2018) United States Environmental Protection Agency (US EPA) recorded in 2017, total plastic products generation were 35.4 million tons that is 13.2% of Municipal Solid. Waste generated. This increase of four million tons from 2010 to

2017, which it is from food packaging categories and drinking package (Umaraw and Verma, 2017).

Moreover; the relationship between environmental pollution and human health is reported negatively on mental health (Gu et al., 2020), gut microbiota (Jin et al., 2017) through several recent researches.

The production and development of new plastic products accelerated after World War II and they changed so much the modern age that, today, life without plastics would be unrecognizable. However, it led to a “throw-away culture” that has revealed this material’s negative note: many plastic products, such as plastic bags and food wrappers, are used for few minutes or hours and then trashed, yet they may persist in the environment for hundreds of years.

According to Geyer et al. 2017, 8300 million metric tons (Mt) of virgin plastics have been produced to date, and in 2015 approximately 6300 Mt of plastic waste had been generated. Moreover, according to statistical evaluations, if current production and waste management trends continue, by 2050 the plastic waste estimated to be about 12.000 Mt on the earth (Figure 2).

Cumulative plastic waste generation and disposal

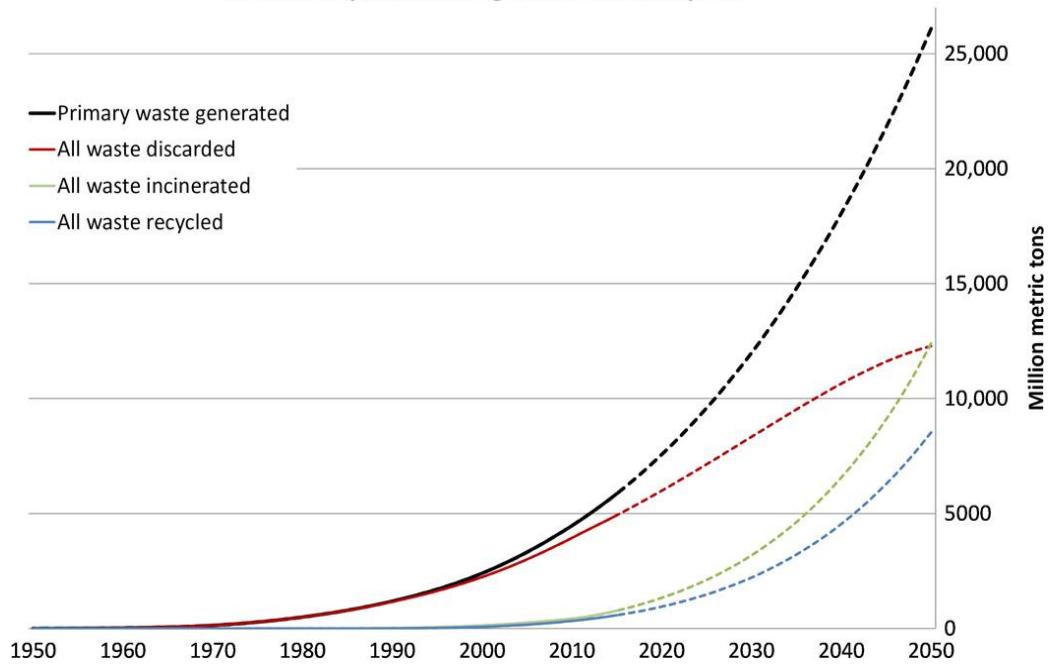


Figure 2: Cumulative plastic waste generation and disposal (in million Mt). Solid lines show historical data from 1950 to 2015; dashed lines show projections of historical trends to 2050 (from Geyer et al. 2017). 1 Mt = 1,000 kilograms. (Geyer et al. 2017).

Hence plastic pollution has become one of the most urgent environmental issues, as the increasing production of disposable plastic products runs over the world's ability to deal with them. The long-term impact and the consequences for human health are not yet fully understood, but nowadays everybody knows the main risks associated with this emergency, as the presence of microplastics in seafood, carbon emissions as a result of the production, recycling and incineration of plastic, damage to people's health and quality of life, especially in coastal environments, exposed to plastic pollution.

Rising awareness of these downsides has provoked new restrictions around the world, particularly on single-use plastics and scientists and producers are looking for alternatives, like bioplastics, materials and those

artifacts, both from renewable and fossil sources, which have the characteristic of being biodegradable and compostable. The concept of bioplastic therefore applies to those products which guarantee organic recyclability in different environments (e.g., composting, anaerobic digestion, soil) at the end of their life (Geissdoerfer et. al., 2017).

The European bioplastic (europeanbioplastics.org) have been categorized the bioplastic in three main groups with different properties (Figure 3):

1. Biobased or partially biobased non-biodegradable plastics such as PE, PP, or PET and biobased technical performance polymers such as PTT or TPC-ET;
2. Biobased and biodegradable, such as PLA and PHA or PBS;
3. Plastics based on fossil resources and are biodegradable, such as PBAT.

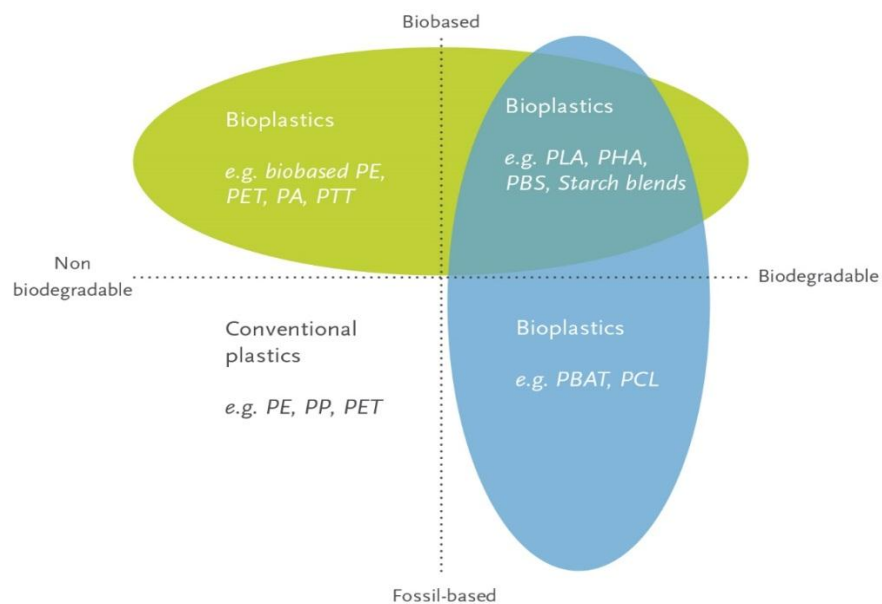


Figure 3. Bioplastics and biobased plastics (europeanbioplastics.org)

Furthermore, European Bioplastics estimate bioplastic production capacity stands at 2.1 million tons per year and is expected to grow to 2.4 million tons in 2024 (Figure 4).

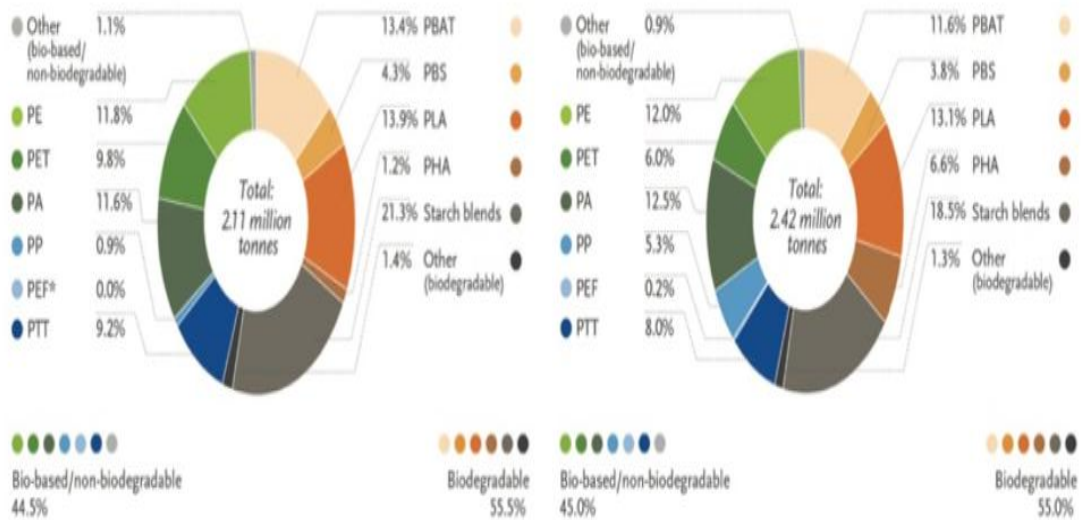


Figure 4. On the left, global production capacities of bioplastics in 2019 (by material type); on the right, global production capacities of bioplastics estimate for 2024 (by material type) (europeanbioplastics.org).

