coatings

MDPI

Article

Extraction and Characterization of Pectin from Jerusalem ArtiChoke Residue and Its Application in Blueberry Preservation

Xiaotong Zhou [1], Liu Liu [1,2,*], Jianke Li [1,2,*], Lin Wang [1] and Xueying Song [1]

[1] College of Food Engineering and Nutritional Science, Shaanxi Normal University, Xi'an 710119, China; auty_zhou@126.com (X.Z.); wangliner417@163.com (L.W.); sxy18734445184@163.com (X.S.)
[2] University Key Laboratory of Food Processing Byproducts for Advanced Development and High Value Utilization, Xi'an 710119, China
* Correspondence: liuliu@snnu.edu.cn (L.L.); jiankel@snnu.edu.cn (J.L.); Tel./Fax: +86-029-8531-0517 (L.L.)

Abstract: To prolong the storage period of blueberry and improve its shelf-life quality, an edible coating based on chitosan was developed, and different contents of pectin were added to the coating. In this study, Jerusalem artichoke residue was used as a source of pectin, which is a byproduct of the processing of inulin. The extracted pectin has a low cost and high quality, which is very suitable for the preservation of coating. The coating was prepared and the chemical properties of the coating were characterized by SEM, XRD, TG, and FTIR. The barrier properties of the coating were analyzed by thickness, water content, solubility, and water vapor permeability. The results showed that the pectin coating exhibited excellent performance in blueberry preservation. Following 16 days of storage, the decay and weight loss rates of blueberry treated with 0.2% pectin coating decreased by 33 and 22%, respectively. Moreover, the organic acid consumption of the coated blueberry slowed and the anthocyanins were better preserved. As a low-cost, safe, and efficient technology, the pectin chitosan composite coating has significant potential in the berries preservation industry.

Keywords: Jerusalem artichoke; pectin; coating; blueberry preservation

Citation: Zhou, X.; Liu, L.; Li, J.; Wang, L.; Song, X. Extraction and Characterization of Pectin from Jerusalem ArtiChoke Residue and Its Application in Blueberry Preservation. *Coatings* **2022**, *12*, 385. https://doi.org/10.3390/coatings12030385

Academic Editor: Elena Poverenov

Received: 30 January 2022
Accepted: 7 March 2022
Published: 15 March 2022

1. Introduction

Jerusalem artichoke (*Helianthus tuberosus* L.) is a perennial herb of Asteraceae sunflower, which is widely planted in northern China. It contains 12–20% of inulin and certain amounts of pectin, protein, cellulose, etc. [1]. At present, Jerusalem artichoke is mainly used to produce inulin, and a large amount of Jerusalem artichoke residue is produced after the extraction of inulin. In general, Jerusalem artichoke residue is discarded directly as waste or used as feed after simple processing. Moreover, it contains about 15% of pectin on a dry basis and the use of this part is necessary. Currently, the comprehensive utilization rate of Jerusalem artichoke residue is very low, and there are few scientific studies on Jerusalem artichoke residue. A study by Tingshuo Zhu et al. [2] evaluated the effects of Jerusalem artichoke residue on the improvement of different salinization soils and microbial communities.

Pectin is mostly a light yellow colloidal solid that can be dissolved in water to form a viscous solution, and is insoluble in organic solvents, such as ethanol [3]. It is a form of natural macromolecular compound, which is mainly distributed in the fruits of plants. In addition, pectin is one of the most complex polysaccharides due to its constantly changing structure during plant growth. In terms of structure, pectin is composed of a main chain, which is polymerized by 1,4-α-galacturonic acid, and a branched chain, which contains a large amount of neutral sugar. In general, it is used as a thickener, emulsifier, and stabilizer in food formulations. As a soluble dietary fiber, pectin has an effect on the regulation of intestinal function. The majority of commercial pectin is extracted from fruits, such as apples. Extracting pectin from Jerusalem artichoke residue is a novel idea, which can

improve not only the utilization rate of Jerusalem artichoke, but also lead to the discovery of new raw materials for the production of pectin.

Blueberry (*Vaccinium* ssp.) is a nutritious fruit rich in anthocyanins, vitamins, and other phenolic compounds [4]. In addition, it has become widely popular among consumers due to its unique taste and high nutritional value. Blueberry is universally acknowledged as a kind of wild berry that decays rapidly after picking, which is manifested by softening, weight loss, color change, mildew, etc. Blueberry can be contaminated at any point in the chain of production and marketing. In addition, it is highly perishable. On the one hand, it is juicy and has thin skin. On the other hand, it is easily affected by water loss and mechanical damage, resulting in the decline of its quality. The accurate control of storage temperature, humidity, and atmosphere is an effective measure to prevent the postharvest corruption of blueberry. However, the cost of these methods is high. Coating preservation is a new strategy for berries, such as blueberries, which aims to extend the shelf life of blueberry to 2 weeks with the use of film-coated pectin. The edible film can be used to extend the storage life of fresh blueberry in the food industry.

The technology of coating is a new type of fresh-keeping technology that has grown rapidly in recent years. Wax-based coatings have been widely used in fresh fruits and vegetables preservation. However, surveys show that many consumers have concerns regarding the safety of wax coating. Therefore, researchers began to develop more safe and effective edible coating materials, such as starch, chitosan, pectin, cellulose, and other natural polymer materials. Carolina Medina-Jaramillo et al. [5] studied the effect of carvacrol/alginate-edible coating on the postharvest preservation of Andean blueberry. The authors found that the plastic film coating on the surface of blueberry resulted in better preservation. Moreover, it significantly reduced the respiration rate and water loss of blueberry throughout the 21 days of low-temperature storage. Pectin is a good film-forming material. In this study, pectin extracted from Jerusalem artichoke residue was added to the edible preservation coating of blueberry as a thickener.

2. Materials and Methods

2.1. Materials and Reagents

Jerusalem artichoke residue was supplied by Shaanxi Sciphar Natural Products Co., Ltd (Xi'an, China). Chitosan (90% deacetylated) was purchased from Aladdin Chemical Co., Ltd. (Shanghai, China) All of the chemical reagents used were of analytical grade.

2.2. Pectin Extraction

The extraction of pectin was performed with the addition of 100 mL of distilled water in a 250 mL beaker, followed by adjusting the pH to 2 with phosphoric acid. A total of 5 g Jerusalem artichoke residue on a dry basis was added to the extract, then heated at 80 °C for 1 h. Thereafter, the mixture was filtered, and the filtrate was collected. The extraction was repeated three times, and the filtrate was merged and concentrated at 80 °C. After the concentrate was cooled to room temperature, four volumes of anhydrous ethanol were added, and left to stand at room temperature for 4 h. Finally, pectin was obtained by centrifugation and precipitation oven drying [6].

2.3. Pectin Characterization

The functional groups contained in pectin were modified according to the method of Yu-Jie Meng et al. [7].

The thermal stability of pectin was measured using a thermo-analyzer system (Q1000DSC + LNCS + FACS Q600SDT, TA Instruments, New Castle, DE, USA). Herein, the temperature was in the range of 50–600 °C, the heating rate was 5 °C/min, and the nitrogen flow rate was 60 mL/min.

The chemical bonds in pectin were tested by Nicolet iS10 spectrometer (Thermo Fisher Scientific Inc., Waltham, MA, USA). An appropriate amount of potassium bromide was

mixed with pectin powder and grinded to develop tablets. The wavelength was in the range of 400–4000 cm^{-1}.

The crystal structure of pectin was tested by X-ray diffractometer (D8 Advance, Brock, Germany). Herein, the scanning range was from 5–50°, and the scanning rate was 5°/min.

Pectin powder was adhered on a metal support with a double-sided conductive adhesive tape and coated with gold. ESEM (FEI Quanta 200, Hillsboro, OR, USA) was used to observe the microstructure of pectin.

