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Research article

Biodegradable film based on barley sprout powder/pectin modified with quercetin and V_2O_5 nanoparticles: Investigation of physicochemical and structural properties

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A B S T R A C T

In this study, barley sprout powder/pectin (BS/Pec) composite film was prepared. Quercetin (Qu) and vanadium oxide (V_2O_5) nanoparticles were used to improve the physicochemical and structural characteristics of the film. The structural, physicochemical and thermal properties of the films were investigated by various techniques such as TGA, SEM, XRD, FTIR, texture analysis, etc. The thickness and tensile strength of the films increased from 120 μm to 2.4 MPa to 220 μm and 6 MPa respectively with the increase of V_2O_5 nanoparticles and quercetin pigment. Nanoparticles of V_2O_5 and quercetin decreased the moisture content of the film from 50% to 20%. Quercetin had little effect in reducing water vapor permeability (WVP), but V_2O_5 nanoparticles had a significant effect in reducing WVP. The pure BS/Pec film had almost 30% antioxidant properties, which increased to 81% with the increase of quercetin. Adding quercetin and V_2O_5 nanoparticles to the film increased the antimicrobial properties of the film against both *Escherichia coli* and *Staphylococcus aureus* bacteria. The SEM images showed the inhomogeneous surface of the BS/Pec film caused by BS powder fibers. The interactions between the components of the films (electrostatic type) was confirmed by FTIR results. The degradation temperature of the overall structure of the film in the presence of nanoparticles indicated the positive effect of nanoparticles in increasing the thermal resistance of the film. Investigating the crystal structure of the film showed that the BS/Pec film has an amorphous/crystalline or semi-crystalline structure. Considering that the prepared film has good mechanical properties and as well as antioxidant/antimicrobial properties, this film as an active composite can be used in food products packaging.

1. Introduction

Conventional plastics are made from petroleum and other non-renewable resources. So, in fact, it should be said that non-biodegradable plastic is made from non-renewable resources cause environmental pollution due to long time remaining in the environment. Biodegradable plastics have the same properties as plastic and are biodegradable by hydrolysis and other chemical reactions. As a result, these types of plastics are either converted into oligosaccharides (a type of carbohydrate) or into monomers through polymer decomposition, or into gaseous components through mineralization [1,2]. One of the important sources of

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environmentally friendly plastics are polysaccharides and their derivatives. Cereal powder and polysaccharides extracted from them are used in the preparation of biodegradable plastics. Barley sprout powder, pectin, proteins and polysaccharides are among the most important biodegradable sources capable of producing plastic [3–6].

Barley is one of the most widely used grains in the diet. This material is used almost everywhere. This seed is versatile and is a chewy food item with a nutty flavor. This seed is used as a supplement in many foods. Therefore, barley sprout powder can be called a full-fledged supplement. This seed has many nutrients in its structure and is very useful for human health. It causes weight loss and improves food digestion, reduces the amount of cholesterol in the body and is very effective in the health of the human heart. Barley sprout powder has all the properties of sprouts and you can use this product orally or use it to make a natural face mask. This product is obtained by drying and grinding barley sprouts. In this way, after sprouting and reaching the ideal size, the barley seeds are completely dried in the dryer and then milled. Organic substances such as ascorbic acid (vitamin C), thiamin (vitamin B1), vitamin A, vitamin K, riboflavin (vitamin B2), tocopherol alpha (vitamin E), niacin (vitamin B3), vitamin B6 and folate are abundant in this seed. Useful metals such as potassium, magnesium, phosphorus, zinc, iron and calcium are also present in this useful substance. Barley sprouts are rich in antioxidants, essential amino acids and useful enzymes. Barley sprouts provide enough fiber to the body and have no harmful cholesterol [7,8].

Pectin is actually a powerful fiber, found naturally in many plants and vegetables, which acts as a bonding agent between plant cells. It is a sugar-acid derivative polymer that is obtained from plant gelatin structures found in fruits and vegetables. The maximum amount of pectin is found in unripe fruits, after reaching the ripening stage, the amount and even the quality of extracted pectin decreases. Galactronic acid is the main chemical building block of pectin found in fruits and vegetables, which is one of the group of anhydro-galactronic acid polymers. Pectin has the ability to form biodegradable plastics [9,10].

