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Investigating physical and mechanical properties of edible film based on pectin and whey protein, containing thyme essential oil using Pickering nanoemulsion method

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ABSTRACT

In recent years, the use of edible films has attracted the attention of many researchers. These films have contributed a lot to the environment due to their biodegradable nature. In this research, the edible film based on pectin and the cultivation of cheese juice contains thyme essential oil emulsion, which was investigated by three methods (condenser under vacuum with oven, nano spray dryer and Tween 80). The findings of the investigation of the film structure and the amount of particle dispersion on the surface of the films showed that the roughness and roughness of the film sample was reduced by adding thyme emulsion from Tween 80. The results indicated that by adding thyme emulsion to edible films based on pectin and cheese culture, the permeability to water vapor decreased significantly. Also, the results showed that by adding the thyme emulsion obtained from the thickener under the influence of vacuum and drying, its increase rate was reduced by adding the thyme emulsion obtained from Tween 80. Adding the thyme emulsion obtained from the vacuum concentrator and nano spray dryer reduced the light permeability of the films. The colorimetric results of the samples showed that the brightness increased with the addition of thyme emulsion produced with a vacuum concentrator and nano spray dryer, but the brightness decreased with the addition of thyme emulsion obtained from Tween 80. The results related to L* indicated a significant difference between the samples of drying powder and thickener under vacuum with the control sample, but no significant difference was observed between the sample of Tween 80 and the sample. The b* index showed that all samples show results with a difference of 5%.

1-Introduction

Films and edible coatings are placed around the desired food materials as thin layers that can facilitate moisture, oxygen, and fat exchange between the environment and the food substance while preventing the reverse transfer. Edible films consist of thin layers of edible materials that are produced separately from the food materials and then applied onto the food. Edible films can be used in the form of bags, capsules, and wraps. Unlike films, edible coatings are formed directly on the food substance. This process is carried out using methods such as waxing, spraying, and dipping[1, 2].

Films and edible coatings must possess suitable characteristics and properties for packaging food materials. The components used in films and edible coatings should be able to prevent moisture and oxygen permeation into the food materials. Additionally, these materials should have desirable mechanical properties and be non-toxic. These properties depend on the type of food material used, the method of formation, and how they are applied[3].

Edible films and coatings can transport various active ingredients such as emulsifiers, antioxidants, antimicrobial agents, flavorings, and colors, leading to improved quality and safety of the food substances. Edible films containing antimicrobial agents such as essential oils and plant extracts are a novel idea in the packaging industry, capable of inhibiting microbial growth on the food surface. Controlled amounts of antimicrobial substances are added to edible films for this purpose[4].

Edible films with essential oils (EOs) are becoming increasingly popular as an alternative to synthetic packaging due to their environmentally friendly properties and ability as carriers of active compounds. By incorporating EOs nanoemulsions into the packaging matrix, these edible films can help to extend the shelf-life of food products while also improving the quality and safety of the food product during storage. It can be concluded that these edible films have the potential to be used in the food industry as a green, sustainable, and biodegradable method for perishable foods preservation[5].

PE[1]s-added packaging film and their application in the enhancement of shelf life of food. Pickering emulsion is an effective approach for the high stabilization of EOs. For the development of PE various edible and biodegradable polysaccharides such as cellulose, chitosan, pectin and protein such as gelatin, and zein are useful. There are many works have been done on PE but only recently some significant attention has been given to the food packaging section[6].

Recent advances show that composite particles combining polysaccharides and proteins have higher stability and functionality compared to single particles due to their optimized interactions at the interfaces. Future research should focus on developing scalable, cost-effective production methods and conducting comprehensive environmental testing and regulatory compliance, particularly for nanotechnology-based packaging. These efforts will be crucial to drive the development of safe and effective biobased active food packaging[7].

Pectin is a hydrophilic compound composed of galacturonic acid units linked by alpha-1, 4 bonds. In the food industry, pectin is used as a stabilizer and gelling agent in products such as jams. It is also used in the production of fruit beverages, low-calorie products, and ice cream. This substance can also be utilized in the production of edible films (Mojaddad, 2016). Whey proteins (also known as serum proteins or whey proteins) are a type of natural digestible material used in food packaging and pharmaceutical industries. The proteins present in milk are divided into two main groups: caseins (80%) and whey proteins (20%)[8].

Thyme, with its essential oils and various medicinal chemical compounds, is one of the most commonly used medicinal plants worldwide. Among medicinal plants used for drug production, thyme ranks second after mint (Nazrli et al., 2017). Recent studies have shown that Shirazi thyme has a wide range of biological properties such as analgesic, antimicrobial, antioxidant, and anti-inflammatory effects[9].

Encapsulation, or microencapsulation, is one of the most important methods used for protecting and increasing the stability of food compounds

1-Pickering emulsion

and controlled release of active substances in food products. Encapsulation technology, as a unique method for packaging materials in nano-sized particles and trapping an active substance within another substance, is rapidly advancing. In the food industry, polyphenols, additives, colors, enzymes, and bacteria are enclosed in small capsules to provide stability and protection against food hazards[10].

In a study, pectin-based biodegradable films were produced to increase the solubility time of these films in water with the help of cationic solutions. The aim of the study was to enhance the solubility time of pectin films in water using cationic solutions[11]. In a research study, the effect of two plasticizers, glycerol and polyethylene glycol, on the structure of pectin films was examined. The results showed that glycerol acts as an internal plasticizer. It was found that glycerol causes the amorphous part to dominate the film structure. In this state, intermolecular attraction decreases, ultimately leading to the breakdown and formation of a new structure in the galacturonic acid loop. It was evident in this study that glycerol produces weaker films. Glycerol also increased the water vapor permeability of the films. When polyethylene glycol was used as a plasticizer in the produced films, it was revealed that the films produced were weaker compared to pectin films without a plasticizer, and the Young's modulus decreased[12].

In a study was to prepare the thyme essential oil (TEO) nano-emulsion (5, 10, 15 and 20%) incorporated edible composite films using tamarind starch (TS), whey protein concentrate (WPC) at selected proportions. The average particle size of nano-emulsions was found to be 18.88 nm and PDI value of 0.304. Synergistic effect of WPC and TEO significantly improved the tensile strength and elongation properties of the films[13].

Loureiro et al. [14] examined the effect of calcium and potassium ions on the coagulation and water retention in a hydrogel. The study showed that the presence of calcium and potassium ions led to a more stable hydrogel structure. The pH value of 3 resulted in a significant increase in water retention of the hydrogels. They also found that adding cornstarch to the hydrogel at a concentration of 5 to 20% increased the water absorption capacity. The addition of cornstarch at 20% concentration significantly decreased the softness of the hydrogel particles[14].

In a study conducted by Shi et al. [15], the properties of corn starch-based hydrogels containing anionic and cationic surfactants were investigated. The particle size distribution and polydispersity index (PDI) of the hydrogel particles showed a more homogeneous structure with a lower PDI. The addition of corn starch resulted in a decrease in the water absorption capacity and swelling of the hydrogel particles. It was observed that the hydrogel particles with corn starch had better dispersion in the anionic/cationic surfactant solutions. The water absorption of corn starch hydrogels increased with the addition of corn starch, and the swelling ability of the particles improved significantly. The addition of moisture to the hydrogel particles led to an increase in the swelling of the hydrogel particles in water, with a more porous structure[15].

Cabello et al. [12] studied the effect of two plasticizers, glycerol and polyethylene glycol, on the structure of pectin films. Their research showed that glycerol acts as an internal plasticizer. They found that glycerol causes the amorphous part to predominate in the film structure. In this state, the intermolecular attraction decreases, ultimately resulting in the breakdown and formation of a new structure in the galacturonic acid loop. It was revealed in this study that glycerol produces weaker films. Glycerol also increased the water vapor permeability of the films. When polyethylene glycol was used as a plasticizer in the produced films, it was observed that the films produced were weaker compared to pectin films without a plasticizer, and the Young's modulus decreased.

In a study to incorporate thyme essential oil into films composed of pectin to provide antimicrobial action to them. The films containing thyme essential oil were more elastic and thicker but less resistant, with high permeability to water vapor and more hydrophilic relative to other formulations. Scanning electron microscopy analysis showed the presence of heterogeneities in the formulations with essential oil[16].