2.4. Preparation of Edible Pectin Chitosan Films

The edible pectin chitosan composite film was prepared according to the method of Ruchir Priyadarshi et al. [8]. Five concentrations of pectin (0, 0.1, 0.2, 0.3, and 0.4 g) were added into 100 mL of water, and stirred with a magnetic stirrer until they were completely dissolved. Then, 1 mL of acetic acid and 2 g of chitosan were added, and stirred until the solution was transparent and clarified. Thereafter, 0.6 mL of glycerol was added, and the film-forming solution at five concentrations was obtained after continuous stirring. The liquid was degassed for 30 min by an ultrasonic cleaner. The edible film was prepared by pouring 15 mL of film-forming solution into a circular container with a diameter of 90 mm. After drying, the edible film was removed, and the prepared film was placed in a vacuum dryer for further use [9].

2.5. Pectin Chitosan Film Characterization

2.5.1. Measurement of Film Thickness, Moisture Content, Water Solubility, and Water Vapor Permeability (WVP)

The film thickness was measured by cross-sectional electron microscope images. Each film was averaged at three locations. The test methods for moisture content, water solubility, and water vapor permeability of films were conducted according to the methods of Jitrawadee Meerasri et al. [10] and Sajad Pirsa [11]. The initial weight of the film was weighed and dried in a 105 °C oven until it reached constant weight. Then, the dried film was immersed in distilled water for 24 h, and dried again to constant weight. Finally, the water vapor permeability of the film was determined by gravimetric analysis.

2.5.2. Fourier Transform Infrared Spectroscopy (FTIR)

The film was adhered to a metal support and tested by Nicolet iS10 spectrometer (Thermo Fisher Scientific Inc., Waltham, MA, USA). The wavelength was in the range 400–4000 cm^{-1}.

2.5.3. Thermal Analysis

The thermal stability of five films was measured using the thermo-analyzer system (Q1000DSC + LNCS + FACS Q600SDT, TA Instruments, New Castle, DE, USA). Herein, the heating rate was 5 °C/min, the nitrogen flow rate was 60 mL/min, and the scanning range was from 50–600 °C.

2.5.4. X-ray Diffraction (XRD)

The film was cut, adhered on a loading platform, and analyzed by X-ray diffractometer (D8 Advance, Brock, Germany). Herein, the scanning rate was 5°/min, and the scanning range was from 5–50°.

2.5.5. Environmental Scanning Electron Microscope

The film was cut to an appropriate size, adhered on a loading platform with a double-sided conductive adhesive tape, and coated with gold. SEM (FEI Quanta 200, Hillsboro, OR, USA) was used to observe the surface and cross-section of the films.

2.6. Blueberry Preservation

2.6.1. Coating Treatment of Blueberry

Blueberries were randomly divided into six groups. One group was the control group, and five groups were immersed in five concentrations of film-forming solution for 5 s. After the blueberry surface was evenly wrapped with a film-forming solution, it was removed and dried. All of the six groups were stored for 16 days, and the relevant indicators were tested every 4 days [12].

2.6.2. Weight Loss and Decay Rates of Blueberry

The weight loss rate of blueberries was measured according to the method of Carolina Medina-Jaramillo [5]. The initial weight of the blueberry was recorded, and then measured every 4 days. The decay rate was slightly modified by the method of Siyao Wang et al. [13]. According to the degree of fruit decay and softening, there were four grades: Level 0 (surface no clear change), level 1 (clear softening phenomenon), level 2 (serious softening, juice leakage), and level 3 (mold spot infection on the surface). The number of each grade was recorded every 4 days.

2.6.3. Determination of pH, Titratable Acid, and Anthocyanin Content in Blueberry

To determine the pH value of blueberry juice, each group of blueberries was squeezed, then tested and recorded using the pH meter. Briefly, 5 g of blueberry juice was transferred into a 50 mL volume bottle in constant volume, diluted on a scale with carbon dioxide-free water, and filtered. Thereafter, 25 mL of filtrate was transferred into a 150 mL beaker and titrate with 0.1 mol/L NaOH standard titrant to pH 8.2. The titratable acid content in blueberry juice was determined using the pH differential method. The anthocyanin content in blueberry juice was calculated according to the method of Jungmin Lee [14].

2.6.4. Hardness of Blueberry

To assess blueberry texture characteristics, such as hardness, a P2 probe was carried out every 4 days using a texture analyzer (Stable Micro Systems TA-XT PLUS, USA). The testing parameters were as follows: Displacement of 5 mm and force of 10.0 g. In addition, the test was repeated six times in each group.

2.7. Statistical Analysis

Statistical analysis was performed using Microsoft Excel 2011, SPSS Statistics 26.0 (IBM, Armonk, NY, USA). RStudio 1.3 was used to analyze and map the experimental data. The confidence level is 95% and $p < 0.05$ was considered statistically significant. The experimental data shown in all of the results were repeated at least three times.

3. Results and Discussion

3.1. Pectin Extraction and Characterization

The pectin extracted from Jerusalem artichoke residue is a light yellow colloidal solid, and the extraction rate is about 10%. Figure 1A shows the results of Fourier transform infrared spectrum of pectin. The wide signal at 3400 cm^{-1} represents the O–H stretching. The signal peak at 2923 cm^{-1} represents the C–H stretching, such as –CH, $–CH_2$, and $–CH_3$. The signal peaks at 1634 and 1735 cm^{-1} represent the C–O stretching vibration of free carboxyl and methyl ester groups. The peak at 1000–1250 cm^{-1} represents the glycosidic bond (C–O–C). These results are almost consistent with the studies of Zahra Rahmani et al. [15] and Shiva Ezzati et al. [16]. The XRD patterns of pectin are shown in Figure 1B. It can be seen that no sharp peaks were present in the spectrum. However, a wide range of background patterns indicate that the pectin extracted from Jerusalem artichoke was an amorphous product. In this study, the extracted pectin has a very similar amorphous structure to the commercial pectin reported by Saeid Hosseini et al. The results showed that there was no significant difference in the crystalline state between the pectin extracted from Jerusalem artichoke residue and commercial pectin [17].

The TGA analysis of pectin demonstrates three main stages: 50–200 °C, 200–400 °C, and 400–600 °C, as shown in Figure 1C. In the first stage, 40% of mass loss occurred, which may be due to the evaporation of free and bound water in pectin. In the second stage, less than 16% of mass loss occurred, which may be attributed to the thermal decomposition of pectin. A small amount of mass loss occurred in the third stage, which may be related to the decomposition of carbon. The final residual weight of pectin was 39.63%, which was about 30% higher than the general commercial pectin. Notably, it has been proven that the pectin extracted from Jerusalem artichoke residue has better thermal stability and is suitable for manufacturing edible films.

The surface morphology of pectin was observed by environmental scanning electron microscope at different magnifications, as shown in Figure 1D,E. It can be seen that the pectin extracted in this study had a certain roughness without cracks and openings. This fruit was similar to the surface morphology of pectin extracted from apple and orange peels in the studies of Melih Güzel and Özlem Akpänar [18].