These volumes are overall contained compared to the 359 million tons of plastics produced every year worldwide, and even the growth forecasts seem inadequate to respond to the ambitious replacement programs for traditional polymers; moreover bio-based polymers are now produced for the most part with renewable sources that can compete with other applications and, in particular, with human or animal nutrition (1st generation bioplastics, coming from cereal sources). The answer to this last problem was the study and development of 2nd and 3rd generation bioplastics, made from agricultural/urban waste and from microbial fermentation, respectively, has been undersought.

In fact, the demand of more alternatives, due to the pressure exerted by the conventional plastics market, pushed the research towards the identification of new renewable resources to produce bioplastics. One of the new frontiers is represented by hydrocolloid films, which are biodegradable plastics based on biopolymers, such as polysaccharides and proteins, contained in crop wastes; these polymers represent the building block of this next-generation advanced and environmentally friendly plastics, that are mostly intended for food wrapping and mulching.

Innovate new technologies to control pollution, waste management and maintain the sustainability of the nature has become urging area for the researchers. Ecofriendly, edible packaging are spotlight of development in new multipurpose substances by their kind properties to effectively protect food along with preserving and wrapping the food item for storage and transportation, which can be consumed and biodegraded with no waste production (Valdés et al., 2015).

Edible films and coatings categorized generally based on biological materials such as proteins, lipids and polysaccharides, alone or; more often; in combination. The composition of edible film/coating affects the efficiency and stability of the packaging material. The ability of edible film to reduce respiration and transpiration rate generally maintain fruit fresh and delay fruit senescence (Tezotto-Uliana et al., 2014).

Chapter Two

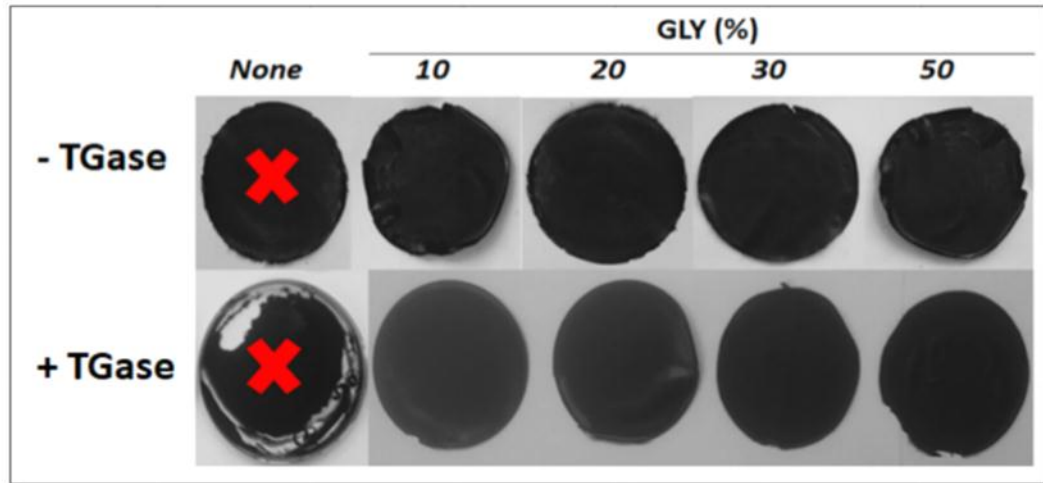
Literature Review

2.1 Edible film

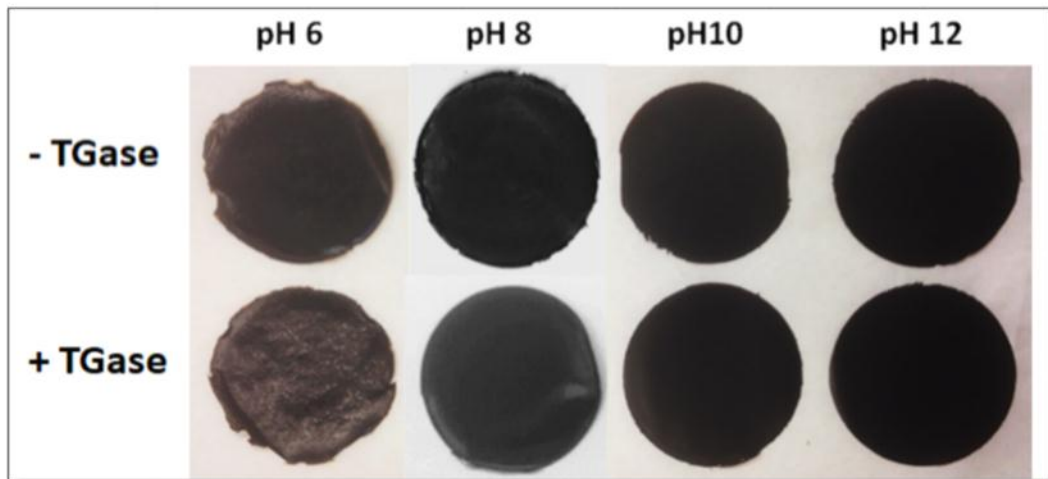
Different polymers types have been used in the formulation of edible coating or film such as pectin (Porta et al., 2016), chitosan (Sabbah et al., 2019), polyglutamic acid (Karimi et al., 2020), alginate (Guerreiro et al., 2015), galactomannans (Cerqueira et al., 2011) and wax (Ochoa et al., 2011). Pectin; which is a major component of plant cell walls; has the advantage of attractive carbohydrate structure, which render it to form good food packaging films (Banerjee et al., 2016).

The effect of blending pectin-nano chitosan (PEC-NCH) films with different ratios was studied in 2020. The results showed that PEC-NCH blend at proportions of 50:50 increased the tensile strength, reduced the water solubility to 37.5%, reduced water vapor and oxygen permeability, beside the effect on growth inhibition of *Colletotrichum gloeosporioides*, *Saccharomyces cerevisiae*, *Aspergillus Niger*, and *Escherichia coli*. These criteria could be a new approach for the active packaging of edible film to extend the product shelf life (Ngo et al. (2020). In the same year; another study performed handleable biodegradable/edible black films from *Nigella sativa* defatted seed cakes (NsDSC) obtained from a protein concentrate (PC) of NsDSC in the presence of

glycerol (GLY) as plasticizer and transglutaminase (TGase) the result showed significant increase in their mechanical strength, tensile strength (TS) and elongation at break (EB) (Figure 5) (Sabbah et al., (2020)).



(A)



(B)

Figure 5: Images of protein concentrate (PC, 400 mg) films either containing different concentrations of glycerol (GLY) and obtained at pH 8.0 (A) or containing 20% GLY and obtained at different pH values (B) after incubation of the FFSs in the absence. (Sabbah et al.,2020).

Recently, the development and characterization of fish gelatin (FG) edible films were loaded with pomegranate (*Punica granatum* L.) seed juice by-product (PSP) residue as reinforcing and antimicrobial agent. Potential antimicrobial activity against Gram positive bacteria *S. aureus* in FG films, enhanced the films stiffness, and provided good ultraviolet and visible light barrier properties, resulting in improvement in opacity, which could be useful for food with high lipid content, also improved the water resistance of the developed bio-composites due to the hydrophobic character of the residue at both concentrations (Valdés et al., 2020).

Viana et al, (2018) created a nano fibrillated bacterial cellulose (NFBC) and pectin edible films with the addition of fruit purees (mango or guava purees) as NFBC produced from *Komagataeibacter xylinus* (formerly *Gluconacetobacter xylinus* (Figure 6). The results demonstrated extraordinary differences in tensile properties, water vapor barrier and water resistance, when compared to the corresponding films without any fruit purees. Furthermore, the partial or total replacement of pectin with NFBC resulted in enhancing physical properties of edible films, making them stronger, stiffer, more resistant to water, and with improved barrier to water vapor (Viana et al., 2018).