Tavares et al. [17] investigated the physicochemical properties and microstructure of a composite edible film composed of chitosan and whey protein. The results of their study showed that adding a small amount of chitosan to the whey protein matrix led to the formation of a composite film with high tensile strength and low flexibility.
Stabilized emulsions with solid particles instead of surfactants are called Pickering emulsions [18]. Due to their wide range of applications in food, pharmaceuticals, and cosmetics, Pickering emulsions have attracted significant attention compared to conventional emulsions that use surfactants, which may have toxic effects. In these emulsions, the particles are irreversibly absorbed due to their high energy attachment at the oil-water interface, resulting in Pickering emulsions that are stable for months or even years. Furthermore, Pickering emulsions maintain the primary structure of classic emulsions[19].
In a study clove essential oil (CEO) loaded nano and pickering emulsions prepared with Tween 80 and whey protein isolate/inulin mixture, respectively were incorporated into pullulan-gelatin film base fluid at three levels (0.2%, 0.4%, and 0.6%). The results showed the improved compatibility between pullulan-gelatin and essential oil-loaded nanocarriers. The active film composed of PE carrier had the structural characteristics of high density, low water content, and low permeability, thus exhibiting excellent mechanical properties, water barrier properties, and appreciable antioxidant activities[20].
In a study, genipin-crosslinked gelatin films incorporated with Pickering emulsion containing cinnamon essential oil (GPG) were fabricated by solution casting method. Results demonstrated that antibacterial GPG featuring excellent mechanical properties were successfully obtained. The Pickering emulsions were confined within the network, averting the aggregation of particles during film formation, thus engendering a uniform and compact surface morphology[21].
Vartiainen et al. [22] examined the effects of montmorillonite nanoparticles on the barrier properties of films produced from sugar beet pectin. In this research, pectin films with concentrations of 10, 20, and 30 percent were produced using the casting method. The findings of the study showed that the nanocomposite films produced from pectin and montmorillonite exhibited barrier properties against oxygen and water vapor.
In a study, the water vapor permeability of emulsion films made from whey protein-fat under different relative humidity conditions was investigated. Initially, a 10% weight-to-weight solution of whey protein isolate was placed in an oil bath at 90 degrees Celsius for 30 minutes. Then it was cooled to room temperature and sorbitol was added. To produce emulsion films, whey protein-sorbitol solutions were heated to 75 degrees Celsius and fatty acids, fatty alcohols, and beeswax were added to determine their effects on water vapor barrier properties. Finally, the molded solutions were dried at 23 degrees Celsius for 18 hours. The results showed that adding fat to films made from whey protein reduced water vapor permeability[23]. Pérez-Gago, Krochta [24] investigated the effects of heating time and solution temperature on the tensile properties and oxygen permeability of whey protein films. In this study, a 10% whey protein isolate solution was heated in a warm water bath at temperatures of 70, 80, 90, and 100 degrees Celsius for 5, 10, 15, and 20 minutes, and glycerol was added as a plasticizer to all forming solutions. The results indicated that increasing the temperature and heating time of the film-forming solution resulted in firmer and more extensible films. Moreover, the oxygen permeability of the heat-denatured whey protein films was lower compared to the non-heated films.
The aim of this research was to produce a type of edible film based on pectin and whey protein along with stabilized emulsions of corn zein, chitosan, and Tween 80, and compare it with the Pickering emulsion method containing thyme essential oil.

2.Matherial and method

Materials Used

The materials used included: chitosan (Sigma Aldrich, USA), pectin (Silva Company, Italy), whey protein (Helymar, USA), corn zein (Zarin Zein Company, Iran), glycerol (Merck, Germany), alcohol (Zakariya Jahrom Company, Iran), Tween 80 (Merck, Germany), and acetic acid (Merck, Germany).

Preparation of Thyme Essential Oil Emulsion using Chitosan-Zein Powder from Spray Dryer

To dry the powder, initially, 5.0 grams of chitosan powder was added to 100 milliliters of distilled water, then 2 milliliters of concentrated acetic acid was added to the previous solution. To improve the solubility of this solution, it was placed on a stirrer at 35 degrees Celsius for 15 minutes. Simultaneously, 5.0 grams of pure zein powder was added to 100 milliliters of 70% alcohol at room temperature and mixed on a magnetic stirrer. After preparing the desired solutions, they were combined and diluted with distilled water to a volume of 800 milliliters. Subsequently, the solution was subjected to ultrasonication for 15 minutes at a power of 60 watts. The solution was dried using a nano spray dryer (Figure 1) at an initial temperature of 64 degrees Celsius and converted to powder. The resulting powder was packaged and stored in a dry and cool place[25].

Preparation of Thyme Essential Oil Emulsion using Chitosan-Zein Powder from Rotary Evaporator

In this method, 45.0 grams of corn zein powder was added to a specific amount of 70% alcohol (30 milliliters) and mixed until fully dissolved. Then, 45.0 grams of chitosan were taken and dissolved in 90 milliliters of distilled water. It is worth noting that for better dissolution of chitosan in distilled water, 1 milliliter of acetic acid was added. Finally, both prepared solutions were combined, transferred to a rotary evaporator for concentration. After the rotary operation, 20 milliliters of thyme essential oil was combined with the solution obtained from the rotary evaporator and stirred on a heater for 2 minutes. The solution was then subjected to ultrasonication for 5 minutes[26].

Preparation of Thyme Essential Oil Emulsion using Tween 80

To prepare the Tween emulsion, the same method as the previous ones was used. In this method, 20.0 grams of thyme essential oil was combined with 0.68 grams of Tween 80, then mixed with 65 milliliters of distilled water[26].

Preparation of Edible Film based on Pectin and Whey Protein with Thyme Essential Oil Emulsion from Nano Spray Dryer

In this method, after preparing the film solution with a ratio of 2% pectin and 5% whey protein, 2.5% of the dry substance of the thyme essential oil emulsion obtained from the nano spray dryer was added. The solution was subjected to ultrasonication for 5 minutes. Finally, the prepared solution was poured into a plate and kept at room temperature for 24 hours[27].

Preparation of Edible Film based on Pectin and Whey Protein with Thyme Essential Oil Emulsion from Vacuum Concentrator

To prepare the edible films based on pectin and whey protein containing the thyme essential oil emulsion obtained from the vacuum concentrator, the film solution was initially prepared (with a ratio of 2 to 5 pectin to whey protein). Then, the thyme essential oil emulsion was added to the film solution, and the solution was subjected to ultrasonication for 5 minutes. After the ultrasonic treatment, the solution was poured into glass plates and kept at room temperature for 24 hours to form the film.

Preparation of Edible Film based on Pectin and Whey Protein with Thyme Essential Oil Emulsion from Tween 80

The third type of prepared films corresponds to the edible films based on pectin and whey protein containing the Tween 80 emulsion. In this method, similar to other methods, the film solution was prepared with a ratio of 2 to 5 pectin to whey protein. Then, 25 milliliters of the Tween 80 emulsion was added to the film solution, and the combined solutions were centrifuged for 5 minutes at 3000 RPM to prevent bubble formation in the films. After the completion of centrifugation, the solution was subjected to ultrasonication for 5 minutes. Finally, the edible films were poured into plates and kept at room temperature for 48 hours[27].

Measurement of Emulsion Particles using DLS

The size of the particles (emulsion droplets) was measured using the Dynamic Light Scattering (DLS) device (ZEN3600, England). For this process, a few drops of the sample were diluted with distilled water at a ratio of one to a thousand and added to a special cell up to the marked line. Then, the cell was placed inside the device, and with the help of the software installed on the computer connected to the Zetasizer, the corresponding graphs and numbers were obtained[26].

Examination of Particle Size and Morphology Using SEM

To investigate the size and morphology of particles produced using the spray dryer and solvent evaporation (rotary) methods, SEM was utilized. After obtaining the chitosan-zein powders from the spray dryer and rotary evaporator methods, a portion of it was taken, poured onto a lamella, and transferred to the SEM machine for the desired tests[27].

Measurement of Water Wettability using Contact Angle Method

To determine the contact angle, the sessile drop method, a common technique in determining the wetting properties of solid surfaces, was employed. In this method, a droplet of distilled water was placed on the film surface using a syringe. The contact angle between the dropped droplet and the film surface was captured using a Canon MV50 camera with a 6x zoom. Ultimately, the ImageJ software was utilized to calculate the contact angle of the water droplet with the film surfaces. This test was repeated three times for each film[28].

Investigation of the Structure of Prepared Films and Particle Dispersion on Film Surfaces with SEM

For the examination of large sample structures on or near the surface, a scanning electron microscope (SEM) was used. In this study, to assess the surface variations of the produced films, film thickness, and particle dispersion on the film surfaces, a scanning electron microscope was employed. The Ziees sigma 300-HV device from Germany was used for this test. The accelerator voltage of this device was set at 0.02-30 kilovolts. Additionally, the image resolution of this device was 2304×3072 pixels[29].

Determination of Mechanical Properties of the Prepared Films

For this test, a 2-ton tensile testing machine (STM-20) from Santam, Iran was used. In this test, the edible films were first cut into rectangular shapes with dimensions of 4×1. The machine was then set with a distance of 40 millimeters between the two jaws and a speed of 1 millimeter per minute for the two jaws[29].

Investigation of Film Permeability to Water Vapor

The film's permeability to water vapor was assessed according to the ASTM E96 standard method. Pennycillin glass tubes with an inner diameter of 3.2 and a height of 5 centimeters were used for this test. Initially, the Pennycillin glass tubes were weighed using a scale, then 10 milliliters of water were weighed and transferred into the tubes. After this step, the mouths of the Pennycillin glass tubes were sealed with water-resistant adhesive, and the prepared films, which had been precut to the outer diameter of the tubes, were placed on them. To ensure no leaks, the edges were covered with cellophane, and the Pennycillin glass tubes were weighed again using a scale with an accuracy of 0.0001 g and the weights were recorded. This process was repeated for three days at specific intervals (every 12 hours). It is worth mentioning that the relative humidity of the air on the test days was also measured. The vapor permeability was calculated using formula (1)[30].

$$WVP = \frac{\Delta m \times X}{A \times \Delta t \times \Delta p} \quad (1)$$

Δm = weight loss of the cup, X = thickness (m), A = exposed surface, and Δp = partial pressure difference (KPa) between inside and outside the cup, and Δt = time (S).