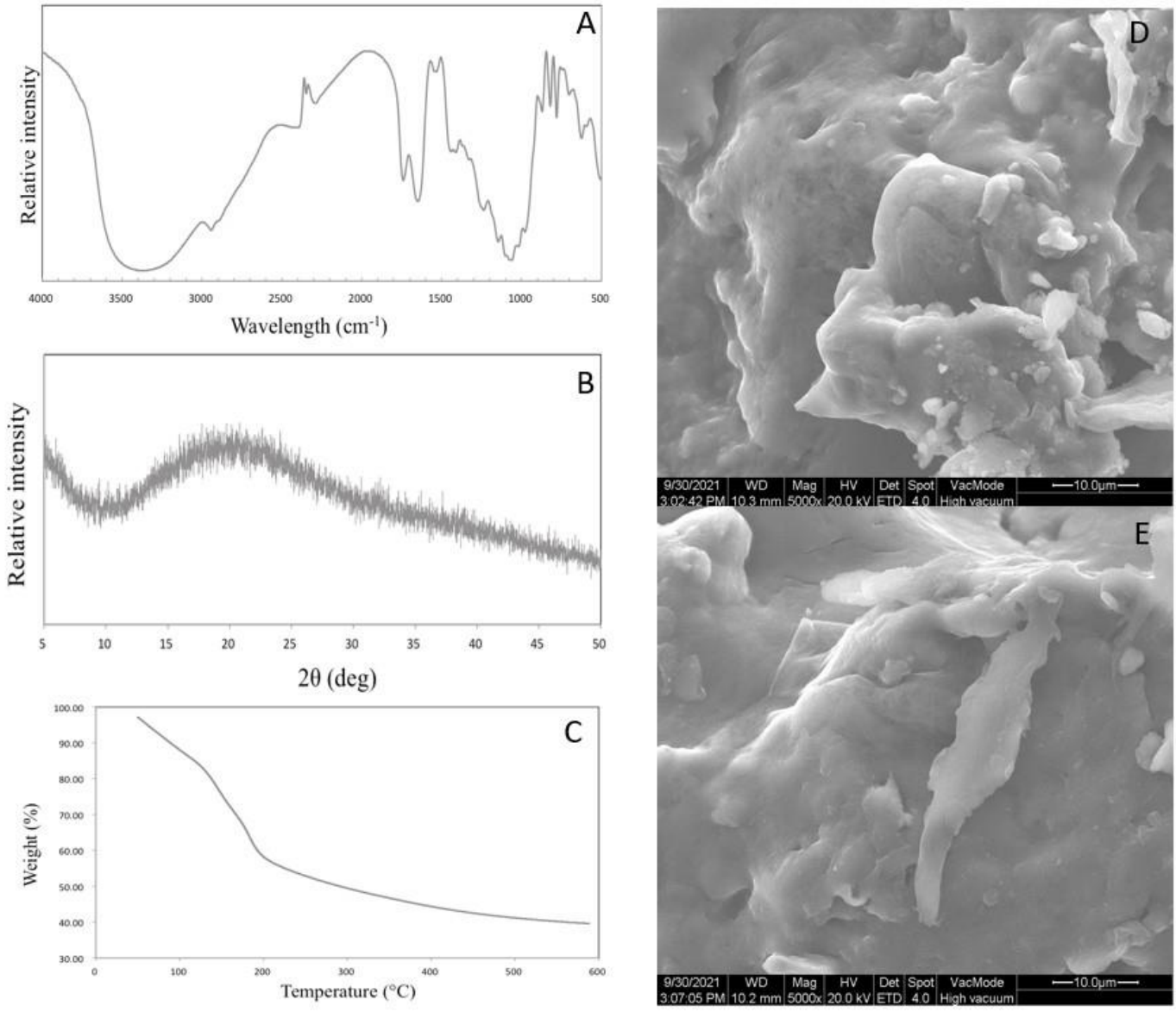


Figure 1. (**A**) FTIR spectra; (**B**) XRD patterns; (**C**) TGA analysis; (**D**,**E**) SEM of pectin.

3.2. Characterization of Pectin Composite Membrane Films

3.2.1. Surface Morphology of the Films

Microstructures of the films are shown in Figure 2A. It can be seen that the edible pectin chitosan composite films are colorless, transparent, smooth, and not granular. The environmental scanning electron microscope images of the films' surface are shown in Figure 2B. At the magnification of 3000 times, it can also be seen that the films' surface with different pectin concentrations is smooth and seamless. This indicated that the increase of pectin content did not affect the surface characteristics of film. The cross-sectional SEM images of film are shown in Figure 2C. In general, the cross-section of film is smooth and the thickness is uniform. Compared with the chitosan film, the cross-section of film containing pectin is significantly rough. Overall, the cross-section of each film is closely continuous. It has been reported that pectin is a typical anionic linear polysaccharide, while chitosan is a cationic linear polysaccharide. Therefore, the addition of pectin helps in the formation of a complex network structure and improves the physical properties of film [19].

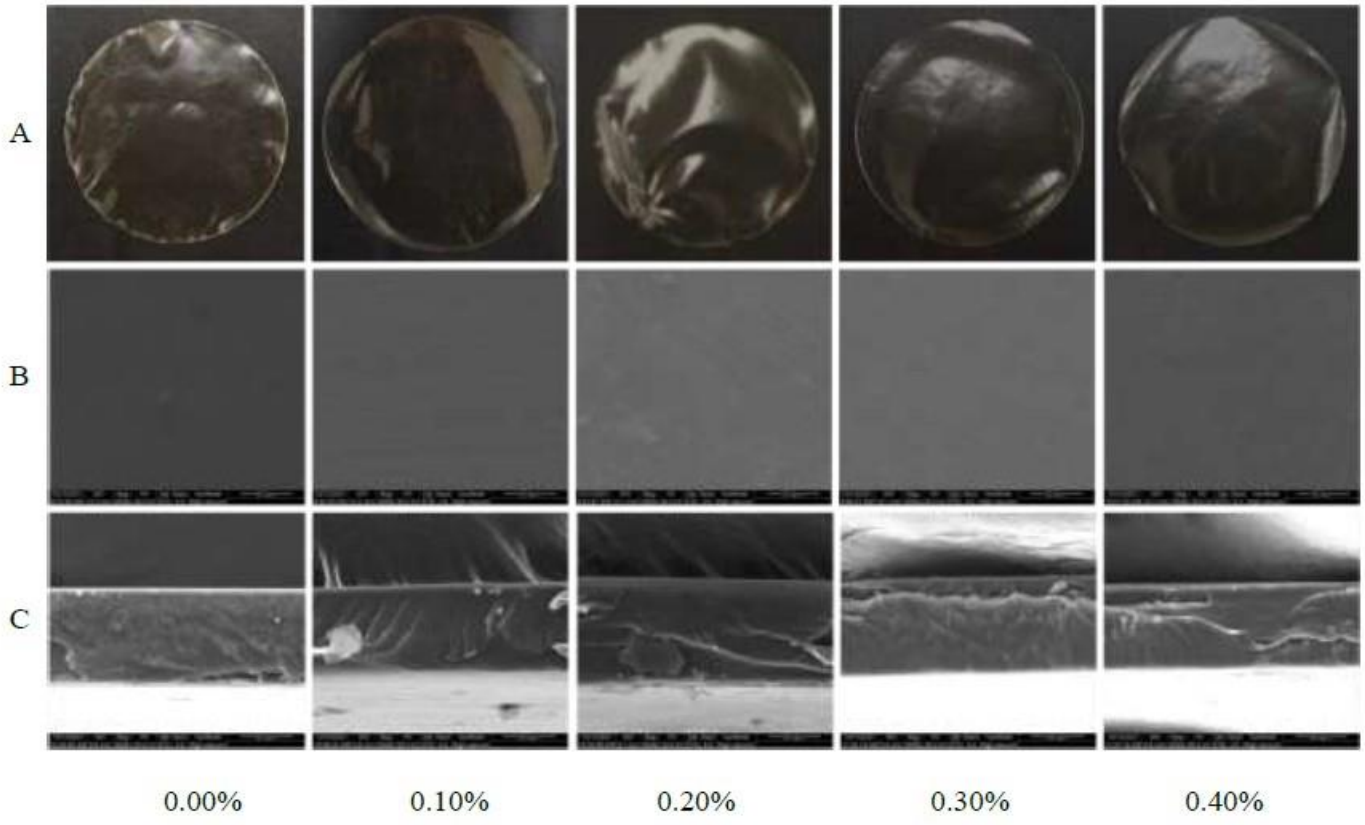
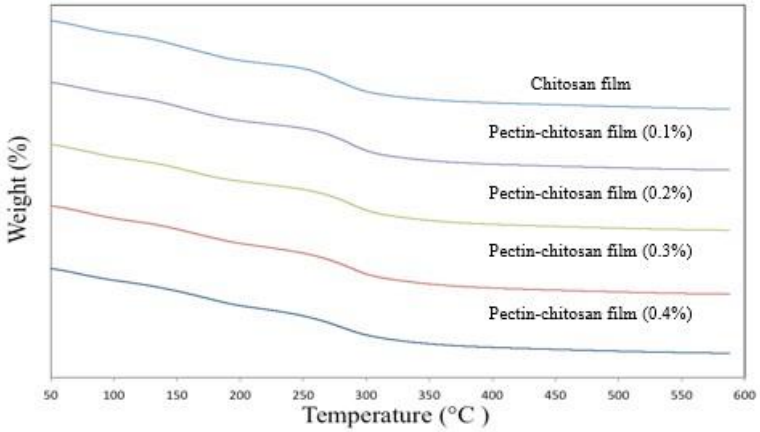


Figure 2. (**A**) Micrographs of surface (**B**) and micrographs of cross-section (**C**) of films.