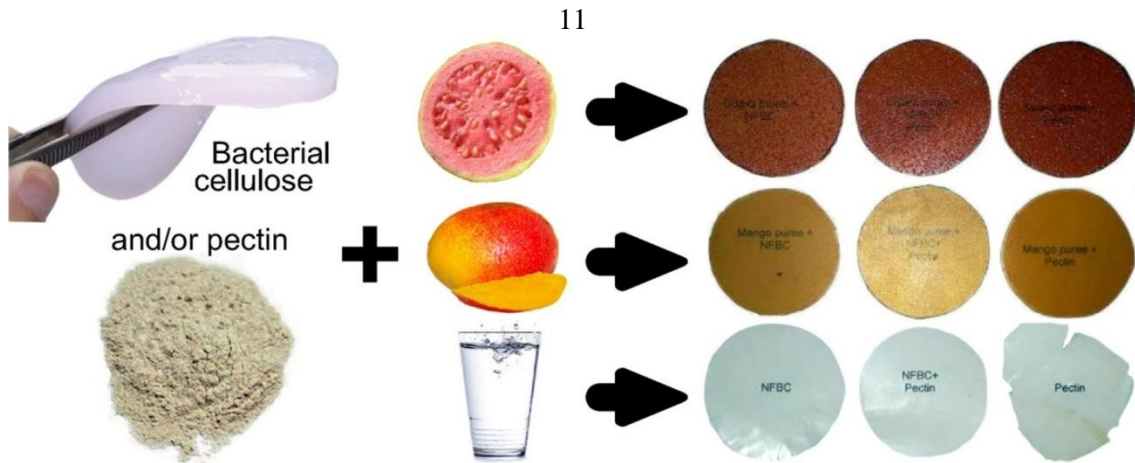


Figure 6: Nano fibrillated bacterial cellulose (NFBC) and pectin edible films with the addition of fruit purees (mango or guava purees) (Vianna et al., 2018).

Porta et al. (2016), produced edible films from hydrocolloids solutions by blending of pectin and proteins extract from *Vicia ervilia* seeds at complexation pH in the absence and presence of microbial transglutaminase (TGase). These finding an improvement of mechanical and barrier properties in the films obtained with protein/pectin blend incubated in the presence of TGase.

Researches have been conducted to assess properties of different edible film such as shelf life, antimicrobial, thickness, tensile strength (TS), elongation at break (EB) and Young's modulus (YM) for the edible film that applied on fruit and vegetable, and the results was compared in Table 1.

2.2 Polysaccharide based film

2.2.1 Pectin based film

Pectin is a structural carbohydrate that is a major component of plant cell walls and one of its attractive characteristics is its ability to form good films.(Banerjee et al., 2016). It composed of α 1,4-linked galacturonic acid, provides strength to plant tissues and helps in adherence. Diverse application of pectin as a gelling and thickening agent in food, as soluble dietary fiber, as a drug delivery carrier, as a film-forming polymer and recently as a prebiotic oligosaccharide has generated significant interest in efficient and economical methods for its production. Nevertheless, the pectin films have limited application utilization in food packaging because of have a poor water vapor barrier and a moderate oxygen barrier.

Many researches have been studied in order to solve this problem, such as incorporating nano-chitosan on pectin-based film. Films formed between pectin chains and nano-chitosan are stabilized by hydrogen links and electrostatic interactions. Nano-chitosan manifest higher antimicrobial activities and barrier properties than chitosan. Potential powerful and safe natural antifungal agent of nano-chitosan can improve the barrier properties and functionality of pectin film coatings (Ngo et al., 2020).

Han et al., (2020) prepared biopolymer pectin-based film extracted from mandarin peel (*Citrus unshiu*) and they integrated it with variant concentration of sage leaf extract (*Salvia officinalis*) (0.6%, 1%, 1.4%) to enhance the antioxidant activity. They have been found that the film that contain sage leaf has higher amount of phenolic compound comparing with the control film (pectin without sage leaf extract). As well as the lowest concentration of sage leaf extract (0.6%) resulted in most desirable physical properties; highest tensile strength and elongation at break. On the other side the addition of sage leaf decreased the thermal stability and water resistance of mandarin peels film. They conclude that mandarin peel contain sage extract can be potentially used for food-based application.

Recently, they have been studied the effect of incorporation different concentration of clove bud essential oil (0.5%, 1.0%, 1.5%) with citrus pectin film the physical, thermal, antioxidant and antimicrobial properties in order to modify functional properties of pectin film.

The inclusion of oil significantly increased the water barrier of film, increasing the oil concentration leads to more opaque films with relatively heterogenous microstructure. As well as this composite of films were more resistance to break and more extensible comparing with control film. Beside the heat stability

was higher for the film containing clove bud oil with slightly higher degradation temperature. The antimicrobial test revealed higher activity comparing to the control film (Nisar et al., 2018).

In 2019 Rai et al., evaluated the structural integrity and functional properties of incorporation corn flour, orange peel powder, beet root powder and rice flour on commercial pectin-based film. They demonstrating that corn flour with pectin film with ratio (1:1) were more stable compared to the rest of film and better functional and physical properties. Which could be an alternative for petroleum-based packaging.

Emulsion of pectin was used to coat Lime fruits, after that the surface was dried and cooled (Figure 7). Quality attributes were evaluated along storage periods at selected temperature range (10–25°C). It had been approved that pectin-based coating on Lime fruit would provide longer shelf life, accordingly refrigerating cost and ease of handling will be decreased on the other hand product safety and customer satisfaction will be guaranteed. Edible films will positively affect customer satisfaction and environmental goals (Maftoonazad and Ramaswamy, 2019).

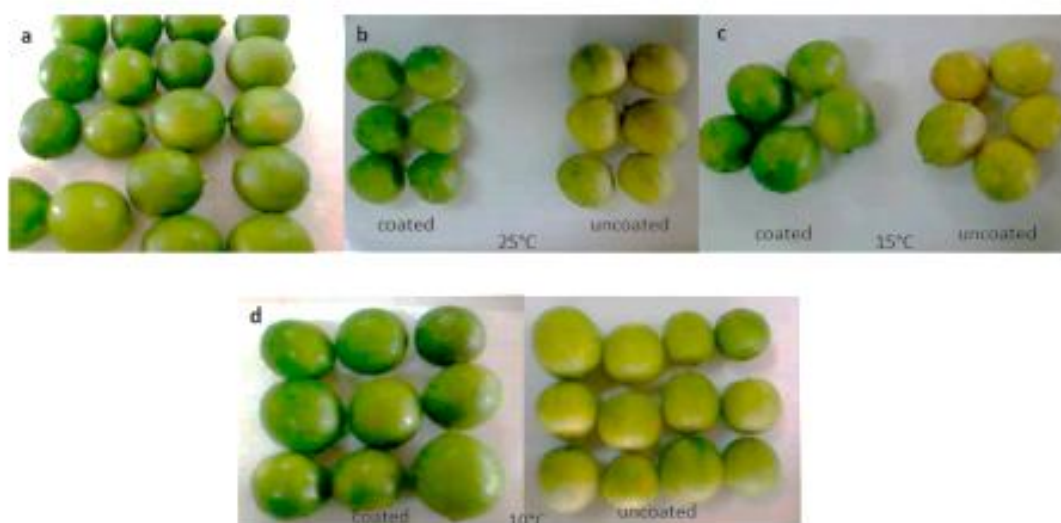


Figure 7: Photographs of lime samples:(a) day 1; (B) day 8; (C) day 25;(d) day 32

2.2.2 Chitosan film

Chitosan has been recommended as an edible film in food packaging due to its biocompatibility, biodegradability and non-toxicity properties. Chitosan (CH) is a linear polysaccharide of hetero-polymer with 2-amino-D-glucose and 2-acetamido-D-glucose monomers linked through β 1,4-glycosidic linkages. Higher degree of deacetylation ($>75\%$) in chitosan and an important molecular weight have shown the strongest antibacterial effects in aqueous solutions regardless of the type of acid used for solubilization. Because of its biocompatibility, antimicrobial activities, low oxygen permeability, and good mechanical properties, Chitosan has been combined with other hydrocolloids such as starch, gelatin, and alginate to produce blended or multi-layered films. Moreover, several natural ingredients, added to

edible films, that present antioxidant or antimicrobial properties such as nisin or lysozyme in order to modify antimicrobial properties. (Hafsa et al., 2016).

2.3 Protein based film

Soy, sesame, and bitter vetch and black cumin are examples on different seeds are widely used to produce vegetable oils or animal feed. Those have been shown to be potential sources of biodegradable/edible films. The leftover cakes after oil extraction, may contain up to 50% recyclable proteins that may produce bio-materials. Moreover, the defatted seed wastes generally contain a high level of polyphenols and, thus, thus powerful antioxidant and antimicrobial activity in edible wrapping or food coating application which increase the total polyphenols that give rise to higher nutritional value as well as keep food from rancidity, oxidation and spoilage by microorganism (Kadam et al., 2018; Toma et al., 2015).

Sabbah et al., recently improved innovative and biodegradable edible black film from *Nigella sativa* defatted seed cakes were obtained which prepared from a protein concentrate (PC) derived from *NsDSC*, in the presence of glycerol (GLY) as plasticizer, and their characterization was carried out. They also investigated the effect of addition transglutaminase (TGase), a food-grade protein cross-linking with obtained biodegradable/edible material. The

results showed that enzyme treated film with 20% of GLY at pH 8 have better mechanical and barrier properties as well as antimicrobial activity, these results give rise to use seed oil cakes in renewable bioplastic food packaging and not only on animal feed or fertilizer (Sabbah et al., 2020).

