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Analysis of Chia (Salvia hispanica) and Nopal Edible Film Formulation under Alkaline Conditions and its Correlation with Water Vapor Permeability

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ABSTRACT: The present study assessed the impact of nopal and chia mucilage concentrations, adjusted to a pH value of 11.0, on the water vapor permeability (WVP) of edible films. These films were additionally formulated with sodium alginate and glycerol. Films incorporating both mucilage types demonstrated WVP values of 1.2 × 10-8 g/Pa·s·m. It was observed that WVP increased linearly with higher mucilage content, with nopal mucilage films exhibiting superior barrier properties. This research provides pertinent insights for the development of edible films utilizing plant-based mucilage.

KEYWORDS: Water Vapor Permeability, mucilage, alkaline, edible films.

I. INTRODUCTION

Edible films are thin coatings applicable to various food items, designed to prolong their shelf life by preserving texture and appearance. They are also utilized to incorporate bioactive compounds such as antioxidants. Typically formulated with polysaccharides, proteins, and/or lipids, these films may encompass a diverse range of additives to achieve specific desired properties.

[1]Today, edible films are gaining popularity in the food industry due to their ability to extend shelf life and enhance food quality. These thin, edible coatings serve as a protective barrier against external factors that hasten deterioration."

[2] One of the most widespread applications of these films involves their use on whole or cut fruits and vegetables. Applying edible films to fruits such as mangoes, avocados, or berries reduces moisture loss and respiratory rate, thereby delaying their ripening and decomposition. They are also employed to prevent dehydration and enzymatic browning in cut fruits or minimally processed vegetables.

[3]Another significant application of edible films is in meat products such as hams, sausages, and cured meats. Films endowed with barrier properties control moisture loss, lipid oxidation, and microbial growth, thereby extending shelf life and enhancing appearance by reducing surface desiccation processes.

[4] In the case of cheeses, edible films diminish moisture loss, shield against microbial contamination, and regulate gas exchange. This preserves moisture content, texture, flavor, and nutritional value over extended periods.

[5] Edible films also find application in preventing retrogradation in baked goods, inhibiting browning reactions in pre-fried potatoes, extending the shelf life of fish and seafood, and safeguarding dry ingredients against ambient humidity. Their versatile use and effectiveness position them as a promising technology for enhancing food quality and shelf life.

[6] Plant mucilages have garnered significant interest as ingredients to produce edible films in recent times. These mucilages are complex polysaccharides naturally found in plants such as Aloe vera, flaxseed, or prickly pear.

[7] Some commonly studied mucilages include tara gum, guar gum, xanthan gum, and arabic gum, among others. Mucilages derived from plant sources exhibit excellent properties for film generation owing to their capability to form highly viscous solutions. Moreover, they can interact with other biomolecules through hydrogen bonding and

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Van der Waals forces, rendering them ideal for producing homogeneous films.

[8] Recent studies have investigated the incorporation of mucilages into edible films, demonstrating the potential for optimizing certain mechanical properties such as flexibility, elongation, and strength. Mucilages can also delay the diffusion of moisture, oxygen, and lipids when employed as coatings.

[9] Chia (Salvia hispanica L.) has recently emerged as a new source of mucilage with significant potential for manufacturing edible films. Chia contains approximately 5-10% of mucilage, primarily located in the seed's seed coat. This mucilage is primarily composed of arabinoxylan and galactoxyloglucan polymers. Chia mucilage exhibits excellent emulsifying, foaming, and gelling properties, making it ideal for cohesive matrix formation. Films produced with chia mucilage display good mechanical properties, water vapor barrier, and transparency. Furthermore, the mucilage imparts natural antioxidant and antimicrobial activity to the films. Chia represents a renewable and sustainable source for obtaining film-forming agents with additional functionalities. Therefore, its mucilage is considered a biopolymer with potential for developing edible biofilms with applications in food preservation and quality enhancement.

[10] The nopal cactus (Opuntia ficus-indica) is a Mexican plant that holds enormous potential for the food and pharmaceutical industries. This plant contains mucilage in its pulp and pads, which has demonstrated excellent properties for forming edible bioplastic films. Nopal mucilage is primarily composed of polysaccharides such as arabinose, xylose, galactose, rhamnose, and galacturonic acid. This mucilage exhibits high water retention capacity and gel-forming abilities. Films produced with nopal mucilage demonstrate good mechanical, thermal, and water vapor barrier properties. Additionally, the mucilage provides antioxidant and antimicrobial activity, highly beneficial for food preservation.