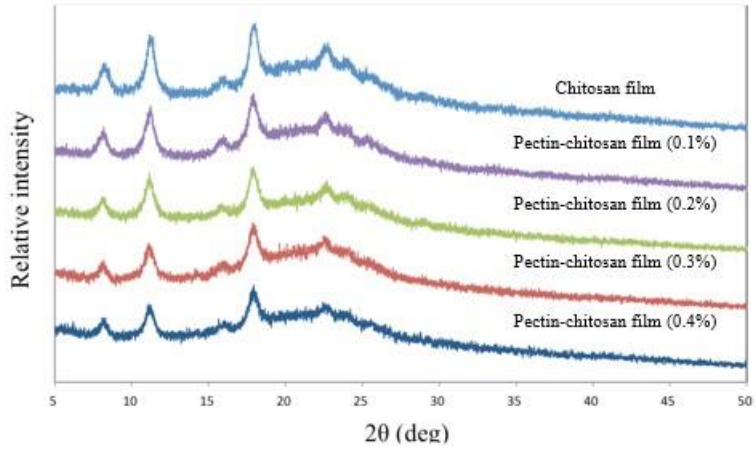
3.2.2. Thermal Gravimetric Analysis of Films

Thermal gravimetric analysis is an effective tool to measure the thermal properties of materials. The TGA results of all films are shown in Figure 3A. Similar to the TGA curve of pectin, the pyrolysis process of the films was divided into three stages. The first stage is 50–200 °C, the second stage is 200–400 °C, and the third stage is 400–600 °C. In the first stage, the mass loss was mainly caused by the loss of free and bound water and the evaporation of glycerol. The weight loss in this stage is the largest in the three stages. With the increase of pectin content from 0 to 0.4%, the mass loss of film in the first stage was 34.66, 33.09, 31.87, 31.91, and 32.04%, respectively. The addition of pectin reduced the heat loss of film in the first stage. In the second stage, the heat loss is attributed to the decomposition of polymer and the fracture of chain. Derong Lin et al. believed that glycosidic bonds in polysaccharides were pyrolyzed into a series of fatty acids, such as

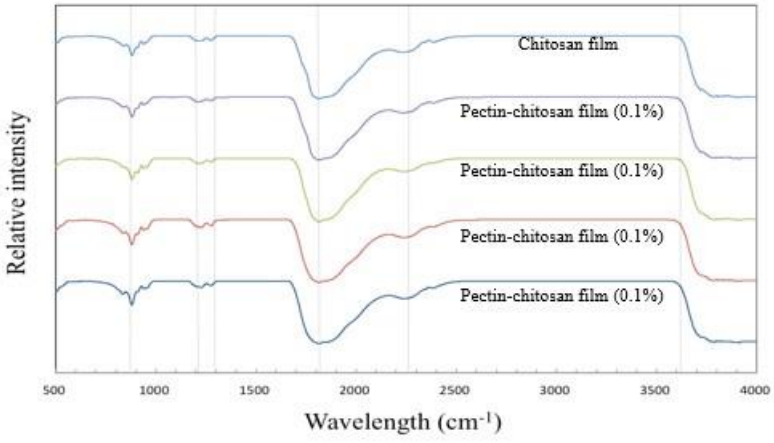
acetic and butyric acid. In the third stage, the loss of sample quality may be related to the decomposition of carbon. Only a small amount of mass loss occurred in each sample at this stage. With the increase of pectin content, the total heat loss of samples was 73.75, 72.78, 71.4, 72.79, and 70.39%. The enhanced thermal stability of film after adding pectin may be related to the enhanced connection between pectin and chitosan molecules [20,21].



(**A**)



(**B**)



(**C**)

Figure 3. TGA analysis (**A**), XRD patterns (**B**), and FTIR spectra (**C**) of films.

3.2.3. XRD Analysis

Figure 3B shows the results of XRD analysis of films. The spectrum shows a small amount of sharp peaks, which proves that the films show semi-crystalline characteristics. The diffraction peaks at 7.95 and 17.78° may be related to the anhydrous structure. The wide peak at 22.20° was the characteristic diffraction peak of chitosan, showing an amorphous structure. With the increase of pectin content, this characteristic peak weakened, which may be due to the interaction between pectin and chitosan that reduces the crystallinity of chitosan [22]. In addition, Juan Kan et al. proposed that the presence of glycerol enhanced the mobility of polymers in edible films and reduced the cohesion within the matrix, which was not conducive to the arrangement of long chains [23].

3.2.4. FTIR Analysis

Fourier transform infrared spectroscopy can be used to analyze the interaction among components in the films, and the results of FTIR spectrum are shown in Figure 3C. First, the peak shape of the pectin–chitosan composite films was typically the same as the control group, which proved that the addition of pectin did not change the chemical structure of film. The absorption peak at 1172 cm^{-1} corresponds to the C–O–C stretching. The broad absorption peak at 1760 cm^{-1} represents the vibration of carboxyl (–COOH). The two characteristic peaks at 802 and 914 cm^{-1} correspond to α-1,6-glycosidic and α-1,4-glycosidic bonds, respectively, which are the main chemical bonds of monosaccharide linkage in pectin and chitosan. The absorption peak at 869 cm^{-1} is the stretching vibration of fingerprint region corresponding to the hydrogen bond (O–H). In addition, the absorption peak at 3681 cm^{-1} appears in the characteristic region, which confirms the existence of hydrogen bond. The signal peaks at 2214 cm^{-1} correspond to the C–H stretching, such as C–H, $C–H_2$, and $C–H_3$. Most of the above chemical bonds are derived from polysaccharide macromolecules, such as pectin and chitosan [24].

3.2.5. Film Thickness

Thickness is an important index to measure the performance of packaging materials, since it is related to the transmission characteristics of materials. The film thickness was calculated from the cross-sectional electron microscope images. Three measurements were randomly selected for each film and the average was taken into account. The results are shown in Figure 4A. The thickness of all films is very thin, reaching the micron level. The thickness of chitosan film is 37.80 μm, which is the smallest of all films. The film with pectin content of 0.2% is the thickest, which is 42.00 μm. With the increase of pectin content, the film thickness increased first, and then decreased. This may be due to the initial addition of pectin, which increased the concentration of film-forming liquid. Therefore, the film formed by drying the same volume of film-forming liquid is thicker. Studies have reported that pectin and chitosan have a strong bio-adhesive capacity. Therefore, with the further increase of pectin content, a closer network structure was formed between pectin and chitosan, in which the thickness of formed film decreased and the strength increased [21,23].

3.2.6. Moisture Content, Water Solubility, and Water Vapor Permeability (WVP) of Films

The moisture content of all films was low (about 30%), as shown in Figure 4B. The highest moisture content was 31.89% in the control group. With the increase of pectin content, the water content of film decreased slightly, and then increased. The film with 0.2% pectin has the lowest moisture content of 28.21%. The XRD and FTIR analyses of the film reported that pectin and chitosan form a close connection, reducing the leakage of hydrophilic groups in the molecule. Due to the competing effect between pectin and water molecules, the binding of chitosan to water is limited. This may be the reason for the lower moisture contents of edible films that are added with pectin. Water is an important condition for the growth and reproduction of microorganisms. As a barrier between food and air, the low water content of edible film is beneficial to prevent microbial contamination. In addition, water loss is an important factor leading to the decline of food

quality, especially fresh fruit quality. Therefore, the preservative film needs to maintain a certain moisture content.