2.3.1 Gelatin based film

Fish gelatin one of promising bio-based and biodegradable bioplastic and alternative to mammalian-based gelatins that is suitable for vegetarian consumers (Hosseini and Gómez-Guillén, 2018). Fish gelatin characterized by being a colorless, transparent, tasteless, water soluble and potential oxygen and CO₂ barrier (Muhammad et al., 2016). Furthermore, it has been shown the potential effect to excellent host of bioactive compound, such as carvacrol, boldine, tea polyphenols, and olive phenols (Bermúdez-Oria et al., 2017; Dou et al., 2018; López et al., 2017; Neira et al., 2019).

2.3.2 Whey protein-based film

Whey protein a cheese industry by product have been successfully used to prepare edible nutritional edible film (Sukyai et al., 2018). Whey protein considered as a high biological value of protein due to the presence of essential amino acids and bioactive peptide. Whey protein-based film successfully prepared with addition of a nano-biocomposites material based on the use of poly-

3-hydroxybutyrate-co-hydroxyhexanoate nanoparticles (PHBHHx-NPs). The mechanical properties, O_2 , the permeability whey protein-based film enhanced due to the addition of PHBHHx-NPs, owing more extensible materials preserving their mechanical resistance (Corrado et al., 2020).

In 2019 Whey protein-based film (WPI) was integrated with *Lactobacillus casei* probiotic culture and applied as a dipping film for Thompson grapes and cherry tomatoes (Figure 8). They concluded by the use of WPI originated depends on fruit as the positive characteristics were suitable and could increase the shelf life of grapes. Also, the addition of *L. casei* enhanced the mechanical strength of the film. As well as the results proved that the *L. casei* probiotic culture could be incorporated in WPI films as it was able to survive in the WPI films for 14 days at 25 °c which could be an alternative to prolong the ripening process of fruit and provide health benefits to the consumer (Dianina et al., 2019).

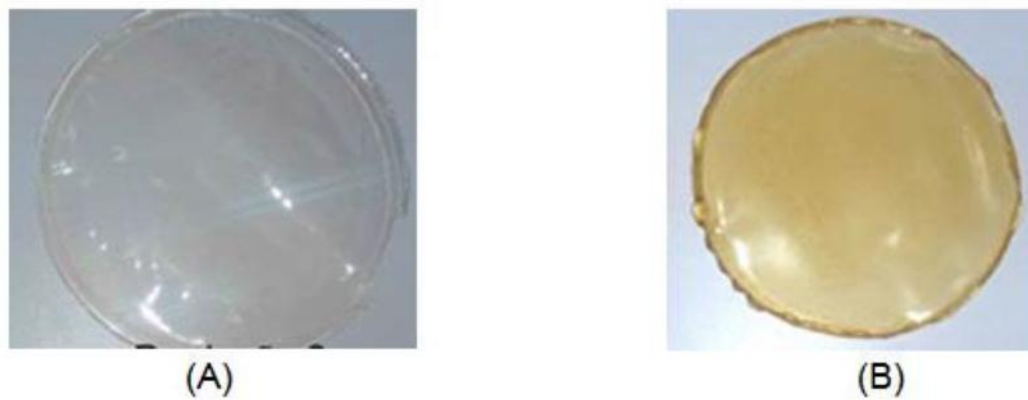
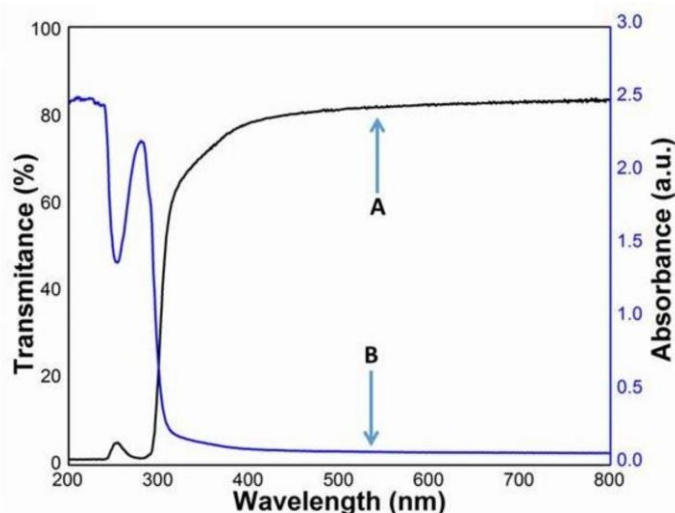


Figure 8: Visual appearance of the films. (A) Control film, (B) with *L. casei* (Dianina et al., 2019).

Abdalrazeq et al., 2019 explored the possibility of preparing whey protein-based film without heat treatment and compare it with the heat treated, under the alkaline conditions (pH 12) with presence of 40% or 50% of GLY (Figure 9). The results led to produce successful film resistant and more flexible than at pH 7. The water uptake of films prepared at pH 12 did not significantly change. While the moisture content decreased with reducing GLY concentration (Abdalrazeq, Manar et al., 2019).



(a)



(b)

Figure 9: Film obtained at pH 12 with unheated whey proteins (WPs) in the presence of 50% glycerol (GLY) (a) and its whole transmittance (A) and absorbance (B) spectra (b) (Abdalrazeq et al., 2019).

2.4 Edible film and coating applied on different fruits and vegetables

Fruits and vegetables are reservoir of vitamins, essential minerals, antioxidants, bio-flavonoids, dietary fibers and flavor compounds. Regarding to the combined report of World Health Organization and Food and Agriculture Organization; minimum recommended daily intake of fruits and vegetable is 400 g, this may reduce the risk of chronic disease along with the mitigation of micronutrient deficiencies (Bhatta et al., 2020). Adding natural color beside creating convenience and handballed food applications could be an effective and attractive way in order to encourage people to consume the recommended intake of fruit and vegetables. Among the numerous and attractive natural sources, the most interesting are the betalains, a water-soluble pigment, consist of

betacyanin's (red-violet) and betaxanthins (yellow), found in plants of the Caryophyllales order (Clement and Mabry, 1996; Herbach et al., 2006).

Table 1: Mechanical and antimicrobial properties comparison of edible film.

Used Edible Film	Effect							Reference
	Mechanical Properties				Barrier properties	Water solubility	Antimicrobial Activity	
	Thickness (μm)	TS (mPa)	EB (%)	YM (mPa)				
P-NCH+ 50% GLY	≈47.7	≈ 9	≈ 10.6	ND	Increase	ND	Inhibition of <i>S. cerevisiae</i> , <i>A. Niger</i> , <i>C. gloeosporioides</i> and <i>E. coli</i> .	Ngo et al., 2020
NsDSC 20% GLY	≈ 140	≈ 2.5	≈ 180	≈ 20	Increase	ND	Inhibition <i>S. aureus</i> Inhibition	Sabbah et al., 2020
GF+10% PSP	≈132	≈ 161	≈ 191	ND	Increase	Decrease	Inhibition <i>S. aureus</i> + <i>S. enterica</i>	Valdés et al., 2020
NFBC+ Pectin + Mango purees 0.57 g Sorbitol	≈100	ND	ND	ND	Increase	ND	ND	Viana et al., 2018.
BVPC+ TGase	≈114.0	≈ 4.3	≈ 64.1	≈ 60.3	Increase	ND	ND	Porta et al., 2016

ND = Not Determined; P-NCH= Pectin: nano chitosan; NsDSC= Nigella sativa defatted seed cakes; GF+PSP = Gelatin fish + pomegranate seed juice by-product; NFBC = Nano fibrillated bacterial cellulose; BVPC+ TGase= Pectin and Vicia ervilia seed proteins + microbial transglutaminase.

Increasing the demands for fresh, nutraceutical-rich and good-quality foods can be accomplished by using edible film and freeze-drying technique (FD). FD considered as a promising technology for the production of high-quality, nutritious food powders and solids to maintain nutritious fresh fruit. The absence of water, oxygen-free environment (if operated under vacuum), and low operating temperatures is thus the best choice to dehydrate fruits and vegetables in order to keep an optimized bio compound content in the final products (Rudy et al., 2015). Edible coating of whey protein/pectin film and transglutaminase for fresh-cut apple, potato and carrot was demonstrated a microbiological development inhibition. However, no changing in hardness and chewiness was observed (Marquez et al., 2017).

Further studies on pectin edible film with different concentrations of aloe vera gel got positive results in post-harvest storing of tomatoes. By using *Alternaria alternata*, mold growth was prevented in tomatoes moreover, by adding savory oil to pectin also the antioxidant activity increased without making any negative effect on sensory properties of tomatoes (Chrysargyris et al., 2016).