[11] As the nopal is a plant that thrives readily in arid conditions, it stands as an abundant and sustainable resource for extracting film-forming agents. Nopal mucilage is being utilized as a promising ingredient in the development of edible bioplastic films, offering potential applications in fruits, vegetables, meats, and other foods due to its cost-effectiveness and abundance.

[12] In analyzing factors influencing film quality, it has been observed that modifying pH during the formulation of edible bioplastic films can significantly impact their final properties. Employing alkaline conditions offers several intriguing advantages: firstly, the solubility and extractability of numerous biopolymers with potential for film formation, such as proteins and polysaccharides, increase in alkaline environments. This facilitates their incorporation and the formation of homogeneous polymeric matrices.

[13] Additionally, complementary studies indicate that the partial depolymerization of biopolymers in an alkaline pH can reduce their viscosity, enhancing the malleability and film-forming capacity. Electrostatic interactions between biopolymers carrying opposite charges are favored in an alkaline pH, promoting the formation of polyelectrolyte complexes that yield stronger and more stable films.

[14] Further studies highlight that the solubility of bioactive compounds such as phenolic-origin antioxidants and antimicrobials also increases under alkaline conditions. Their effective integration can enhance the functionalities of the films.

[15]The water vapor permeability represents a critically important property in the quality of edible films, serving as a crucial factor considered by various research groups in their endeavors to optimize formulations for food-related applications of these films.

[16] An adequate control of water vapor permeability in films is essential for several reasons: firstly, it regulates the moisture transfer between food and the surrounding environment. Secondly, low permeability reduces water loss in fresh foods, preventing dehydration, while high permeability inhibits water condensation that facilitates microbial growth. This factor significantly impacts the texture and mouthfeel of coated fruits and vegetables, as well as the crispiness kinetics in dehydrated or fried products.

[17] Ultimately, water vapor permeability impacts the physicochemical stability of food components, particularly sensitive compounds like vitamins, aromas, pigments, or bioactive compounds. It significantly determines the film's effectiveness in delaying oxidation reactions, enzymatic browning, aroma loss, and microbial growth. This study assessed the effect of various concentrations of chia or nopal seed mucilage adjusted to an alkaline pH on the water vapor permeability properties of the edible films derived from them.

II. METHODS

Raw materials.

The raw materials used in the film formulation included chia seeds (Salvia hispanica) purchased from the Hidalgo Market in Puebla and nopal (Opuntia ficus-indica) obtained from the San Pedro Cholula Municipal Market, Puebla

Mucilage extraction

To extract chia mucilage, a chia:water suspension in a 1:10 (w/v) ratio was continuously stirred for 20 minutes at room temperature. Subsequently, it was subjected to a water bath at 80°C for 10 minutes, followed by temperature reduction to 40°C to separate the chia seed mucilage through filtration. Regarding the nopal, fresh spineless cladodes were used. They were washed, brushed, cut into 1x1 cm pieces, and blended with distilled water in a 1:1 (w/v) ratio for 2 minutes. The blend underwent heating in a water bath at 80°C for 2 hours and was then centrifuged at 3500 rpm for 20 minutes to separate the mucilage.

Film production

To produce the edible films, the chia and nopal mucilages were adjusted to pH 11.0 using 3N KOH. Film-forming solutions were prepared with 1.5% (w/v) sodium alginate, 5.5% (w/v) glycerol, and varying proportions of mucilages: 20%, 30%, and 50% (w/v) for chia; 5%, 15%, and 30% (w/v) for nopal.

The solutions were homogenized for 5 minutes at 10,000 rpm and subjected to 121°C for 15 minutes. The casting method was employed to form the films, removing the solvent through drying at 45°C for 24 hours. The resulting films were stored at a water activity (aw) of 0.46 for a minimum of 24 hours before evaluating their properties.

Water vapor permeability (WVP) determination.

[18] The desiccant method by Joaqui et al. (2013) was employed. Pesafilters were filled with silica gel, dried at 85°C for 24 hours (0% RH), and covered with the film, sealing them with Parafilm. These were then placed in a desiccator with a saturated NaCl solution (75% RH) at 25°C. Periodically, the weight gain (ΔW) of the pesafilters, indicating the water vapor passage through the bioplastic film, was determined. The permeability (P) was calculated using d(ΔW)/dt, considering the film's area (A) and thickness (E), along with the difference in vapor pressure (ΔP) and relative humidity (ΔRH) between the interior and exterior of the pesafilter: $WVP = (d(\Delta W)/dt) * A * E / (\Delta P * \Delta RH)$.