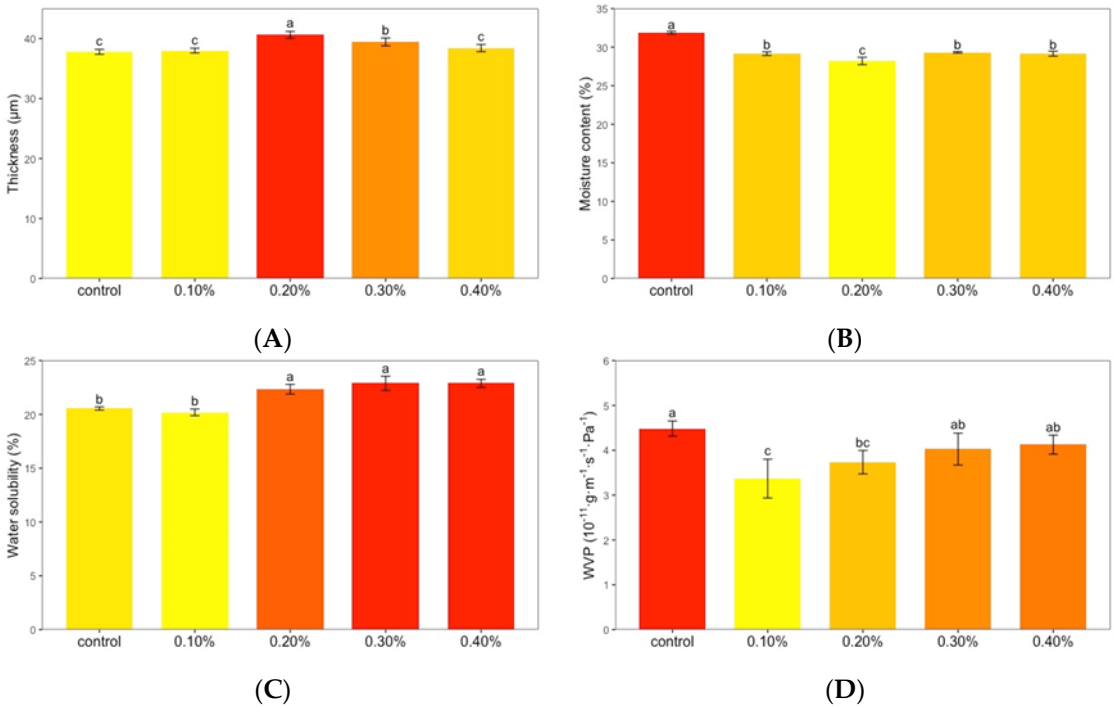


Figure 4. Thickness (**A**), moisture content (**B**), water solubility (**C**), WVP (**D**) of films. Control represents the chitosan film without pectin.

Figure 4C shows the results of water solubility of the films, and the water solubility of all films is between 20 and 23%. The water solubility of control film was the lowest. Following the addition of pectin, the water solubility of film increased slightly. The solubility of film was enhanced due to the addition of pectin [25].

To prevent or delay food spoilage, food and environment separation is one of the main functions of preservative film. WVP is an important indicator to measure the water vapor permeability of film. In general, due to the low water vapor permeability of the preservative film, the external water does not enter the food easily, resulting in food corruption and deterioration. At the same time, the water in the food is not easy to lose, resulting in the decline of food dehydration quality [26]. Figure 4D shows the water vapor permeability of five films. The highest water vapor permeability of chitosan film was 4.48×10^{-11} g·m^{-1}·s^{-1}·Pa^{-1}. The water vapor permeability of the film with pectin decreased significantly. With the increase of pectin content, the water vapor permeability of film increased slightly. The water vapor permeability of 0.1% pectin film is very low, at only 3.37×10^{-11} g·m^{-1}·s^{-1}·Pa^{-1}. It has been proven that the pectin composite film is suitable for food, especially fruit preservation [27,28].

3.3. Coating Storage of Blueberry

3.3.1. Weight Loss and Decay Rates of Blueberry

On the one hand, the loss of commerciality of blueberry is caused by fungal infections, fruit physiological changes, and even decay. On the other hand, it is due to postharvest water loss and nutrient consumption, which is caused by fruit weight loss. Studies have shown that chitosan coating can effectively reduce the quality of blueberry during storage. Figure 5 shows the changes of weight loss and corruption rates of blueberry in five groups of coating treatment and one control group during storage for 16 days. The weight loss rate of control group was significantly higher than the other groups. Following 16 days of storage, the weight loss rate of blueberry in the control group reached 31.55%. It proved that

the coating treatment could reduce the weight loss of blueberry during storage. Compared with the control group, the weight loss rate of blueberry coated with 0.2% pectin decreased by 22.00%. The above-mentioned analysis showed that the water vapor permeability of pectin–chitosan composite films was lower than the chitosan film. After the blueberry was coated with pectin, the moisture was not easy to lose, which reduced the weight loss of blueberry during storage [29].

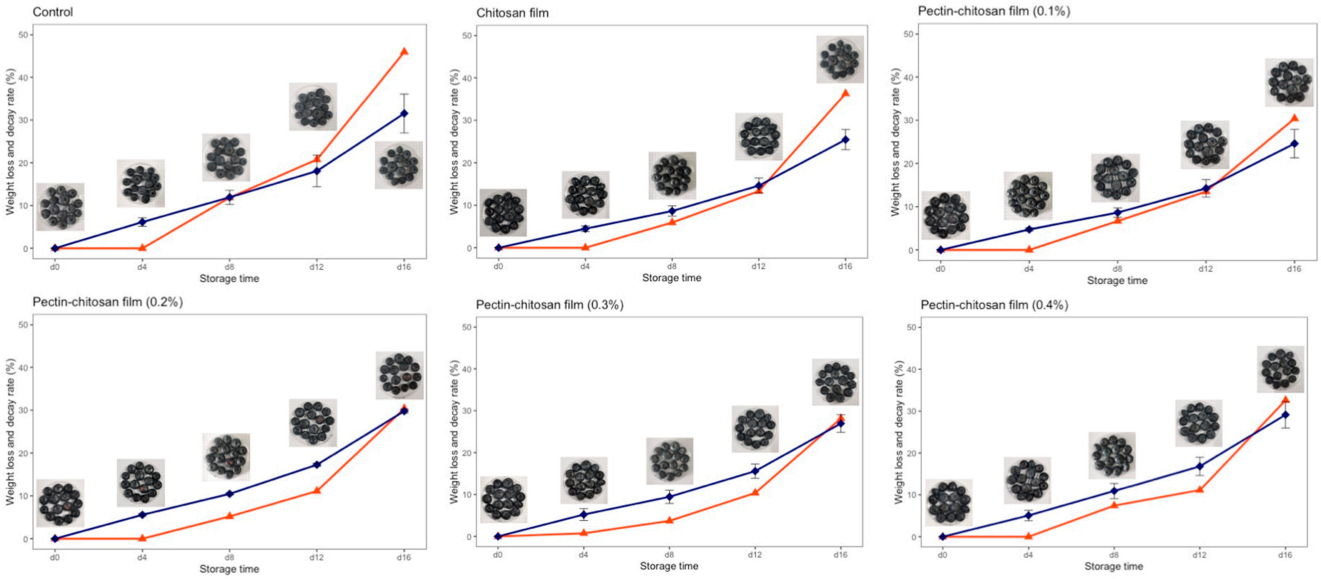


Figure 5. Weight loss and decay rates of blueberry under different treatments. Control represents the untreated blueberries.