Additionally, edible coating treatment of 2% low methoxylated pectin, 1% carnauba wax, 1.5% glycerol and 0.05% ascorbic acid was applied to broccoli, carrot, cauliflower, zucchini, celery, carrot and chayote vegetables followed by heat treatment for

2 min at 60°C maintain the sensorial qualities of vegetables (Hernández et al., 2014). Dubey et al (2019), developed a nanocomposite edible films based on aloe vera gel, glycerol and nanoparticles solution and applied it as a coating of Mango (*Mangifera indica* L.) and stored for 9 days at room temperature, results showed that the glycerol and Zinc oxide nanoparticles solution concentration significantly affected all the film properties and the quality parameters of mango during storage and extended the shelf life of fresh mangoes when stored at room temperature. Mannozi et al (2017) developed edible coating material from pectin mixed with sodium alginate and applied it on blueberries. The use of pectin mixed with sodium alginate improved the firmness, induced more intense blue hue color and lower lightness and reduced the growth kinetics of yeasts and mesophilic aerobic bacteria compared to the uncoated samples of blueberry.

Essential oils and bioactive ingredients such as antioxidant can be integrated into the matrix, hence enhancing functional criteria of the formulation (de Moraes Crizel et al., 2016). Pectin (Porta et al., 2016), chitosan (Sabbah et al., 2019), polyglutamic acid (Karimi et al., 2020), alginate (Guerreiro et al., 2015), galactomannans (Cerqueira et al., 2011), and wax (Ochoa et al., 2011) are polymers that have been used in the formulation of edible coating or film. Barrier properties, the wettability and respiratory rate are very important parameters to take into account when

evaluating the potential of a biomaterial in the formulation of edible films.

Mango peels solution added or not antioxidant extract of mango seed kernel has been developed as an edible film for peach coating in order to increase the polyphenol content and reduce gas transfer, reduce CO₂ and ethylene which ensure greater shelf life of treated fruit (Figure 10). Mango by- product may thus be suitable for the production of low-cost biodegradable and active packaging (Torres-León et al., 2018)

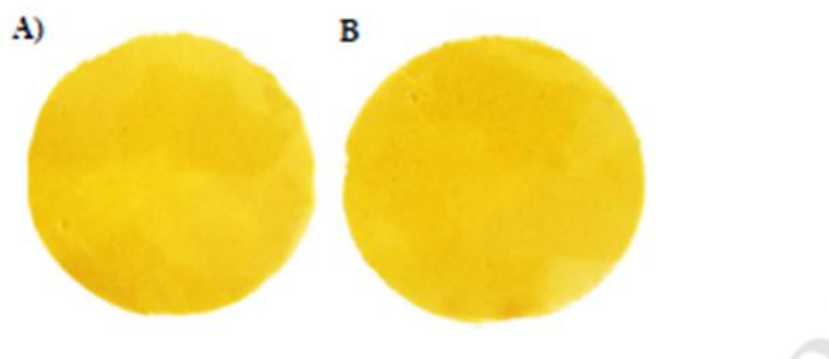


Figure 10: Edible films based on mango by-products: (A) Mango peel-based film and (B) Mango peel-based film with antioxidant Kernel (Torres-Leon et al.,2018)

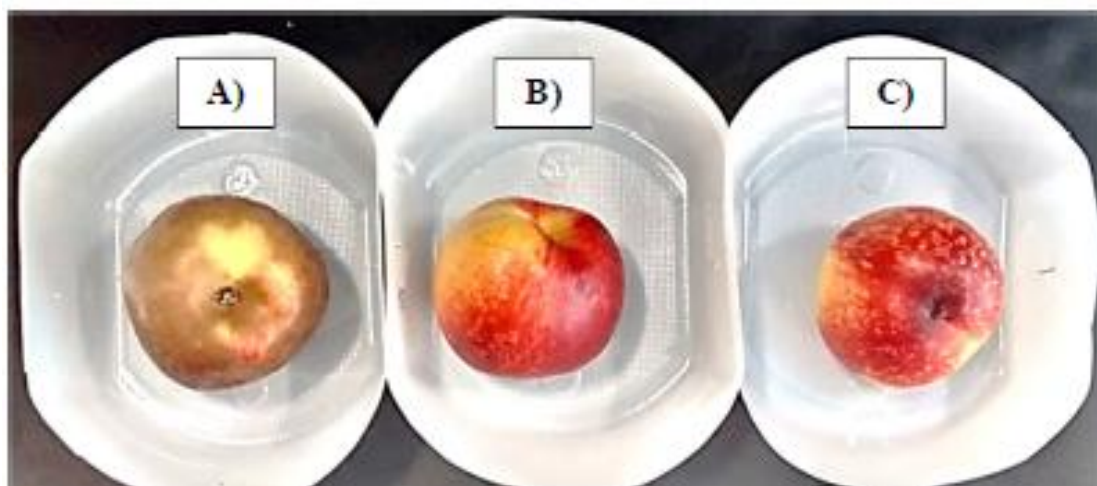


Figure 11: Visual aspect of peaches on the eighth day of storage (20 °C, 80% RH). (A) Uncoated peach (B) Coated with mango peel (C) Coated with mango peel integrated with antioxidant Kernel (Torres-Leon et al.,2018)

In 2019 a research has been conducted on the addition of turmeric and ginger extract to edible film of canna tuber and aloe vera in order to wrap a tomato to determine the ability of both extracts in increasing the shelf life of tomatoes by testing the texture and shrinkage weight of tomatoes fruit. They conclude that ginger and turmeric extract addition into edible film maintain the quality of tomato. Reduction of 50 % tomato weight that have been coated with edible film and without ginger or turmeric extract may increase to 48 days (Figure 12), while for film integrated with ginger extract the 50 % of tomato weight shrinkage occurred in 65 days whereas the turmeric extract 50% of shrinkage occurred within 60 days (Figure 13). Meanwhile for the texture changes; 63% texture reduction within 7 to 7.5 days for the edible film without ginger or turmeric extract, and in 12 days texture reduction for the

film that containing ginger extract while turmeric extract to 186 days (Figure 14). The addition of ginger and turmeric extract can function as an antioxidant bioactive compound that reduce the aspiration rate and improve the mechanical properties of the edible film (M Alsaïad et al.,2019).

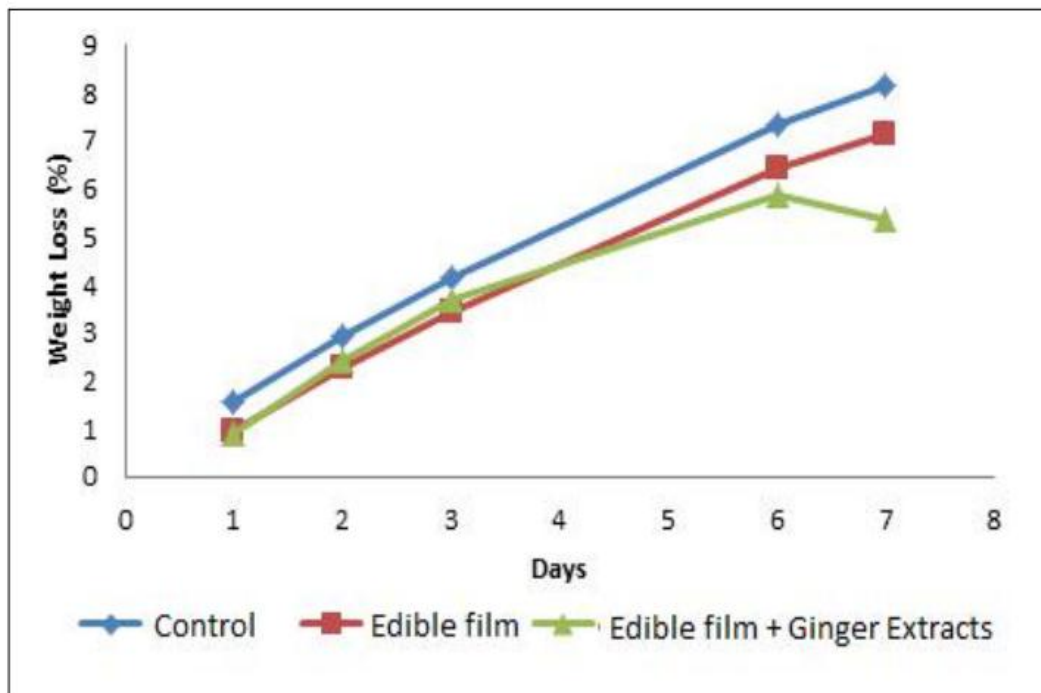


Figure 12: Correlations of addition of ginger extracts in edible film to tomatoes weight loss