Vanadium pentoxide (V_2O_5) is a porous, relatively rare and valuable metal that is very strong and resistant, which is used in the steel industry to make special steel, in the chemical industry as a catalyst, in ceramics as a color, and in photography and medicine. The color is yellowish brown. This material is recently used in the preparation of films and substrates with catalytic properties. Also recently, the antimicrobial property of this nanoparticle has been confirmed [11].

Quercetin as a powerful antioxidant (flavonol) is found in most fruits, vegetables, leaves, and seeds. It can be used in food supplements, wines, or foods. Quercetin is one of the types of polyphenols and flavonoids found in coffee. This substance plays a role in the antioxidant, anti-cancer, anti-viral, and anti-inflammatory properties of coffee. Also, quercetin fights free radicals and helps to boost the flow of nutrients in the blood vessels. In other words, quercetin is a type of flavonoid that has biological properties and may improve physical and mental performance. It has been shown that this compound has anti-cancer, anti-inflammatory, antiviral and antioxidant properties. Quercetin is the most abundant flavonoid in human diet [12,13].

Considering the very useful biological properties of barley sprouts, very useful organic and mineral substances in this natural substance, as well as the very suitable properties of pectin, in this study, the combination of barley sprout powder and pectin was used for preparation of composite films. To increase the biological properties of this polymer, a combination of vanadium oxide nanoparticles with catalytic and antimicrobial properties and quercetin pigment with strong antimicrobial and antioxidant properties were used. The effect of quercetin pigment (as powerful antioxidant) and V_2O_5 nanoparticles and on the physicochemical (structural/mechanical) properties of the composite film was investigated.

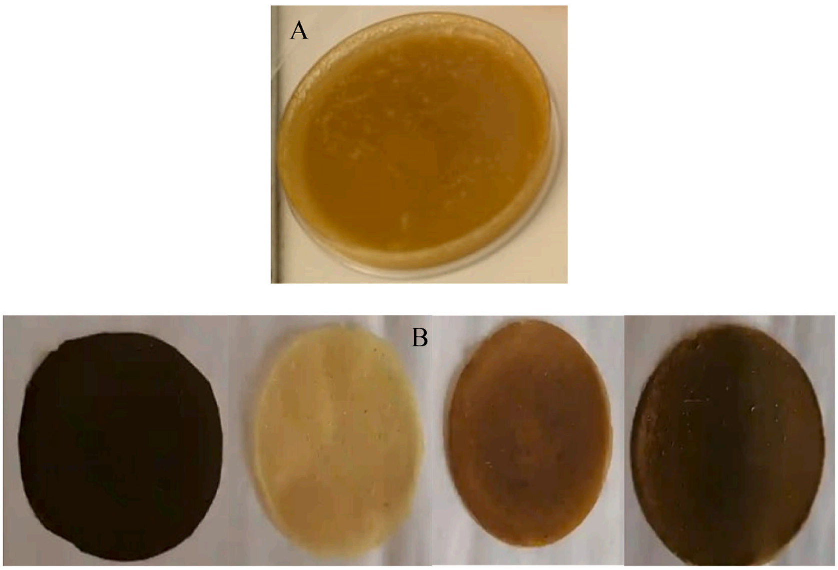

Fig. 1. Preparation of film solution (A), some prepared films (B).

2. Material and methods

2.1. Materials

Barley sprout powder was purchased from medicinal plant store in Urmia (West Azerbaijan-Iran). High-methoxyl pectin with code (EINECS, 232-553-0) was obtained from Jahan-Shimi Company (Iran-Tehran). Vanadium oxide (V_2O_5) nanoparticles with a purity of 99% were purchased from Araz-Kimyaye-Jahan Company (Iran-Tabriz). Quercetin powder with a purity of 99.9% was obtained from Merck (Germany). Glycerol, 2,2-diphenyl-1-picrylhydrazyl (DPPH), ethanol methanol and other materials were obtained from Aldrich Co. (USA) and Merck Co. (Germany). All chemicals were used without purification. Double-distilled water was used for solution preparation and other laboratory tests.