At the beginning of storage, blueberries were full and glossy. At the end of storage, the blueberries in each group showed varying degrees of gloss darkening, softening, and collapse. The control group appeared as more soft after 4 days of storage, mold infection appeared on the 8th day of storage, and the decay rate reached 45.53% at the end of storage. The coating with 0.3% pectin had the best anticorrosive ability, and the decay rate was only 3.7% on the 8th day of storage and 28.15% on the 16th day of storage. Compared with the control group, the decay rate of 0.3% pectin coating group decreased by 38.71%. After the coating treatment, the decrease of blueberry decay rate may be related to the antibacterial properties of pectin. Rosaria Ciriminna et al. [30] considered that pectin was a long-neglected spectral fungicide and had bactericidal activity against the most widely distributed pathogenic and opportunistic microorganisms.

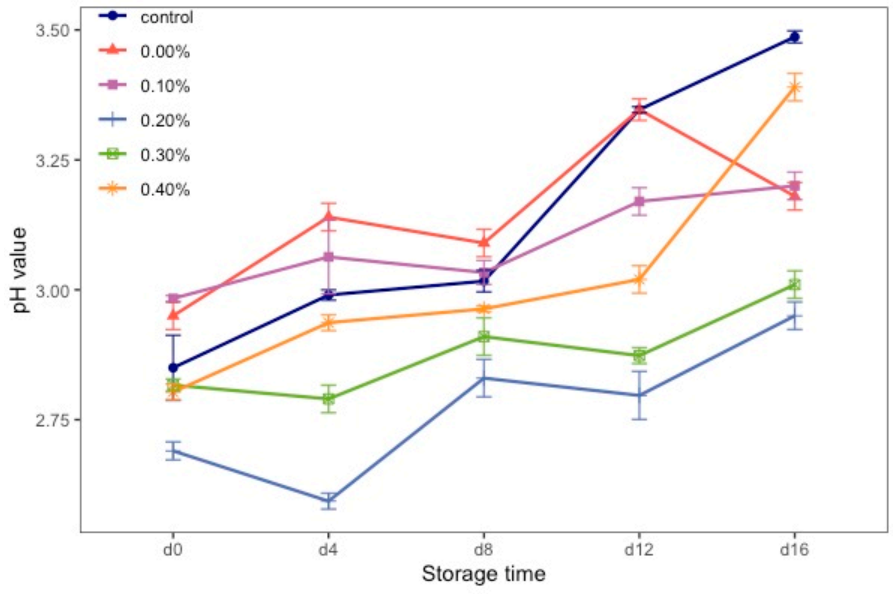
3.3.2. Changes of pH, Titratable Acid, and Anthocyanin Contents in Blueberry during Storage

The changes of pH and titratable acid of blueberry under different treatments during storage are shown in Figure 6A,B. In general, due to the continuous consumption of organic acids during storage, the pH value of blueberry showed an upward trend, and the titratable acid showed a downward trend. The test results are typically consistent with the expectations. The pH values of fresh blueberry were all lower than 3.0. Following 16 days of storage, the control group with the highest pH value increased to 3.49. The pH value of blueberry treated with coating was lower than the control group. In particular, the pH value of coating with 0.2% pectin group was lower than 3.0 during the whole storage period, and the pH growth rate was the lowest. The titratable acid content of blueberry in each group increased first, and then decreased during storage, which was lightly different from the expectations. The reason for this phenomenon is that in the early stage of storage, the water loss of fruits prevailed, and the concentration of organic acids in blueberries increased, resulting in the increase of titratable acid content. In the last stage of storage, organic acid consumption prevailed, and the content of titratable acid decreased gradually. The

inflection point of blueberry without the coating treatment was the earliest and appeared on the 4th day. The inflection point of blueberry treated with chitosan coating with 0.2 and 0.4% pectin appeared on the 8th day. The inflection point of coating with 0.1 and 0.3% pectin was delayed to the 12th day of storage. At the end of storage, the highest titratable acid content was the coating with 0.2% pectin, which was 53.70 mg/100 g [31,32].

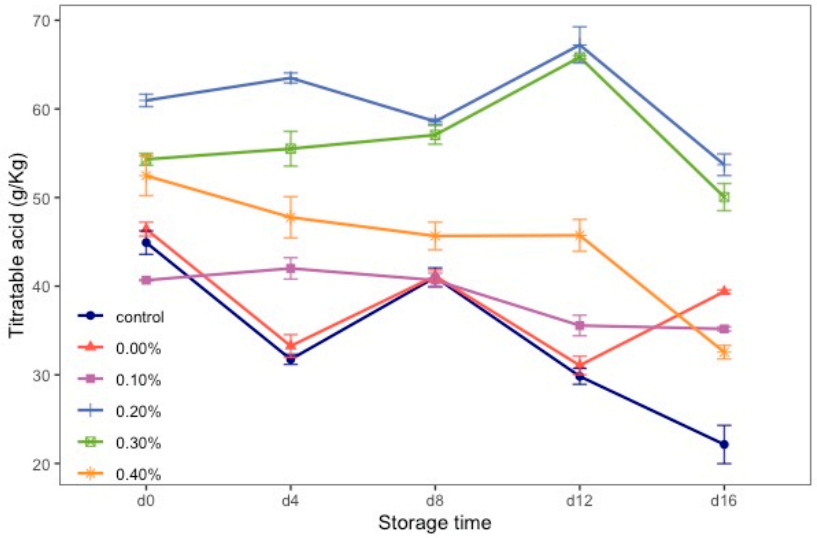
Anthocyanins are secondary metabolites and important nutrients in blueberry. There are 15 kinds of anthocyanin in blueberry. Anthocyanins have strong antioxidant capacity and high nutritional value. Blueberry is also widely popular among consumers since it is rich in anthocyanin. The changes of anthocyanin content can indicate the quality of blueberry during storage. Figure 6C shows the changes in anthocyanin content of blueberry in each group during storage. The anthocyanin content of blueberry in each group showed a downward trend. Phenolic compounds can synthesize anthocyanin and accumulate in blueberry. In the early stage of storage, anthocyanin synthesis is fast, while the consumption is low. For example, three groups of blueberry with 0.1, 0.2, and 0.3% pectin coating showed a slight increase at the beginning of storage. At the last stage, the consumption of anthocyanin accelerated and various nutrients were depleted [33].

During storage, organic acids in blueberry were consumed, the antibacterial ability was weakened, and corruption was accelerated. Anthocyanins are easily oxidized and consumed. However, in acidic environments, anthocyanins are not easily decomposed and can be well preserved. The coating treatment of blueberry blocked the contact between blueberry and air, and slowed down the consumption of anthocyanin and other nutrients in blueberry. At the same time, the previous test proved that the coating treatment could effectively reduce the consumption of organic acids, maintain the acidic environment inside blueberry, and facilitate the preservation of anthocyanin. Horizontally, the coating containing 0.2% pectin showed a very good performance in blueberry preservation. During storage, the acidity and anthocyanin contents of blueberry treated with 0.2% pectin coating were the highest in the six groups [33].

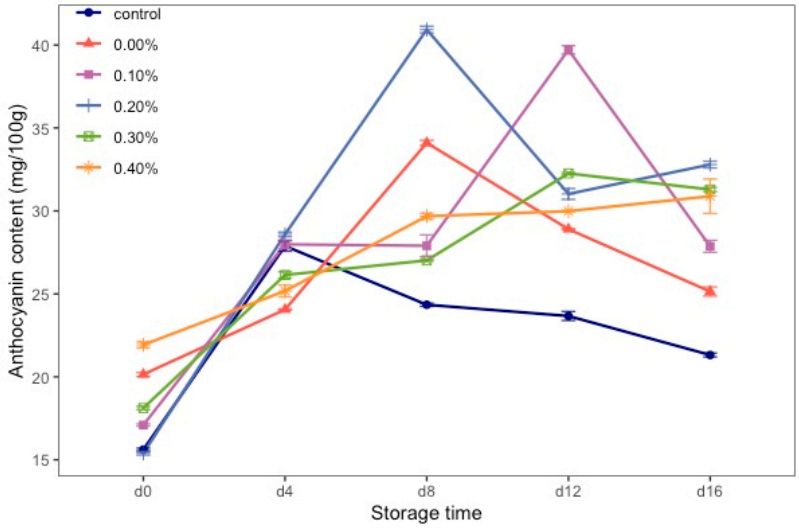


(**A**)