Statistical analysis

An ANOVA was conducted, considering the chia mucilage proportion (20%, 30%, and 50% w/v) as a factor, at a pH value of 11.0, with water vapor permeability as the response variable. The same procedure was applied for films containing nopal (5%, 15%, and 30% w/v). Significant differences between means (α=0.05) were analyzed, and variables were correlated by fitting a linear model depicting direct dependency

III. RESULTS

Considering that biopolymers carry electric charges, factors like pH can impact the stability of their structures. Common interactions that can induce either binding or repulsion between their molecules include hydrophobic, ionic, or hydrogen bonding interactions. In the formulations used in this study, two polysaccharides were employed: sodium alginate and either chia mucilage or nopal mucilage. This setup allows the molecules to interact, forming networks through more specific binding zones, thereby generating more robust gels.

[19]In other research, it has been demonstrated that pH affects the gel formation of chia mucilage, which can occur within pH values ranging from 3 to 12. However, gel formation does not occur at lower or higher pH values. Electron microscopy studies revealed the formation of fibers that construct a three-dimensional network, responsible for the gel formation.

The films were produced using three different concentrations of chia and nopal mucilages at alkaline pH values. Water vapor permeability was determined, an essential characteristic for assessing water barrier properties.

Both chia and nopal mucilage exhibited a linear relationship between WVP (Water Vapor Permeability) and mucilage concentration, depicting a positive slope where permeability values increased concerning the mucilage percentage. WVP showed a linear increase with rising mucilage concentration within the film matrix. The slope of the linear regression fitted to the data indicates that WVP increased by 10^-10 g/Pa·s·m for each percentage of added mucilage. In this experimental study, a direct relationship was observed between mucilage concentration and water vapor permeability (WVP) of the edible films produced, both from chia mucilage (Figure 1) and nopal mucilage (Figure 2). However, despite increasing levels of mucilage elevating WVP, films with lower proportions of this component (20-30% for chia; 5-15% for nopal) exhibited better barrier properties, displaying the lowest values (1.2 *10-8 g/Pa·s·m).

These outcomes could be linked to the distinct structures of the polysaccharides. Chia mucilage is a linear polymer, comprising a glucose, xylose, and glucuronic acid tetramer in a 1:2:1 ratio, featuring a sugar unit as a side chain. Conversely, nopal mucilage possesses a highly branched complex structure, showcasing extensive interactions between its constituent monosaccharides, resulting in numerous hydrogen bonds among its free hydroxyl groups. This possibly led to the films made from nopal mucilage exhibiting lesser water interaction, evident in the attainment of stiffer films with higher mucilage concentrations, unlike those derived from chia mucilage, where a 50% mucilage concentration was viable. Additionally, it has been reported that nopal mucilage at pH 10 exhibits lower solubility and water retention than at its original pH (5.8). This phenomenon is attributed to the polysaccharide being more ionized under alkaline conditions, which proves advantageous for water barrier properties.

[21] The water vapor permeability values obtained in this study are higher compared to those reported in other studies, which typically range around 0.33 *10-10 g/Pa·s·m for chia mucilage films. However, these studies utilized glycerol concentrations of 0.5% in their formulations.

In this study, the percentage of glycerol used in the formulations (5.5%), acting as a plasticizer by reducing the interaction between biopolymer molecules, is highly hygroscopic. This property favors the film's interaction with water, increasing its absorption capacity, thus making it more permeable and less efficient as a water vapor barrier.

A one-way analysis of variance (ANOVA) was conducted to statistically establish the impact of the mucilage percentage on water vapor permeability (WVP).

The statistical analysis using ANOVA demonstrated significant differences ($p<0.05$) in water vapor permeability (WVP) among films made with different proportions of chia and nopal mucilages (Table 1 and Table 2). These results validate that the variation in mucilage percentage effectively impacts barrier properties, dismissing random effects. Thus, it confirms that the precise formulation of plant mucilage concentration allows the regulation of water vapor permeability properties in these edible matrices, proving to be a useful strategy for developing protective coatings based on mucilages.

Table 1 summarizes the ANOVA results for chia mucilage.

Table 2 presents the summary for nopal mucilage.