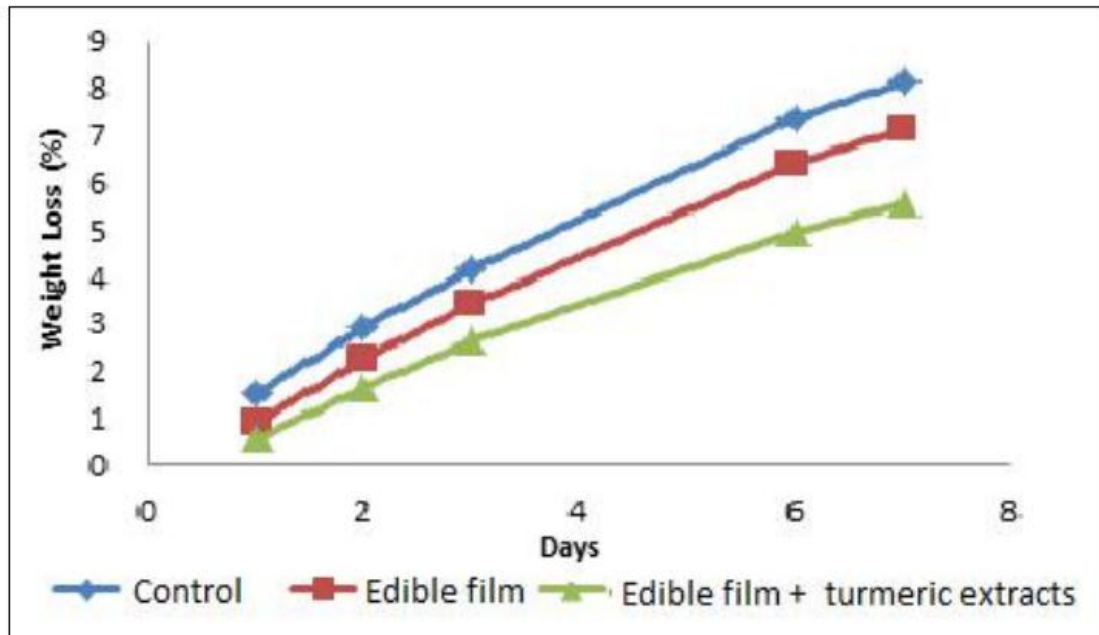


Figure 13: Correlations of addition of turmeric extracts in edible film to tomatoes weight loss

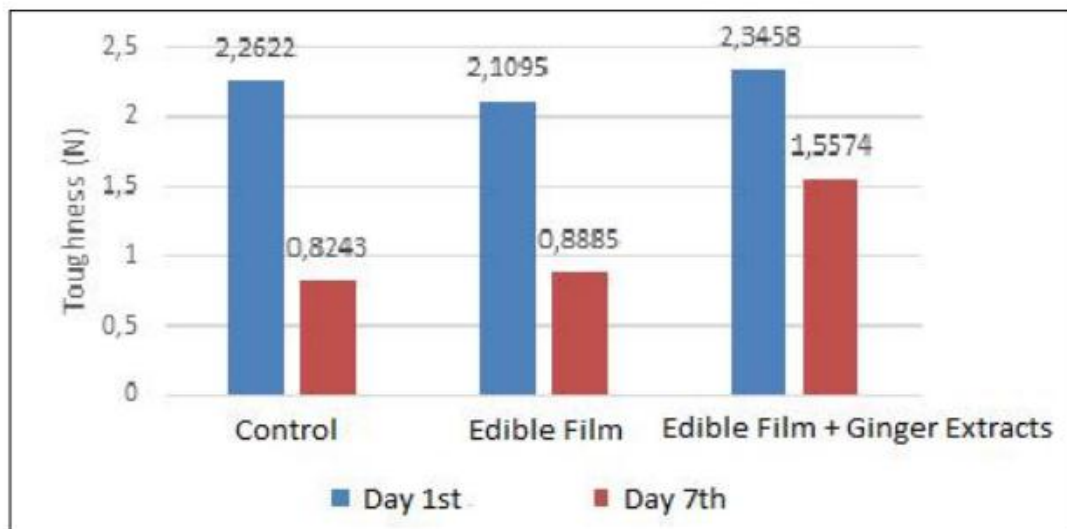


Figure 14: The Texture Difference between Tomatoes unwrapped, wrapped by edible film and wrapped by edible film with turmeric extracts.

This project considered as the initiation for food industry to provide consumer with characterized products. This includes easier utilization with specified serving size, in addition to defined nutrition facts. This allows the consumer to decide the appropriate product. Edible pectin film loaded with an encapsulated essential oil-chitosan nano particle; in addition to its property of environmentally friendly; will facilitate food (juice powder) consumption, since it is water soluble package.

Nguyen et al., 2019 have been carried out a research focused on crosslinking of whey protein with pectin using transglutaminase in different ration to preserve mango fresh cut. Different parameters have been measured such as weight loss, total phenolic, carotenoid's, ascorbic acid, sensory score. They demonstrating that using 75%:25% whey protein: pectin in film forming coating was suitable for the application (Minh et al., 2019).

Other application has been done on 2020 by Nurrochman, A et al., 2020 using banana peels to make edible film combined with cinnamon oil as the essential oil exhibit antimicrobial activity. A good functional, physical and mechanical properties have been shown (Nurrochman, A et al., 2020).

Varasteh et al., 2018 exploring the using of chitosan for pomegranate dipping. different concentration of aqueous chitosan solution has been used (0, 1, 2%) air dried and stored at 2 and 5°C with 90% relative humidity for up to 135 days. The highest weight loss value was 18.19% and 9.33 % was the lowest after 135 days of storage (Varasteh et al., 2018).

Chapter Three

Methodology

3.1 Materials

PEC (Aglupectin USP) purchased from Silva Extracts s.r.l. (Gorle, BG, Italy), glycerol (about 87%) was obtained from the Merck Chemical Company (Darmstadt, Germany), synthetic colors (Azorubine, Tetrazine, Sunset color) purchased from Sun pharm drug store company (Palestine), NESCAFE 3 in 1 instant coffee from supermarket.

3.2 Film preparation

Pectin film was prepared in presence and absence of 30% (w/v of pectin) of glycerol according to (Porta et al., 2016), by dissolving 0.6 g in 100 mL of distilled water and mixed until completely dissolved. For pectin film containing glycerol, 30% (w/v of pectin) of glycerol was added after pectin solubilization and mixed under magnetic stirring. Then, the pH of the solution was adjusted to pH 7.2 by using 0.1 N of NaOH. About 50 mL of each film forming solutions were cast on plastic petri dishes (diameter 8 cm) and dried at $25 \pm 5^{\circ}\text{C}$ for 48hrs. Finally, dried films were peeled-off and conditioned at 45% RH prior to characterization. The following films was prepared in presence of 30% (w/v of pectin) of glycerol.

3.3 Film properties

Pectin films were characterized for their thickness by detecting it in six different points of the film by using an Electronic digital micrometer, (DC-516, sensitivity 0.001 mm). Tensile strength (TS), elongation at break (EB) and Young's modulus (YM), were determined on six strips (5 cm x 1 cm) of each different film by using an Instron universal testing instrument model no. 5543A (Instron Engineering Corp., Norwood, MA, USA) (ASTM D882-97, 1997) with 5 cm gauge length, 1 kN load cell and 5 mm/min speed.

The opacity of the films was tested six times for each film, as described by (Giosafatto et al., 2019) and calculated as follows:

$$\text{Opacity (mm}^{-1}\text{)} = A_{600}/X$$

Where A_{600} was the absorbance at 600 nm and X was the film thickness (mm).

Also film permeabilities to O_2 (ASTM, 2010), CO_2 (Testing and Materials, 2005) and water vapour (WV) (F-13, 2013) were carried out in triplicate at 25 °C and 50% RH by using a TotalPerm apparatus (Extra Solution s.r.l., Pisa, Italy).

To test PEC film ability to be heat-sealed, a routine device to thermo-weld polyethylene film strips was used (Citynet Medical (Milano, Italy) (C.A. 50 Hz. 200–250 V) by subjecting double

layers of PEC to a pressure at high temperatures for 10 seconds. After that, two layers of PEC film (10 cm x 5 cm) were sealed on three sides to obtain a bag. To evaluate the quality of thermo-sealed strips the sealed side were analysed by an optical microscope at 50× magnification.

3.4 Pectin film containing synthetic food color

Pectin film was prepared according to (Porta et al., 2016) as described previously, after complete dissolution of pectin, different concentration of synthetic colors (Azorubine, Tetrazine, Sunset color) (0.1, 0.3, 0.6% (w/v)) were added and mixed under magnetic stirring until completely dissolved, the pigmented film were prepared both in the presence and absences of glycerol 30% (w/w of pectin). The pH of the solution was adjusted to pH 7.2 by using 0.1 N of NaOH.

About 50 mL of each film forming solutions were cast on plastic petri dishes (diameter 8 cm) and dried at $25 \pm 5^{\circ}\text{C}$ for 48hrs. Finally, pigmented dried films were peeled-off and conditioned at 45% RH prior to characterization.