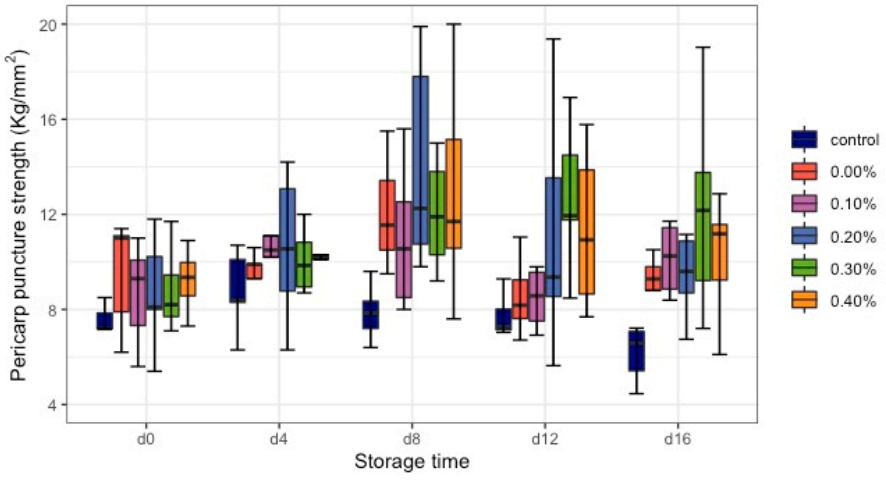
Figure 6. *Cont.*



(**B**)

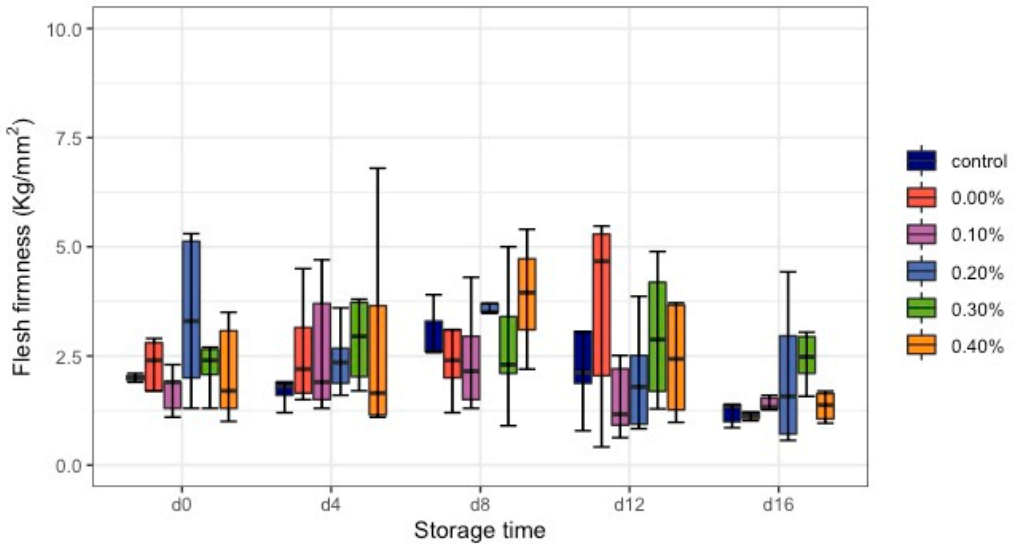


(**C**)



(**D**)

Figure 6. *Cont.*



(**E**)

Figure 6. The pH value (**A**), titratable acidity (**B**), anthocyanin content (**C**), pericarp puncture strength (**D**), and average flesh firmness (**E**) of blueberry during storage.

3.3.3. Changes of Blueberry Hardness during Storage

Hardness is one of the key quality parameters affecting fresh fruit quality and consumer acceptance [32]. Figure 6D,E shows the changes of puncture strength of pericarp, and the average firmness of pulp of blueberry with different treatments during storage. The puncture strength of pericarp can reflect the ability of blueberry peel to resist external force penetration. At the early stage of storage, the peel and coating were dehydrated and dried, and thus the peel puncture strength increased. The average firmness reflects the softening degree of blueberry pulp. At the last stage of storage, the degradation of cell wall components in extracellular enzymes led to the softening of blueberry. Therefore, the puncture strength of pericarp and the average firmness of pulp showed a downward trend. The hardness of blueberry treated by coating was higher than the control group. This may be due to the fact that coating provides structural rigidity for the fruit surface, while reducing the metabolic activity in blueberry and minimizing softening [33].

4. Conclusions

In this study, Jerusalem artichoke residue (a food byproduct) was used to extract pectin, which increased the added value of Jerusalem artichoke industry. The extracted pectin has high purity, good state, and good heat resistance, which is suitable for coating materials. The extracted pectin and chitosan were developed into composite films. The characterization of the film showed that the film with pectin had a smooth and dense surface and good thermal stability. The film is thin and has a good barrier function, which is suitable as a berry fruit preservative film. The results showed that pectin coating could effectively improve the storage resistance of blueberry. The coating with 0.2% pectin showed the best performance. Following 16 days of storage, the weight loss rate of blueberry treated with 0.2% of pectin coating was 22% lower than the control group, the decay rate was 33% lower, the anthocyanin content was 35% higher, and the consumption of organic acids slowed down. In addition, the hardness of blueberry treated by coating was significantly higher than the untreated group. In conclusion, the pectin chitosan composite film can be used as a new edible coating to prolong the shelf life of blueberry.

Author Contributions: Conceptualization, L.L. and J.L.; methodology, X.Z. and L.L.; software, X.Z.; validation, X.Z. and X.S.; formal analysis, L.W.; investigation, L.L.; resources, L.L.; data curation, X.Z. and L.L.; writing—original draft preparation, X.Z.; writing—review and editing, X.Z. and L.L.;

visualization, X.S. and L.W.; supervision, L.L. and J.L.; project administration, L.L. and J.L. All authors have read and agreed to the published version of the manuscript.