Measurement of Solubility and Moisture Content of Films

The solubility and moisture content of the produced films in water were determined using the method of Cuq et al. [31] and Silva et al. [25]. In this method, the produced films were initially placed in an oven at 105 degrees Celsius to dry completely. After drying, their initial weights were recorded. Then, the dried film samples were placed in containers containing 50 milliliters of distilled water. The containers were placed on a shaker at a speed of 60 revolutions per minute for 24 hours. After the specified time, the samples were smoothed with a strainer and placed back in the oven at 105 degrees Celsius to dry. Finally, the solvent solubility percentage and moisture content of the edible films were calculated using formulas (2) and (3).

(2) $$\%ws = \frac{w_0 - w_1}{w_0} \times 100$$

(ws) is the solvent solubility, (w0) is the initial weight of dried edible films, and (w1) is the weight of the dried film after immersion.

In formula (2): (ws) denotes the solvent solubility, (w0) represents the initial weight of dried edible films, and (w1) is the weight of the dried film after immersion.

(3) $$\%w = \frac{w_0 - w_1}{w_0} \times 100$$

In formula (3): (w) indicates the moisture content, (w0) is the initial weight of the edible films, and (w1) represents the weight of the dried film after drying.

Measurement of Color Intensity of Produced Films

In this study, to assess the color intensity of the produced films, photographs of samples were taken using a digital camera in 3 repetitions. Subsequently, color indices L, a, b, and ΔE were evaluated using Photoshop software[28].

Light Permeability in Edible Films

In this method, the edible films were initially cut into rectangles and attached with adhesive tape to one side of a spectrophotometer cell. The cell was then transferred to the spectrophotometer device, and the light absorption of the films in the spectrum (700 nanometers) was measured. A blank cell was also used as a control sample[28].

Statistical Analysis

In this study, the difference between different treatments was determined based on a completely randomized design using Analysis of Variance (ANOVA) at a 5% significance level. The comparison of means was performed using the Duncan test with SPSS software version 22. Additionally, the graphs were plotted using Excel software version 2019.

3.Result and Discution

Examination of Particle Morphology using SEM

The results related to the examination of the morphology of chitosan-zein corn particles produced using the rotary evaporator and spray dryer with the scanning electron microscope (SEM) are shown in Figure (1). The images obtained from the scanning electron microscope provide valuable information about the interaction and dispersion between the constituent particles. As shown in Figure (1), the image corresponds to the powder obtained from the spray dryer. In some areas, the particles from the spray dryer powder have been reduced to nano-sized and are easily distinguishable. However, in the image related to the powder obtained from the rotary evaporator, no such distinction is evident. Based on the above figure, the creation of Pickering nanoemulsions is likely occurring.

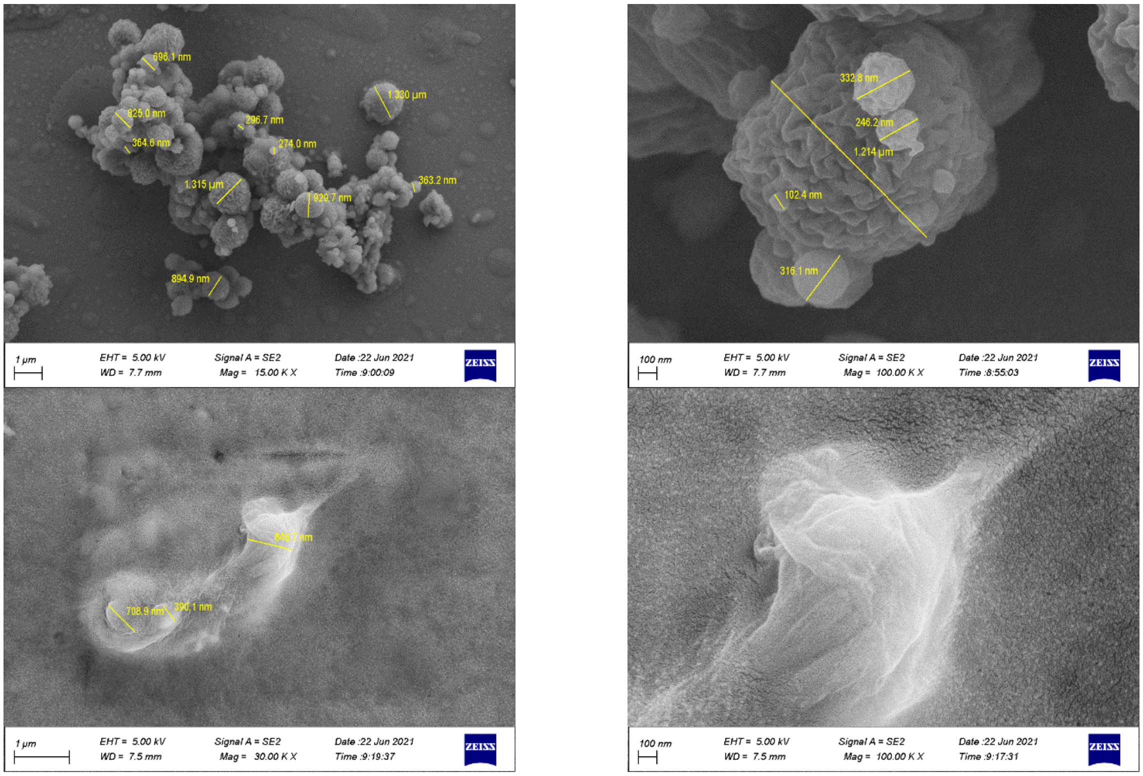

Figure 1: Image of the cross-sectional surface of chitosan and corn zein particles produced using the spray dryer and rotary evaporator.

Results of Emulsion Particle Measurement using DLS

One of the non-destructive physical methods for determining the distribution and size of particles in solutions and emulsions is the use of Dynamic Light Scattering (DLS). This method utilizes visible light radiation. Generally, DLS measures the intensity of scattered light present in the suspension. In Figure (3), the particle sizes of the emulsions produced using spray dryer, rotary evaporator, and Tween 80 methods are shown. As observed in this figure, the largest particle size corresponds to the emulsion produced using the powder obtained from the spray dryer, and the smallest particle size is associated with the emulsion produced using the rotary evaporator. Additionally, no significant difference in the level ($p > 0.05$) was observed among the samples.

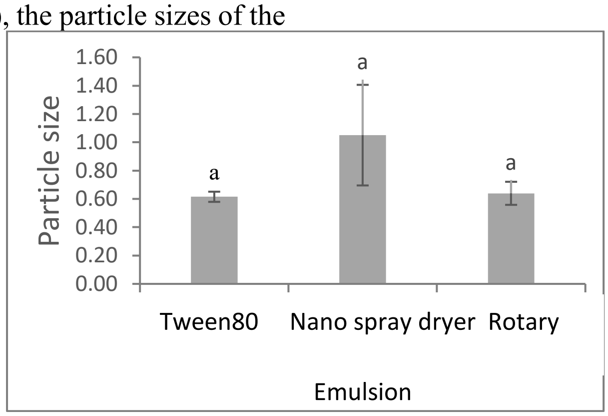

Figure 2: The Effect of Emulsion Type on Particle Dispersion (Particle Size)

Results of the examination of the structure of prepared films and the dispersion of particles on the film surfaces using SEM

In Figure (3), a view of the produced films is presented. In Figure (4), the results of the examination of the structure of composite edible films based on pectin and whey protein containing thyme emulsions prepared using methods (vacuum concentrator, nano spray dryer, and Tween 80) and the dispersion of particles on the film surfaces using scanning electron microscopy are shown. In the control sample, a uniform and homogeneous structure is not observed on the film surface. As evident in the image, adding thyme emulsion obtained from Tween 80 reduces the roughness and ruggedness of the film sample. The film containing thyme emulsion obtained from the vacuum concentrator exhibits uniform dispersion, and the roughness of this film is much lower compared to the other produced films. This is due to the uniform distribution of thyme emulsion droplets obtained from the vacuum concentrator powder and the appropriate stability of this emulsion during the film production process. The films containing thyme emulsion obtained from the nano spray dryer, despite having a rough structure similar to the control and Tween 80 films, have a more uniform dispersion compared to these two films. The thickness of the produced films is one of the important factors in food packaging that affects the microbial properties and the shelf life of food products. Images related to the thickness of the produced films are shown in Figure (5). The results indicate that the thinnest thickness in the produced films is related to the control sample with a size of 34.94 micrometers. By adding thyme emulsions obtained from the nano spray dryer, vacuum concentrator, and Tween 80, the thickness of the produced films increased, meaning that the distribution of thyme emulsion in the edible films led to an increase in the volume of the films. The highest thickness is related to the edible films produced containing thyme emulsion obtained from the vacuum concentrator with a thickness of 122 micrometers. Ghanbarzadeh, Oromiehi [32] examined the mechanical properties of biodegradable composite films based on whey protein and corn zein. They stated that the roughness of the surface of the edible films prepared based on corn zein containing glycerol softener was 7.19 nanometers, and the roughness of the whey protein edible film containing glycerol was 1.79 microliters. However, regarding the elements in the films, in the control and Tween films, due to the absence of chitosan and zein, potassium and phosphorus elements are not observed, and sulfur and calcium elements are likely derived from these two elements as potassium and phosphorus are not observed, and sulfur and calcium elements are likely combined. Also, the reason for the high presence of sodium and chlorine in the control and Tween films is probably due to the crystallization of salt on the film surface, which has caused a noticeable decrease in film surface roughness compared to the other two films, but in the two films that have been Pickeringized with chitosan and zein emulsion, salt has not appeared on the surface, and the effect of these two substances on the surface is more pronounced. Zanganeh et al. [2] stated in their research that the film produced using whey protein has a smooth, continuous, and uniform surface but uneven, in the form of fine wrinkles. Huang et al. [33] examined the effect of 14 hydrocolloid obtained from various gums on the characteristics of emulsions in their research. They reported that adding gum reduces the particle size and stabilizes the emulsions. Hosseini et al. [34] examined the effect of Balangu seed gum and whey protein on the stability of oil-in-water emulsions. They stated that by increasing the concentration of gum to 15.0%, the particle size decreased, but afterward, the particle size increased. Silva et al. [25] stated that adding pectin causes a significant change in the structure composition of the whey protein film surface. They stated that whey protein films without pectin have a smooth, continuous surface and a more uniform structure compared to films with added pectin because with an increase in pectin concentration, the heterogeneity of the film structure increases. Table (1) shows the constituent elements of edible films containing thyme emulsion.