3.5 Fruit juice powder packed in edible film

Fruit juice powder purchased from the supermarket was packed in three different films; About 1.8 g of fruit powder filled in 11×5 cm of PEC films that sealed in bags shape as shown in Figure 5 by using impulse sealer PFS-200.

3.6 Product solubility, pH and total soluble solids

Fruit juice powder to check the solubility of edible film. For this purpose, about 1.8 g fruit juice powder was introduced in the bag and then heat-sealed on the open side. Then the filled pectin bags (were dissolved in distilled water at different temperature (4, 20°C) and the physical observation of time required until it completely solubilized was recorded.

Moreover, pH using pH meter were measured as well as total soluble solids (brix value) using KRUSs, Germany refractometer for the fruit juice powder wrapped with different type of pectin film.

3.7 Statistical analysis

JMP software 10.0 (SAS Institute, Cary, NC, USA) was used for all statistical analyses. One-way ANOVA and the least significant difference (LSD) test for mean comparisons were used. Differences at $p < 0.05$ were considered significant and are indicated with different letters.

Chapter Four

Results and discussion

4.1 Pectin film characteristic

Since glycerol is one of the most popular plasticizers used in preparation of edible films and coatings which improves the mechanical properties of the film due to hydrophilic nature of the biopolymer chains, stability and compatibility (Chillo et al., 2008), preliminary experiments have been carried out to prepare pectin film with and without glycerol to investigate the mechanical and permeability properties. Table 2 shows the mechanical properties of pectin films in presence and absence of 30% (w/w of pectin) of GLY. It shows that the presence of GLY significantly enhanced film elongation at break (EB) as well as their thickness. Conversely, very marked reductions of tensile strength (TS) and Young's module (YM) values of the PEC films were detected only when GLY is present.

Table 2. Effect of GLY on seal strength, film thickness, opacity, and mechanical properties of PEC films.

PEC films	Seal strength (N/m)	Thickness (μm)	Opacity (mm^{-1})	TS (MPa)	EB (%)	YM (MPa)
- GLY	18.5 ± 1.3	37.5 ± 2.4	1.91 ± 0.16	35.4 ± 6.4	0.8 ± 0.2	5350.5 ± 565.1
+ GLY	52.4 ± 2.3^a	56.8 ± 3.1^a	2.05 ± 0.30	28.1 ± 3.8^a	8.8 ± 3.1^a	1060 ± 45.70^a

^a Values significantly different at $p < 0.05$ from those obtained without GLY.

Also, the permeability tests of the PEC films blended with GLY indicated interesting features. In fact, Table 3 reports that the PEC films containing GLY as plasticizer showed, with respect to the one without GLY, a permeability toward O_2 markedly lower while a small but significant reduction in WV and CO_2 permeability was observed. Furthermore, we investigated the ability of the PEC films, prepared in the absence or presence of GLY to be heat-sealed. The results indicate that both films can be sealed.

Table 3. Gas and WV permeability of PEC films prepared in the presence or in the absence of 30% GLY.

PEC films	Permeability (cm ³ mm m ⁻² day ⁻¹ kPa ⁻¹)		
	CO ₂	O ₂	WP
- GLY	0.132 ± 0.01	0.148 ± 0.007	0.160 ± 0.047
+ GLY	0.110 ± 0.02 ^a	0.040 ± 0.014 ^a	0.109 ± 0.011

^a Values significantly different at $p < 0.05$ from those obtained without GLY.

Fig 15 show the homogeneous sealing of PEC film containing GLY that is similar to the one observed in PEC film obtained without GLY (data not showed). However, by test the seal strength of the two films we observe that in the film containing GLY the strength is three time higher that in the film without GLY (Table 1).

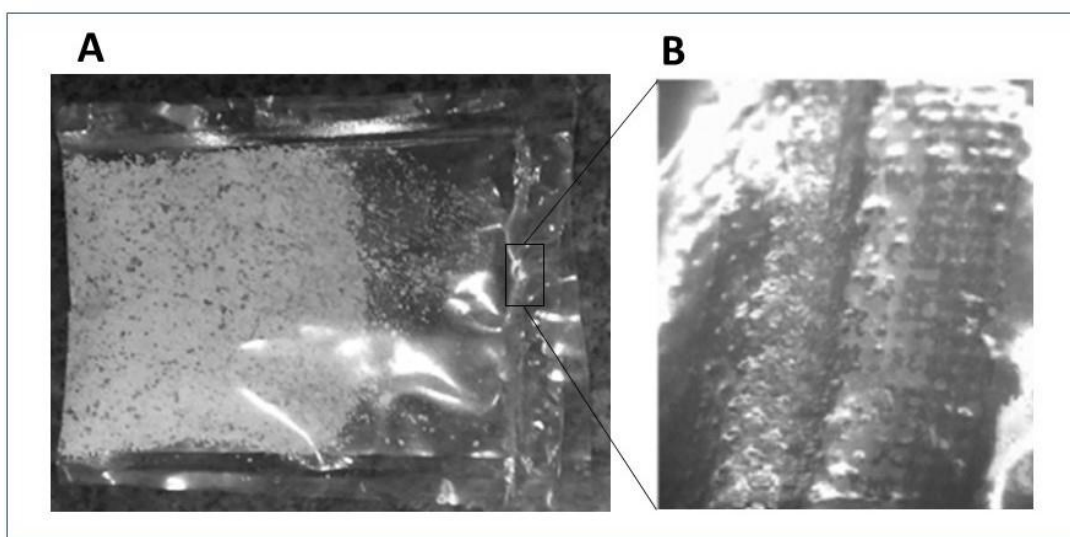


Figure 15: Fruit juice powder wrapping by a heat-sealed PEC film containing 30% (w/w of pectin) GLY (panel A), and the magnification (50×) under optical microscope of the film heat-welding (panel B).

All the results clearly demonstrated that GLY represent a useful strategy to give rise edible films specifically tailored for desired applications. Therefore, we decided to continue our studies by using of 0.6% PEC films obtained in the presence of 30% (w/w of pectin) GLY.

Many studies indicate the superior effect of glycerol as plasticizer of better activity than other substances used (Sothornvit and Krochta, 2001; Vanin et al., 2005). High boiling point, lack of odor, water solubility and miscibility with those components are the main advantages of this plasticizer (Jangchud and Chinnan, 1999).

In study reported by Myllärinen et al, (2002) the results showed that the low glycerol contents increased the brittleness of the films based on changes in elongation at break. Moreover, it is well known that plasticizer molecules decrease intermolecular interactions and increasing intermolecular distance by water molecules binding and shield active centers along polymer chains (Costanza et al., 2019). Further study demonstrate that Color, mechanical properties and water vapor permeability were affected by adding different concentration of glycerol and pectin (Galus et al., 2013).

They also investigated and characterized the effect of glycerol as a plasticizing agent in pectin-based films used for temperature sensing; glycerol improves elongation at the break of

pectin films up to a critical concentration, but it suppresses their responsivity to temperature. This behavior was explained by the increasing of water content in the films due to the presence of glycerol. The combination of the different studies presented elucidates that a concentration around 2.5% v/v of glycerol is the most desirable ratio to improve the mechanical properties of the material without critical losses of the temperature sensing capability (Galus et al., 2013).

4.2 Effect of synthetic food colors on pectin film

Different amount of synthetic color was mixed with pectin film solution containing glycerol in order to make colored films. Figure 16, 17 and 18 showed the results obtained with azorubine, tartrazine and sunset respectively. Handleable and homogeneous films with an acceptable color intensity can be obtained at 0.3 and 0.6 % w/v of all color. Low concentration of colored substance negatively affects the films casting, in fact no film was obtained with 0.1% of azorubine while brittle and cracked films was obtained by using tartrazine and sunset, respectively.

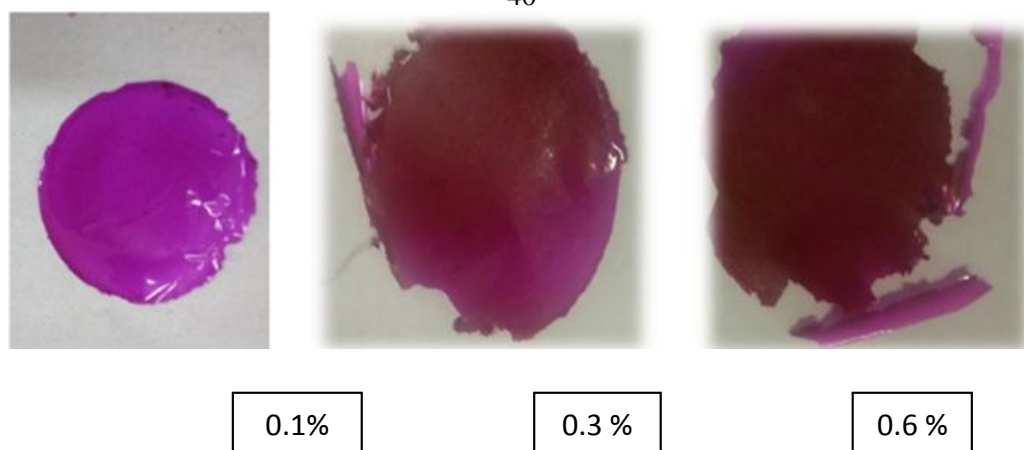


Figure 16: PEC film with different concentration of azorubine (E122) synthetic food colors (0.1,0.3,0.6%) prepared with 30% (w/w of pectin) GLY.