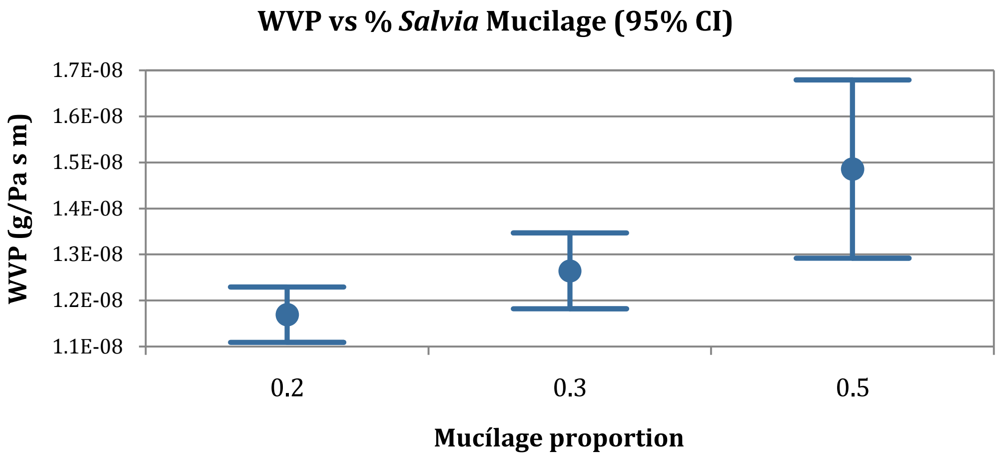

FIGURE 1. GRAPHICAL REPRESENTATION DEPICTING THE MEANS AND CONFIDENCE INTERVALS OF WVP IN CONTRAST OF SALVIA'S MUCILAGE PROPORTION.

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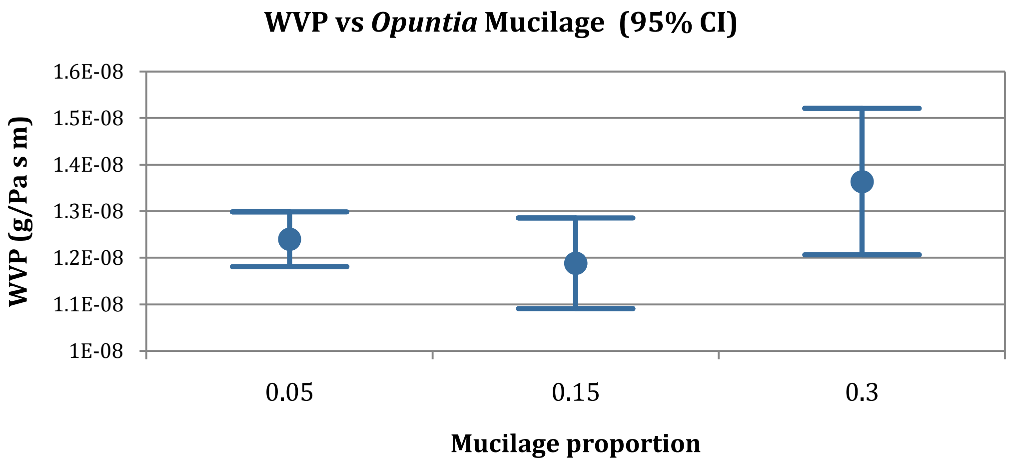

FIGURE 2. GRAPHICAL REPRESENTATION DEPICTING THE MEANS AND CONFIDENCE INTERVALS OF WVP IN CONTRAST OF OPUNTIA'S MUCILAGE PROPORTION.

ANOVA Salvia

Sourceof Variación	d.f.	SS	MS	F	p-value
Between Groups	2	0.0000	0.0000	16.6818	0.0009
WithinGroups	9	0.0000	0.0000		
Total	11	0.0000			

TABLE 1. ONE-WAY ANOVA TEST AMONG WVP VS SALVIA'S MUCILAGE PROPORTION.

ANOVA Opuntia

SourceofVariación	d.f.	SS	MS	F	p-value
Between Groups	2	0.0000	0.0000	6.5540	0.0175
WithinGroups	9	0.0000	0.0000		
Total	11	0.0000			

TABLE 2. ONE-WAY ANOVA TEST AMONG WVP VS OPUNTIA'S MUCILAGE PROPORTION.

IV. CONCLUSION

Edible films were produced using chia and nopal mucilages adjusted to pH values of 11.0, with water vapor permeability (WVP) values ranging between 1.16 – 1.48*10-8 g/Pa·s·m. Films exhibiting higher efficiency in permeability were those made with nopal mucilage at lower concentrations (5% and 15%). The ANOVA statistically verifies that the mucilage proportion significantly alters water vapor permeability in these edible films.

Nopal mucilage was once considered waste in certain processes; however, due to the functional properties recently attributed to it, it is being incorporated into numerous formulations within the food industry. Presently, various studies are underway to apply these types of polymers in the production of edible films, not only for the advantages they confer as coating agents for food but also for evaluating their potential to generate functional films that may have beneficial effects on health either independently or as carriers of bioactive substances.

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