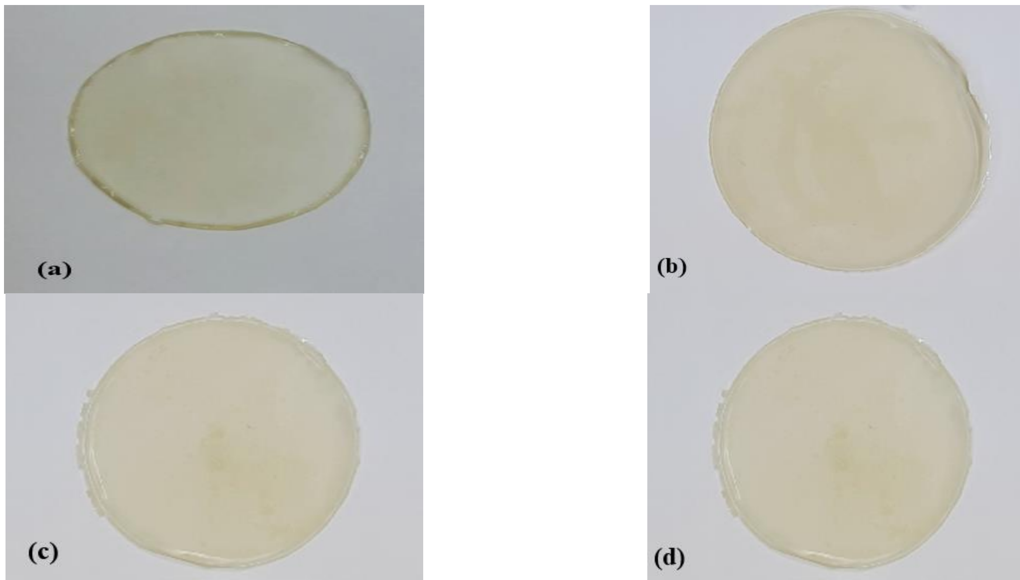

Figure 3: Edible films produced based on pectin and whey protein: Control sample (a), sample produced with thyme essence in the form of emulsion stabilized with zein-chitosan particles obtained from the nano spray dryer (b), sample produced with thyme essence in the form of emulsion stabilized with zein-chitosan particles obtained from the vacuum concentrator (c), sample produced with thyme essence in the form of emulsion stabilized with Tween 80 (d).

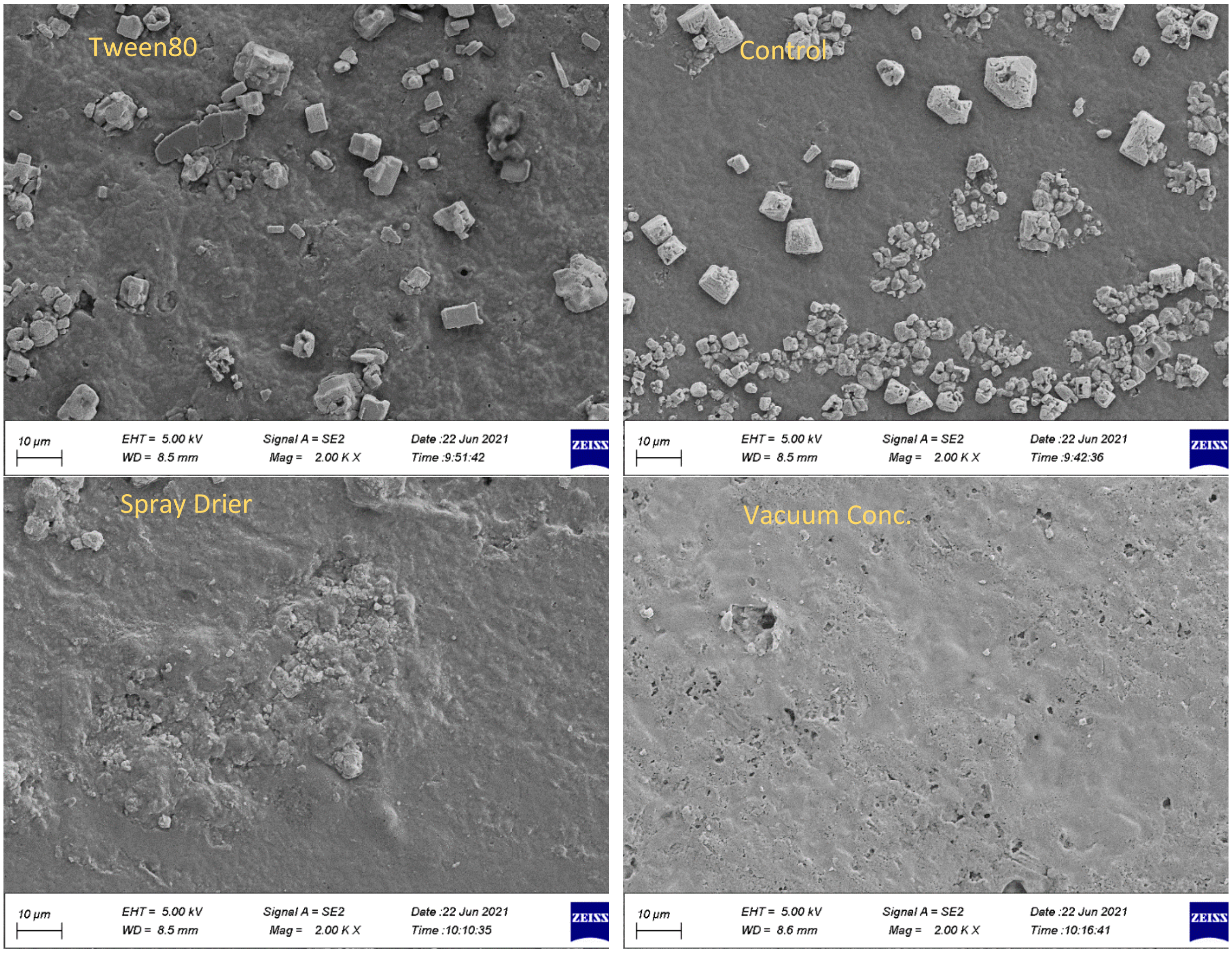

Figure 4: Images from the scanning electron microscope (SEM) of the edible films based on pectin and whey protein containing thyme emulsion obtained from zein-chitosan particles and Tween 80

Regarding the absence of sulfur, calcium, potassium, and phosphorus elements in the two control films and those containing Tween, it is likely due to the presence of the emulsion obtained from zein and chitosan particles in the film. Whereas, in the other two films, these particles are visible. Additionally, the elements present in the SEM analysis regarding the emulsion with the nano spray dryer and the vacuum concentrator along with Tween are observed. Furthermore, in the two control and Tween films, due to the NaCl particles crystallized on the film surface, it is likely due to salt crystals. However, in the other two films, NaCl compounds are less visible, probably due to Pickering, which prevents crystal formation on the surface, resulting in a smoother surface.