Funding: This study was financially supported by the Shaanxi Province Technology Innovation Guidance Special Plan (2021QFY07-04).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data sharing is not applicable to this article.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Sawicka, B.; Skiba, D.; Pszczółkowski, P.; Aslan, I.; Sharifi-Rad, J.; Krochmal-Marczak, B. Jerusalem artichoke (*Helianthus tuberosus* L.) as a medicinal plant and its natural products. *Cell. Mol. Biol.* **2020**, *66*, 160–177. [CrossRef] [PubMed]
2. Zhu, T.; Shao, T.; Liu, J.; Li, N.; Long, X.; Gao, X.; Rengel, Z. Improvement of physico-chemical properties and microbiome in different salinity soils by incorporating Jerusalem artichoke residues. *Appl. Soil Ecol.* **2021**, *158*, 103791. [CrossRef]
3. Mohnen, D. Pectin structure and biosynthesis. *Curr. Opin. Plant Biol.* **2008**, *3*, 266–277. [CrossRef] [PubMed]
4. Pérez-Lavalle, L.; Carrasco, E.; Valero, A. Strategies for Microbial Decontamination of Fresh Blueberries and Derived Products. *Foods* **2020**, *9*, 1558. [CrossRef]
5. Medina-Jaramillo, C.; Quintero-Pimiento, C.; Díaz-Díaz, D.; Goyanes, S.; López-Córdoba, A. Improvement of Andean Blueberries Postharvest Preservation Using Carvacrol/Alginate-Edible Coatings. *Polymers* **2020**, *12*, 2352. [CrossRef]
6. Reichembach, L.H.; de Oliveira Petkowicz, C.L. Extraction and characterization of a pectin from coffee (*Coffea arabica* L.) pulp with gelling properties. *Carbohydr. Polym.* **2020**, *245*, 116473. [CrossRef]
7. Meng, Y.; Wang, S.; Guo, Z.; Cheng, M.; Li, J.; Li, D. Design and preparation of quaternized pectin-Montmorillonite hybrid film for sustained drug release. *Int. J. Biol. Macromol.* **2020**, *154*, 413–420. [CrossRef]
8. Priyadarshi, R.; Kim, S.-M.; Rhim, J.-W. Pectin/pullulan blend films for food packaging: Effect of blending ratio. *Food Chem.* **2021**, *347*, 129022. [CrossRef]
9. Kodoth, A.K.; Ghate, V.M.; Lewis, S.A.; Prakash, B.; Badalamoole, V. Pectin-based silver nanocomposite film for transdermal delivery of Donepezil. *Int. J. Biol. Macromol.* **2019**, *134*, 269–279. [CrossRef]
10. Meerasri, J.; Sothornvit, R. Characterization of bioactive film from pectin incorporated with gamma-aminobutyric acid. *Int. J. Biol. Macromol.* **2020**, *147*, 1285–1293. [CrossRef]
11. Pirsa, S. Biodegradable film based on pectin/Nano-clay/methylene blue: Structural and physical properties and sensing ability for measurement of vitamin C. *Int. J. Biol. Macromol.* **2020**, *163*, 666–675. [CrossRef]
12. Alvarez, M.V.; Ponce, A.G.; Moreira, M.R. Influence of polysaccharide-based edible coatings as carriers of prebiotic fibers on quality attributes of ready-to-eat fresh blueberries. *J. Sci. Food Agric.* **2018**, *98*, 2587–2597. [CrossRef] [PubMed]
13. Wang, S.; Zhou, Q.; Zhou, X.; Wei, B.; Ji, S. The effect of ethylene absorbent treatment on the softening of blueberry fruit. *Food Chem.* **2018**, *246*, 286–294. [CrossRef]
14. Lee, J.; Durst, R.W.; Wrolstad, R.E. Determination of Total Monomeric Anthocyanin Pigment Content of Fruit Juices, Beverages, Natural Colorants, and Wines by the pH Differential Method: Collaborative Study. *J. AOAC Int.* **2005**, *88*, 1269–1278. [CrossRef] [PubMed]
15. Rahmani, Z.; Khodaiyan, F.; Kazemi, M.; Sharifan, A. Optimization of microwave-assisted extraction and structural characterization of pectin from sweet lemon peel. *Int. J. Biol. Macromol.* **2020**, *147*, 1107–1115. [CrossRef]
16. Ezzati, S.; Ayaseh, A.; Ghanbarzadeh, B.; Heshmati, M.K. Pectin from sunflower by-product: Optimization of ultrasound-assisted extraction, characterization, and functional analysis. *Int. J. Biol. Macromol.* **2020**, *165*, 776–786. [CrossRef] [PubMed]
17. Hosseini, S.; Parastouei, K.; Khodaiyan, F. Simultaneous extraction optimization and characterization of pectin and phenolics from sour cherry pomace. *Int. J. Biol. Macromol.* **2020**, *158*, 911–921. [CrossRef] [PubMed]
18. Güzel, M.; Akpınar, Ö. Valorisation of fruit by-products: Production characterization of pectins from fruit peels. *Food Bioprod. Processing* **2019**, *115*, 126–133. [CrossRef]
19. Gao, H.S.; He, Z.; Sun, Q.; He, Q.; Zeng, W.C. A functional polysaccharide film forming by pectin, chitosan, and tea T polyphenols. *Carbohydr. Polym.* **2019**, *215*, 1–7. [CrossRef]
20. Lin, D.; Zheng, Y.; Wang, X.; Huang, Y.; Ni, L.; Chen, X.; Wu, Z.; Huang, C.; Yi, Q.; Li, J.; et al. Study on physicochemical properties, antioxidant and antimicrobial activity of okara soluble dietary fiber/sodium carboxymethyl cellulose/thyme essential oil active edible composite films incorporated with pectin. *Int. J. Biol. Macromol.* **2020**, *165*, 1241–1249. [CrossRef]
21. Song, X.; Liu, L.; Wu, X.; Liu, Y.; Yuan, J. Chitosan-Based Functional Films Integrated with Magnolol: Characterization, Antioxidant and Antimicrobial Activity and Pork Preservation. *Int. J. Mol. Sci.* **2021**, *22*, 7769. [CrossRef] [PubMed]
22. Liu, T.; Liu, L.; Gong, X.; Chi, F.; Ma, Z. Fabrication and comparison of active films from chitosan incorporating different spice extracts for shelf life extension of refrigerated pork. *LWT Food Sci. Technol.* **2021**, *135*, 110181. [CrossRef]

23. Kan, J.; Liu, J.; Yong, H.; Liu, Y.; Qin, Y.; Liu, J. Development of active packaging based on chitosan-gelatin blend films functionalized with Chinese hawthorn (*Crataegus pinnatifida*) fruit extract. *Int. J. Biol. Macromol.* **2019**, *140*, 384–392. [CrossRef]
24. Chen, Y.; Xu, L.; Wang, Y.; Chen, Z.; Zhang, M.; Chen, H. Characterization and functional properties of a pectin/tara gum based edible T film with ellagitannins from the unripe fruits of Rubus chingii Hu. *Food Chem.* **2020**, *325*, 126964. [CrossRef] [PubMed]
25. Sami, R.; Soltane, S.; Helal, M. Microscopic Image Segmentation and Morphological Characterization of Novel Chitosan/Silica Nanoparticle/Nisin Films Using Antimicrobial Technique for Blueberry Preservation. *Membranes* **2021**, *11*, 303. [CrossRef]
26. Li, S.; Yi, J.; Yu, X.; Wang, Z.; Wang, L. Preparation and characterization of pullulan derivative/chitosan composite film for potential antimicrobial applications. *Int. J. Biol. Macromol.* **2020**, *148*, 258–264. [CrossRef]
27. Sun, L.; Sun, J.; Chen, L.; Niu, P.; Yang, X.; Guo, Y. Preparation and characterization of chitosan film incorporated with thinned young apple polyphenols as an active packaging material. *Carbohydr. Polym.* **2017**, *163*, 81–91. [CrossRef]
28. Ezati, P.; Rhim, J.W. PH-responsive pectin-based multifunctional films incorporated with curcumin and sulfur nanoparticles. *Carbohydr. Polym.* **2020**, *230*, 115638. [CrossRef]
29. Paolucci, M.; Di Stasio, M.; Sorrentino, A.; La Cara, F.; Volpe, M.G. Active Edible Polysaccharide-Based Coating for Preservation of Fresh Figs (*Ficus carica* L.). *Foods* **2020**, *9*, 1793. [CrossRef]
30. Ciriminna, R.; Fidalgo, A.; Meneguzzo, F.; Presentato, A.; Scurria, A.; Nuzzo, D.; Alduina, R.; Ilharco, L.M.; Pagliaro, M. Pectin: A long-neglected broad-spectrum antibacterial. *Chem. Med. Chem.* **2020**, *15*, 2228–2235. [CrossRef]
31. Mannozzi, C.; Tylewicz, U.; Chinnici, F.; Siroli, L.; Rocculi, P.; Rosa, M.D.; Romani, S. Effects of chitosan based coatings enriched with procyanidin by-product on quality of fresh blueberries during storage. *Food Chem.* **2018**, *251*, 18–24. [CrossRef] [PubMed]
32. Jiang, Y.; Yu, L.; Hu, Y.; Zhu, Z.; Zhuang, C.; Zhao, Y.; Zhong, Y. Electrostatic spraying of chitosan coating with different deacetylation degree for strawberry preservation. *Int. J. Biol. Macromol.* **2019**, *139*, 1232–1238. [CrossRef] [PubMed]
33. Medina-Jaramillo, C.; Quintero-Pimiento, C.; Gómez-Hoyos, C.; Zuluaga-Gallego, R.; López-Córdoba, A. Alginate-Edible Coatings for Application on Wild Andean Blueberries (*Vaccinium meridionale* Swartz): Effect of the Addition of Nanofibrils Isolated from Cocoa By-Products. *Polymers* **2020**, *12*, 824. [CrossRef] [PubMed]