However, in the films containing 0.1 % of color, no cracks were observed and a more flexible and handleable films were obtained comparing with film obtained with 0.6 % of color. This result support the idea of using colored pectin films in the making of single dose soluble fruit juice powder.

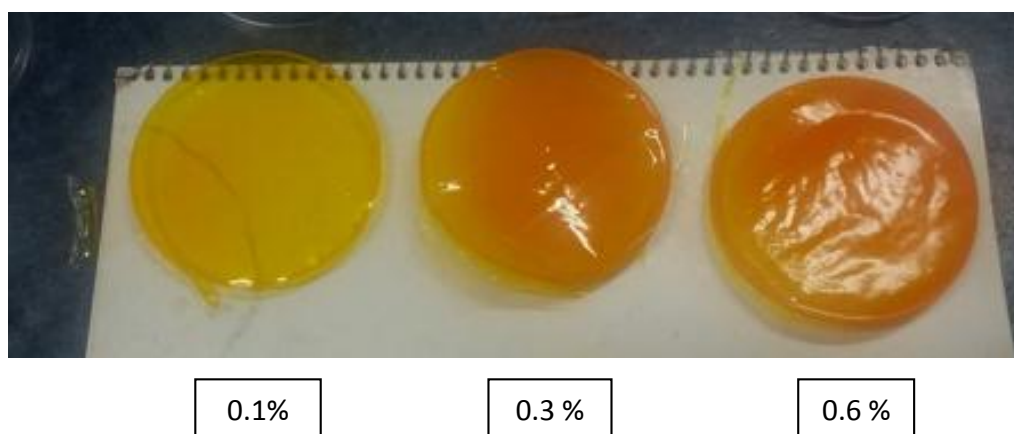


Figure 17: PEC films with different concentration of tartrazine synthetic food colors with 30% (w/w of pectin) GLY.

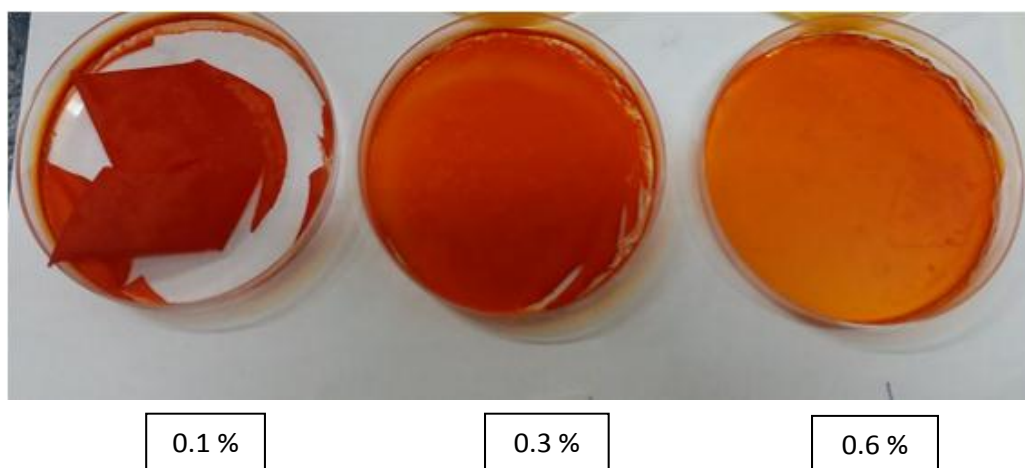


Figure 18: PEC films with different concentrations of sunset yellow food colors obtained with 30% (w/w of pectin) of GLY.

4.3 Pectin film and powder solubility

In order to test the solubility of pectin packaging; a fruit juice powder was used and packed in a heat-sealed PEC film. The solubility was evaluated by immersing the pectin bag filled with fruit juice powder into distilled water under continuously stirring until the total solubilization (Figure 19). Different temperature values of distilled water were used 4°C and 20 °C to evaluate the effect of temperature. The results in Table 4 showed that the highest solubility was at 25° C with the completely solubilized was obtained within 30 seconds for the PEC film with glycerol. The lower temperature can be useful to preserve the thermolabile compound of fruit juice.



Figure 19: Pectin film containing 30% (w/w of pectin) GLY filled with fruit juice powder and dissolved with water, the product was totally solubilized at 4 and 25 °C.

Table 4. PEC film in presence of GLY filled with processed fruit juice powder, solubilized at different temperature values in distilled water.

Temperature (°C)	Observation
4	Completely solubilized after 57 sec
25	Completely solubilized after 30 sec

Recently, they observed physical and mechanical properties improvements, good homogeneity and optical properties, high solubility with complete solubilization at 80 °C (Puscaselu et al., 2019). Moreover, the pectin films (HDM) incorporated in different concentrations of spent coffee grounds (5–20% w/w HDM) and obtained by continuous casting resulted in important pectin-based film properties changes, allowing an increase in color and thermal

stability and significantly improved the water vapor permeability rate improving or at least preserving the physicochemical properties (Mendes et al., 2019) .

Chapter Five

Conclusion

Due to synthetic polymers environmentally negative impact, green polymers with natural ingredients gained its importance. Our results confirm that pectin film is good for packaging single doses of fruit juice powder and the addition of food color didn't affect the film structure. Successful production of edible pectin film water-soluble package and ready to use.

The preliminary studies confirm that edible film prospects a great field in green production that help achieve global vision towards sustainability. Moreover; maintains high quality and sustainable production chains, by conserving natural resources and waste reduction, through using different numerous technologies, such as; active/intelligent packaging, nano/micro capsulation and bacteriocin incorporation to ensure food security. Furthermore, supporting and enhancing healthy gut microbiota in functional food by using symbiotic, bioactive compounds (i.e., polyphenols) or essential oil (i.e., Melissa (*M. officinalis* L) to provide healthy food for consumer. For this purpose, a future applicability of edible films on the improvement of the quality of healthier fruit juice powder could be investigated.

Further analysis could be done on the mechanical and barrier properties of all film types as well as the determination of total phenolic compounds performing by the Folin Ciocateau method (Singleton and Rossi, 1965) and antioxidant activity, by adopting methodology proposed by (Kondo et al., 2002). Moreover, Sensory evaluation in order to estimate the shelf life of fruit powder wrapped with edible film and mixed with distilled water by 12-member using a 5-point descriptive scale (Mohamed et al., 2011).

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تأثير الأغلفة الصالحة للأكل على جودة مسحوق الفاكهة الصحي

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الملخص

التطور التكنولوجي، البيئة المستدامة، صحة الانسان وسلامته والابتكارات وارتباطها مع خطط تقليل تكاليف الانتاج؛ جميعها تشكل تحديات لتطوير قطاع الصناعات الغذائية والتغذية. حفظ الغذاء المصنع باستخدام غلاف قابل للأكل، ذو ذائبية عالية بالماء ومن مكونات عضوية ومعدة للاستهلاك المباشر، يشكل جزء من الحلول المرتبطة بالصناعات الغذائية والتي تلامس التحديات التي يواجهها المصنع على مستوى الصناعة نفسها من حيث تكاليف الانتاج والاثـر على الاستخدام النهائي للمنتج من قبل المستهلك.

هدف الرسالة تغليف العصير المجفف باستخدام غلاف طبيعي وعضوي قابل للأكل مما يتيح أفقاً لتحضير العصير الصحي بواسطة تدعيم الغلاف القابل للأكل بمواد تحتوي على مركبات حيوية مثل الزيوت العطرية التي تتميز بخواص عديدة أهمها؛ احتوائها على مضادات الأكسدة التي تساهم بإطالة فترة صلاحية المنتج وتقليل استخدام المواد الحافظة والذي بدوره ينعكس ايجاباً على صحة المستهلك. حيث توصلنا الى ان تغليف العصير المجفف بواسطة غلاف البكتين و المغلق حرارياً قابل للذوبان خلال 30 ثانية بماء على درجة حرارة 25 درجة مئوية بينما يحتاج الى 57 ثانية بماء درجة حرارته 4 درجة مئوية.