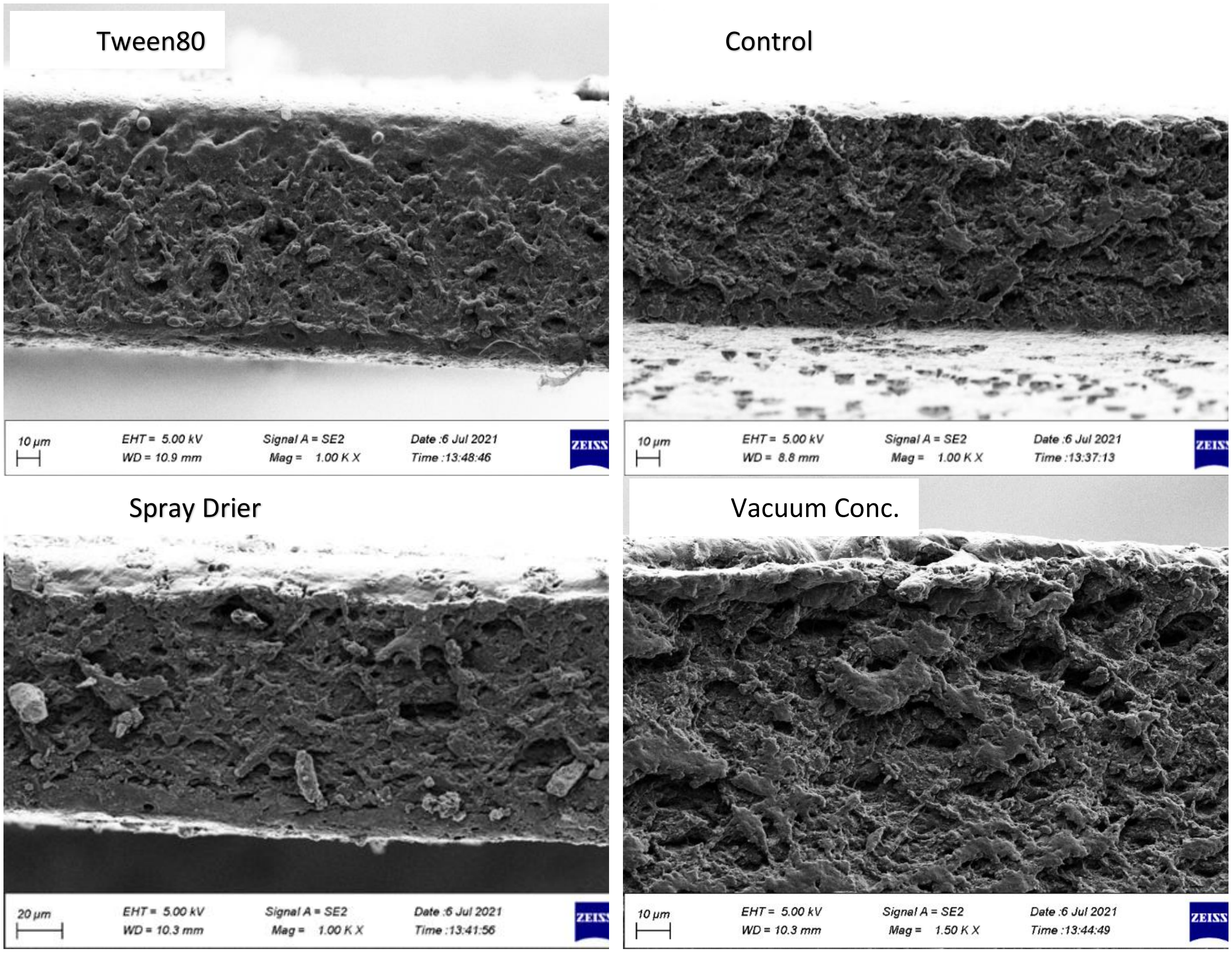

Figure 5: Images of thickness for samples of edible films based on pectin and whey protein containing thyme emulsion obtained from zein-chitosan particles and Tween 80 using a scanning electron microscope (SEM)

Table 1: Constituent elements of the edible films containing thyme emulsion (part of the SEM data)

Elements	Control	Tween	Vacuum Concentrator	Nano Spray Dryer
C	44.96	41.07	79.28	71.48
O	6	5.19	15.37	19.70
Cl	22.20	23.97	3.25	4.22
Na	27.11	29.77	1.39	4.36
S	–	–	0.29	0.10
K	–	–	0.19	0.07
Ca	–	–	0.13	0.03
P	–	–	0.10	0.04

Measuring Hydrophilicity Using Water Contact Angle Method

Many researchers categorize surfaces into three general groups based on the contact angle measurement. The first group consists of surfaces with a contact angle of less than 30 degrees. In this group, a water droplet completely wets the surface, making it hydrophilic. The second group includes surfaces with a contact angle between 30 and 89 degrees, indicating that these surfaces are relatively hydrophilic. The third group comprises surfaces with a contact angle greater than 90 degrees, commonly referred to as hydrophobic surfaces.

In general, the contact angle is defined as the angle formed at the intersection of two tangent lines to a liquid (or solid) surface at the point of contact with air, serving as an indicator of the hydrophobicity of the studied surface[35]. In the present study, the contact angle of the control sample was approximately 55 degrees, while other samples showed angles of less than 30 degrees. Based on the results obtained from the analysis of variance, it can be concluded that the composite film of pectin-protein with thyme emulsion is hydrophilic. Figure 1 illustrates the results of measuring hydrophilicity using the water contact angle method for the film samples. The contact angle decreased in all samples except for the control sample. The lowest contact angle was noted for the edible film containing thyme emulsion derived from Tween 80, with a contact angle of 21.07 degrees. Additionally, the results indicated that the contact angle of the surface with water increases as the hydrophobicity of the surface increases. The contact angle of the control film showed a significant difference compared to other samples at a significance level of ($p<0.05$).

In another study, the effects of oleic acid and glycerol on the permeability characteristics, contact angle, and appearance of edible films made from carboxymethyl cellulose were evaluated. Their results indicated that oleic acid increases the surface hydrophobicity (contact angle) of the films, while glycerol reduces surface hydrophobicity. They also noted that increasing glycerol content in films containing oleic acid resulted in a decrease in the contact angle. The findings from this research were consistent with those of the current study[32].

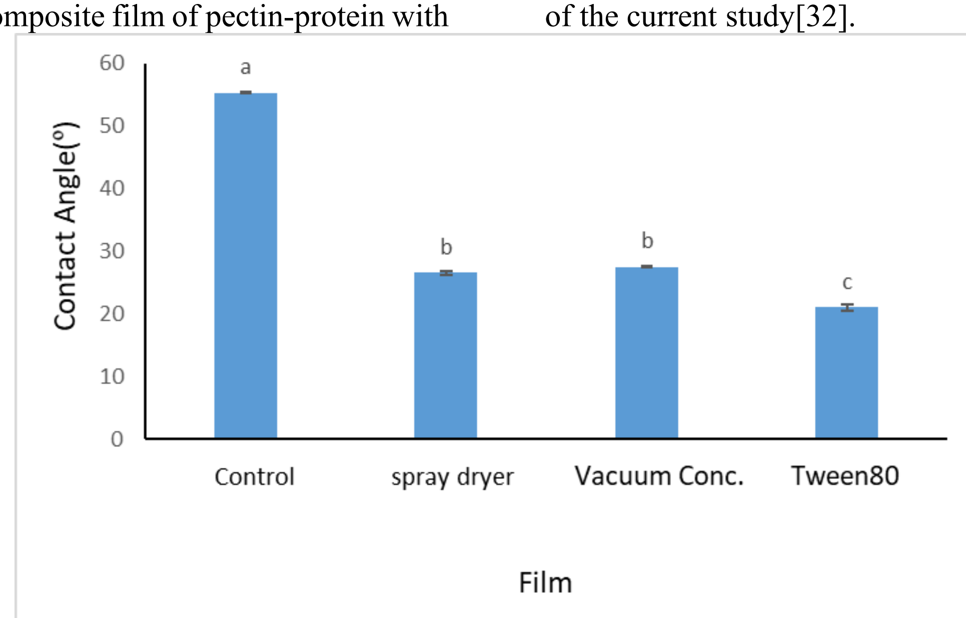

Figure 6: The contact angle of edible films made from pectin and whey protein containing thyme emulsion derived from zein-chitosan particles and Tween 80.

Investigation of the Permeability of Films to Water Vapor

One of the most important indicators in edible films used for food packaging is the permeability of these films to water vapor. The mechanism involved in water vapor permeation through the film is diffusion, where vapor dissolves in the film matrix from an area of high concentration to an area of lower concentration. The structure and characteristics of the film determine how this permeability will manifest. When protein is heated, the chain length and structural coherence of the matrix increase due to covalent disulfide bonds formed between the polypeptide chains[3]. Generally, more coherent films have less porosity and, as a result, lower water vapor permeability[36, 37]. Figure (7) shows the permeability of pectin-protein-based edible films to water vapor. As seen in this chart, the control sample exhibited the highest permeability to water vapor among the other produced edible films. Adding thyme emulsion reduced this permeability. Furthermore, the edible film containing thyme emulsion obtained from a vacuum concentrator showed a significant difference at the level ($p>0.05$) compared to the other samples. No significant difference was observed among the edible films containing thyme emulsion derived from Tween 80 and the spray-drying device, but they were significantly different from the other samples. The lowest permeability in this study was associated with the edible film containing the emulsion obtained from Tween 80. The results indicated that adding thyme emulsion to the pectin and whey protein-based edible films significantly reduced the permeability. In a study, the physicochemical properties of the whey protein-monoglyceride edible film and its effect on moisture loss and sensory characteristics of fresh sheep meat were examined. Their research findings showed that adding monoglyceride to the whey protein-based edible film resulted in decreased permeability to water vapor[38]. A study also showed that by adding a small amount of pectin up to 0.5% to whey protein-based edible films, the permeability of the films increased. The reason for this was attributed to a reduction in the structural coherence of the protein matrix[25]. The combination, nature of each component, and basically the interaction between these components, significantly affects the permeability of the film[39]. The addition of essential oils in edible films can have different effects on the WVP of the films[40]. On one hand, essential oils can slow down the moisture penetration rate by increasing the tortuosity of the pathway[41]. On the other hand, essential oils can cause phase separation within the film matrix, resulting in the enlargement of pore sizes in the film, and thereby increasing the diffusion of gases or vapors[42, 43].

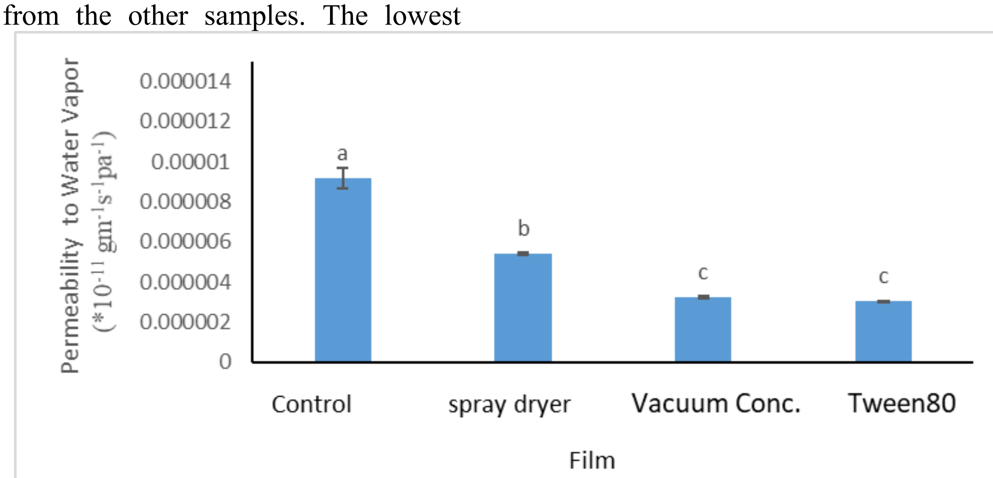

Figure 7: The permeability of edible films made from pectin and whey protein containing thyme emulsion obtained from a vacuum concentrator, nano spray dryer, and Tween 80.

The solubility of the produced films

The results from measuring the solubility of edible films based on pectin and whey protein containing thyme emulsion obtained from a nano spray dryer, vacuum concentrator, and Tween 80 are presented in Figure (8). The results showed a significant difference among the samples containing different thyme emulsions at a level of ($p<0.05$). It was also found that adding the thyme emulsion obtained from the vacuum concentrator and nano spray dryer increased the solubility of the edible films. However, adding the thyme emulsion derived from Tween 80 decreased the solubility of the edible films. In a study, the physicochemical and microstructural properties of whey protein isolate films with added pectin were examined. They stated that the whey protein films maintained their integrity after being submerged in water for 24 hours, which could be attributed to intermolecular disulfide bonds formed during the heat treatment of the whey protein solution among the polypeptide chains. They also noted that adding pectin to the whey protein films did not significantly change the amount of moisture absorbed per unit weight of dry solids[25]. Additionally, the results from the water affinity measurement in this study indicated that the composite films of pectin and whey protein containing thyme emulsion exhibited hydrophilic properties.

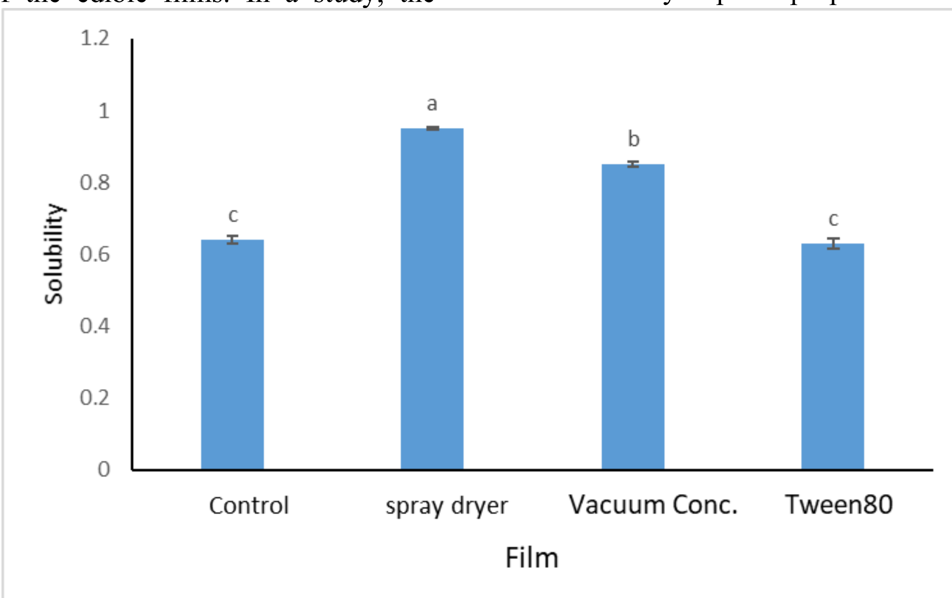

Figure 8: The solubility of edible films made from pectin - whey protein containing thyme emulsion obtained from a vacuum concentrator, nano spray dryer, and Tween 80.

The results of the study on the light permeability of films

The results of the study on the light permeability of edible films are presented in Figure (9). As shown in the chart, by adding thyme emulsion obtained from the vacuum concentrator and the nano spray dryer, the light permeability of pectin and whey protein-based edible films significantly decreased. However, with the increase of thyme emulsion derived from Tween 80, there was a slight reduction in light permeability. No significant difference was observed between the control sample and the sample containing Tween 80 emulsion at the level of ($p>0.05$). Additionally, no significant difference was found between the films containing emulsions obtained from the nano spray dryer and vacuum concentrator ($p>0.05$). Silva et al. [25] stated in their research that adding pectin significantly reduces the clarity of whey protein-based edible films. They also noted that increasing the pectin concentration from 0.5% to 1% significantly lowered the film clarity. The transparency of the films depends on the microstructure and structural arrangement of biopolymer molecules[39, 44]. Thermodynamic incompatibility between proteins and polysaccharides can lead to phase separation and discontinuity in the refractive index, which affects the film clarity[44]. Whey protein-based films are typically transparent and colorless. Yoo, Krochta [45] and Silva et al. [46] also found that adding polysaccharides reduces the clarity of whey protein-based edible films, linking the results to thermodynamic incompatibility between biopolymers.

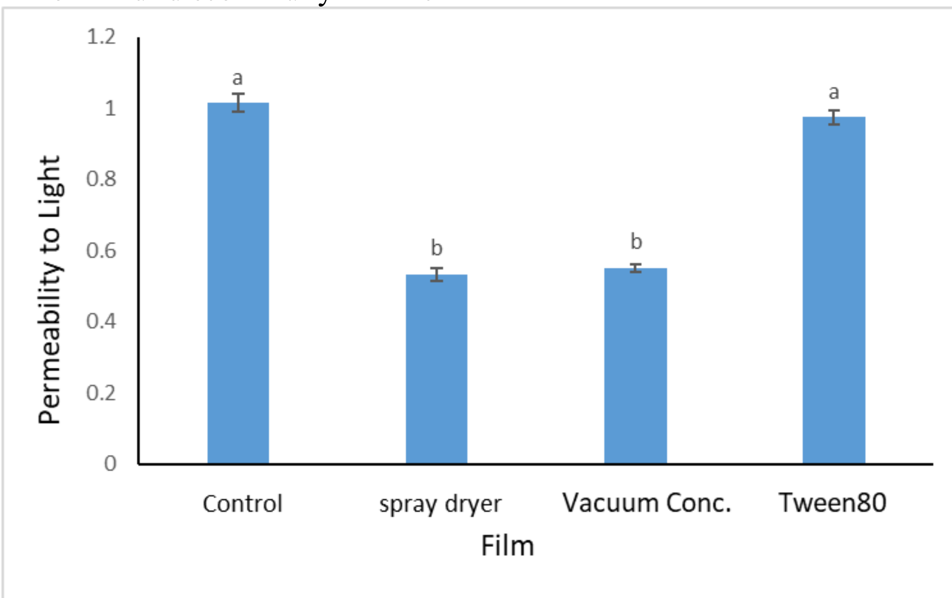

Figure 9: The permeability to light of food films made from pectin and whey protein containing thyme emulsion obtained from a vacuum concentrator, nano spray dryer, and Tween 80.

Studies on Coloring of Edible Films

The characteristics of edible films used in food packaging are extremely important, as the color of food is the first parameter that consumers perceive from the product, greatly influencing their acceptance of it. Many food consumers are very interested in seeing the true color of packaged foods to ensure the quality and freshness of the product.

The results obtained from the edible films produced are shown in chart (10-a). As demonstrated, the L* factor, which indicates the brightness of the edible samples, increased with the addition of thyme emulsion produced from the vacuum concentrator and spray dryer, but the brightness decreased with the addition of thyme emulsion obtained from Tween 80. As shown in chart (10-a), a significant difference is observed between the vacuum concentrator and spray dryer samples compared to the control sample, while no significant difference can be seen between the Tween 80 sample and the control sample. The a* parameter indicates the redness of the samples when positive and the greenness when negative. As shown in chart (10-b), a significant difference is observed between the vacuum concentrator and spray dryer samples compared to the control sample, while there's no significant difference (p>0.05) observed between the Tween 80 sample and the control sample. Additionally, the *b index, which indicates the yellowness of the samples when positive and the blueness when negative, is presented in chart (10-c). The results of this index showed that all samples had a significant difference at the level (p>0.05). Alizade et al. [1] examined the physical and antimicrobial properties of chitosan edible films containing resin essential oil. They reported during their research that the addition of resin essential oil increased the brightness of the samples. They also mentioned that the greenness of the samples (a*) decreased while the yellowness of the sample, indicated by the (b*) factor, increased. Zanganeh et al. [2] examined the characteristics of films made from seed mucilage and whey protein isolate. They reported that as the percentage of whey protein isolate increased, the lightness and greenness of the edible films improved, while the yellowness decreased. However, with an increase in the percentage of mucilage, the yellowness of the samples went up. Jo et al. [47] reported that as the dosage of radiation increased, the parameters L* and a* of the edible films made from pectin decreased, while the parameter b* increased. Weller et al. [48] investigated the effect of sorghum and carnauba waxes on edible films based on corn zein. Their research results showed that adding these waxy compounds to the corn zein edible films increased the turbidity of these films.

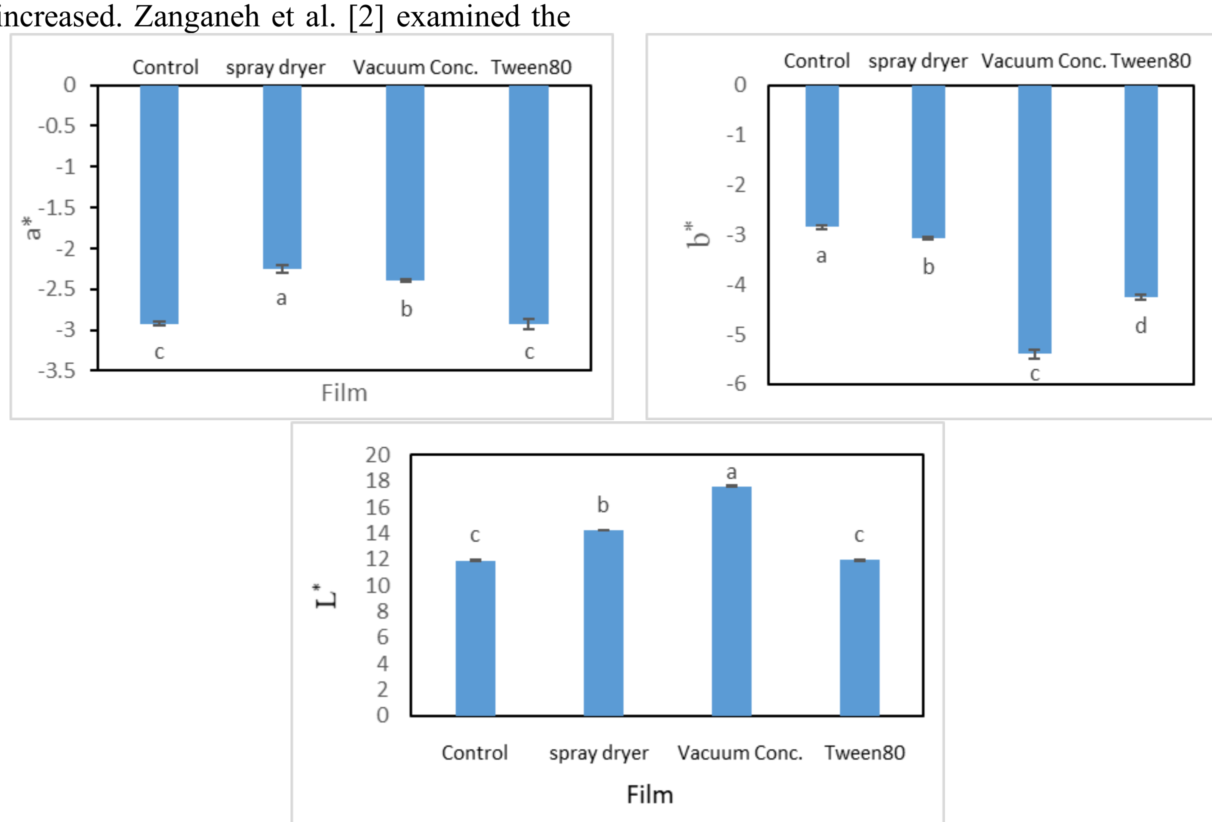

Figure 10: Color measurement of food films made from pectin-casein protein containing thyme emulsion obtained from a vacuum concentrator, nano spray dryer, and Tween 80.

Results of the Moisture Content in Edible Films

The results related to the edible films based on pectin and whey protein containing thyme emulsion derived from three methods (nano-spray dryer, vacuum concentrator, and Tween 80) are presented in figure (11). The results showed that the highest moisture content was found in the edible film containing thyme emulsion obtained from the vacuum concentrator, while the lowest moisture content was in the edible films containing thyme emulsion derived from the nano-spray dryer. Bazaria, Kumar [49] indicated that hot air reduces moisture levels. The results from the variance analysis of the edible films showed that the moisture content in all samples lacked significant differences at the ($p>0.05$) level. Zanganeh et al. [2] stated in his research that increasing the percentage of whey protein led to a decrease in moisture absorption of the edible films. This difference could be attributed to the type of polysaccharide and the percentage of isolated protein used in the edible films since increasing the percentage of isolated protein also increases the protein network, ultimately enhancing the binding to water molecules, resulting in higher moisture content in the films. R, Nur Hanani [50] reported that adding vegetable oils to kappa-carrageenan-based edible films reduces their moisture content. Edible films based on pectin have very good gas resistance, but due to their hydrophilic nature, they are highly sensitive to moisture. In general, pectin-based edible films have high sensitivity to moisture due to their high polarity[51].

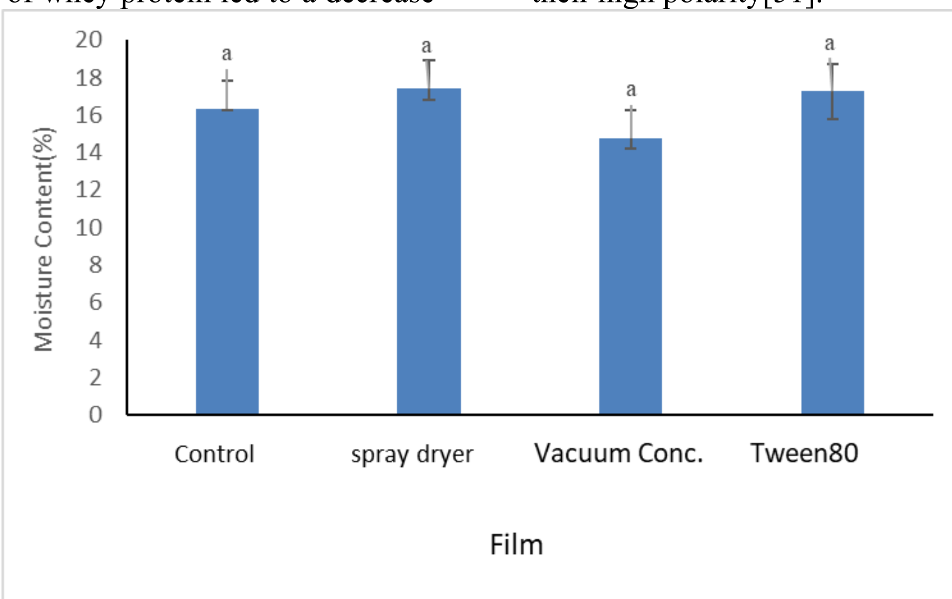

Figure 11: The moisture percentage of edible films made from pectin and whey protein containing thyme emulsion derived from zein-chitosan particles and Tween 80.

Mechanical Properties Analysis

The mechanical properties of edible films depend on the type of base material and structural integrity[52]. The results related to stress are shown in Figure 12-a. As seen in this chart, the stress level increased with the addition of thyme emulsion obtained from Tween 80 and was higher than that of other samples. ANOVA analysis of the samples showed that there was no significant difference between the control sample and the edible film containing thyme obtained from the vacuum concentrator at the level of ($p>0.05$), but significant differences ($p<0.05$) were observed among the other samples. The maximum energy levels of the edible films are presented in Figure 12-b. As shown in Figure 4-b, the addition of thyme emulsion to the edible films reduced the maximum energy levels of the samples. There was also no significant difference ($p>0.05$) observed among the samples. The ratio of tensile stress to tensile strain in cases where the relationship between these two is linear is called Young's modulus or the modulus of elasticity. As seen in Figure 12-c, with the addition of emulsion to the edible films, Young's modulus increased in all samples. Additionally, there was a significant difference at the level of ($p<0.05$) between the control sample and the samples containing the thyme emulsion obtained from Tween 80 and the nano-spray dryer. The results related to strain are shown in Figure 12-d. As the results indicate, adding thyme emulsion to the edible films caused a decrease in the strain levels of the samples. No significant difference was observed at the 5% level between the control sample and the Tween 80 sample. However, significant differences were noted at the 5% level between the control sample and the samples containing thyme emulsion obtained from the nano-spray dryer and the vacuum concentrator. A study mentioned that adding pectin reduced the mechanical properties of edible films, which was caused by the interaction between whey protein and pectin disrupting the cross-linking of the film, leading to reduced integrity between the polymer chains and ultimately resulting in a decrease in tensile strength and elongation of the layers[25]. The results related to maximum energy showed that adding thyme emulsion produced with Tween 80 increased the maximum energy, whereas adding thyme emulsion obtained from nano spray drying and vacuum concentrator reduced the maximum energy. A study observed that adding alginate reduces the tensile strength of whey protein layers and relates the outcome to strong electrostatic repulsion between alginate and whey protein at a pH of 6[53]. During one study, the effect of a combination of glycerol plasticizer and surfactants—Tween 20, Span 80, and soy lecithin—on the physical properties of potato starch-based edible films was investigated. Adding this plasticizer to the films increased their flexibility. They also noted that Tween 20 at low concentrations reduced the surface tension of the edible films, but lecithin at high concentrations caused this effect. The absence of glycerol led to surfactants having a significant impact on the mechanical properties of the films[54].

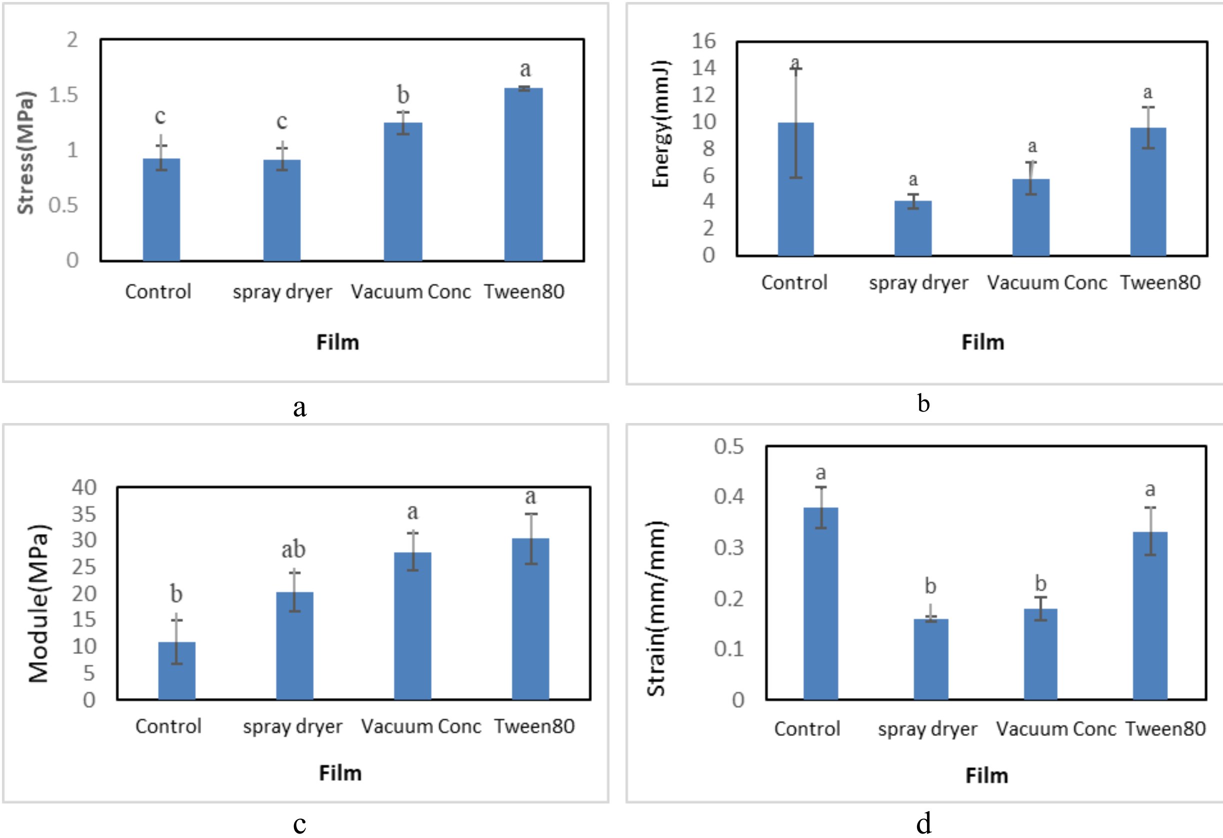

Figure 12: Figure 12: Mechanical properties of edible films made from pectin and whey protein containing thyme emulsion obtained from the vacuum concentrator along with the oven, nano spray dryer, and Tween 80 (stress (a), maximum energy (b), modulus (c), and strain (d)).

4.Conclusion

The aim of this research was to create a type of edible film based on pectin and whey protein containing stabilized emulsions of corn zein, chitosan, and Tween 80. As the results showed, the morphology of the chitosan-corn zein particles observed under a scanning electron microscope (SEM) indicated that the particles obtained from the nano spray dryer were well-separated. Additionally, the particle size measurements indicated that the largest particle size in this study corresponded to the emulsion produced using the nano spray dryer. The rheological behavior of the prepared emulsions showed that with an increase in shear rate, the shear stress also increased in all emulsion samples, which can be attributed to the chitosan present in the formulation. The results also indicated that the combined film of pectin and whey protein containing thyme emulsion had a higher contact angle and was hydrophobic in nature. The structure of the produced film and the dispersion of the particles showed that in the control sample, a uniform and homogeneous structure was not observed on the film surface. By adding the thyme emulsion to the produced edible films, the permeability to water vapor significantly decreased. Additionally, adding the thyme emulsion obtained from the vacuum concentrator and dryer increased solubility, while adding the thyme emulsion derived from Tween 80 decreased solubility. Introducing the thyme emulsion from the vacuum concentrator and nano spray dryer dramatically reduced the permeability of the edible films to light. The results of the colorimetric analysis of the samples showed that the brightness of the samples increased with the addition of thyme emulsion produced from the vacuum concentrator and nano spray dryer, but it decreased when the thyme emulsion obtained from Tween 80 was added. The results regarding the a* parameter indicated a significant difference between the dry powder and vacuum concentrator samples compared to the control sample. The results related to the b* index showed that all samples had a significant difference from each other at the level of ($p<0.05$). The results related to stress showed that adding the thyme emulsion obtained from Tween 80 increased the stress levels, making it higher than other samples. When the emulsion was added to the edible films, the Young's modulus increased in all samples. The results regarding maximum energy indicated that adding the thyme emulsion produced with Tween 80 increased the maximum energy, while adding the thyme emulsion obtained from the nano spray dryer and vacuum concentrator led to a decrease in maximum energy. The results showed that the strain decreased in all samples with the addition of the emulsion. The findings indicated that the highest moisture content was in the edible film containing the thyme emulsion obtained from the vacuum concentrator. Because in the nano spray drying method, the pore sizes were reduced due to the smaller particles, the permeability of the produced films to water vapor decreased, which could increase the shelf life of food products. Additionally, due to the reduced particle size, the produced films had high transparency. Finally, the results demonstrated that the Pickering effects improved the performance of the films.

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چکیده

در چند سال اخیر استفاده از فیلم‌های خوراکی به‌جای مواد پلاستیکی موردتوجه بسیاری از محققین قرارگرفته است. این فیلم‌ها به دلیل زیست‌تخریب‌پذیر بودنشان کمک بسیار زیادی به محیط‌زیست کرده‌اند. در این پژوهش فیلم خوراکی بر پایه پکتین و پروتئین آب‌پنیر حاوی امولسیون اسانس آویشن که با سه روش تولید (تغلیظ کننده تحت خلأ همراه با آون، خشک‌کن پاششی نانو و توئین ۸۰) شده مورد بررسی قرار گرفت. یافته‌های حاصل از بررسی ساختار فیلم و میزان پراکندگی ذرات در سطح فیلم‌ها نشان داد که با اضافه نمودن امولسیون آویشن حاصله از توئین ۸۰ میزان زبری و خشنی نمونه فیلم کاهش داد. نتایج حاکی از آن بود که با افزودن امولسیون آویشن به فیلم‌های خوراکی که بر پایه پکتین و پروتئین آب‌پنیر، میزان نفوذپذیری به بخارآب به‌طور چشمگیری کاهش یافت. همچنین نتایج نشان داد با اضافه نمودن امولسیون آویشن حاصله از تغلیظ کننده تحت خلأ و خشک‌کن میزان انحلال‌پذیری نیز افزایش ولی با افزودن امولسیون آویشن حاصله از توئین ۸۰ میزان انحلال‌پذیری کاهش یافت. اضافه نمودن امولسیون آویشن حاصله از دستگاه تغلیظ کننده تحت خلأ و خشک‌کن پاششی نانو سبب کاهش میزان نفوذپذیری فیلم‌ها به نور شد. نتایج مربوط به رنگ‌سنجی نمونه‌ها نشان داد که میزان روشنایی با افزودن امولسیون آویشن تولیدشده از دستگاه تغلیظ کننده تحت خلأ و خشک‌کن پاششی نانو، افزایش یافت اما با افزودن امولسیون آویشن به‌دست‌آمده از توئین ۸۰ میزان روشنایی را کاهش داد. نتایج مربوط به پارامتر a^* از تفاوت معنی‌داری بین نمونه‌های پودر خشک‌کن و تغلیظ کننده تحت خلأ با نمونه شاهد حاکی بود ولی میان نمونه توئین ۸۰ با نمونه شاهد هیچ‌گونه اختلاف معنی‌داری مشاهده نگردید. نتایج شاخص b^* نشان داد که تمامی نمونه‌ها با یکدیگر اختلاف معنی‌داری در سطح ۵ درصد داشتند.