2.2. Film preparation

For preparation of barley sprout powder/pectin composite film and its composites with quercetin and vanadium oxide nanoparticles. For this purpose, 1 g of barley sprout powder was dissolved in 50 mL of distilled water. Dissolution was done on a heater stirrer at a temperature of 40 °C. A magnet with a speed of 200 rpm was used to stir the solution. The dissolution will continue for 1 h. In another solution, 1 g of pectin powder was dissolved in 50 mL of distilled water. Dissolution was done on a heater stirrer at a temperature of 40 °C. A magnet with a speed of 200 rpm was used to stir the solution. The dissolution was continued for 1 h. Barley sprout solution and pectin solution were mixed together and 800 mg of glycerol was added to their collection as a plasticizer and stirring of the solution was continued for 30 min. Next, quercetin pigment in three levels (0, 20 and 40 mg) and vanadium oxide nanoparticles in three levels (0, 50 and 100 mg) (according to the statistical design, Table 1) was added to the solution and stirred for 40 min at temperature 40 °C was continued. Finally, 30 mL of the prepared uniform solution was poured into plastic plates with a diameter of 10 cm and dried at room temperature for 24 h (Fig. 1-A). The dried films were separated from the plates and stored in zipped bags in the dark at room temperature until the tests are performed (Fig. 1-B).

2.3. Film tests

2.3.1. Thickness

At 5 different section, the film thickness was measured (by a micrometer with 0.01 mm accuracy) (Insize Digital Outside Micrometer 3108-25A) and then the average was obtained. The average thickness was used to study some mechanical properties.

2.3.2. Mechanical properties

The mechanical properties (tensile strength (TS) and elongation at breaking point (EB)) of the films are important factors for food packaging. To check the mechanical properties, the samples was conditioned for 24 h in relative humidity (RH) = 55%. At first, the films were cut with a special cutter in dimensions of 1 × 8 cm in. The films were in a dumbbell shape. The films were placed between the two jaws of the machine. The initial distance between the two jaws was 50 mm. The upper jaw was moved relative to the lower jaw at a speed of 5 mm/min. The mechanical properties were recorded by a computer. For this test, a brand histometer (Zwick/Roell model FR010, Germany) was used [3].

2.3.3. Solubility in water

Film solubility is defining as the ratio of solids dissolved in water to the initial weight of film pieces, after immersing them in distilled water (According to the ASTM definition). To determine solubility, 2 × 2 cm of each film were weighed (initial weight = W_i) after drying in an oven (105 °C) and then were immersed in 50 mL of distilled water at a temperature of 25 °C (for 6 h). Then, the insoluble materials of film were filtered and dried in an oven (105 °C) and weighted and considered as the final weight (W_f). The solubility of each film was calculated by equation (1) [3]:

$$\text{Solubility}\ (\%) = \frac{\mathbf{W_i - W_f}}{\mathrm{i}} \times 100 \tag{1}$$

W_i: Initial sample weight.
W_f: Sample weight after drying.

2.3.4. Moisture content

To perform this test, the films were first cut (dimensions 3 × 3 cm). Then the cut films were stored in relative humidity of 55% (for 24 h at 25 °C). The desiccator contained silica gel. At this stage, the weight of the films was measured (initial weight = W_i). Then, to completely remove the moisture from the films, the film was heated at 100 °C (for 24 h). Then the films were again measured and recorded (final weight = W_f). equation (2) was used to calculate the moisture content of the film [3].

$$\text{Moisture}\ (\%) = \frac{\boldsymbol{W_i - W_f}}{\boldsymbol{W_i}} \times 100 \tag{2}$$

W_i: Initial film weight.
W_f: final film weight.

2.3.5. Water vapor permeability (WVP)

Standard method (ASTME-96 standard method) was used for WVP determination [14]. To perform this test, anhydrous calcium chloride (2.5 mg) was poured into the falcons. The opening area (0.019625 m^2) of the falcons were covered with composite films by melted paraffin. Saturated $CaCl_2$ solution was used to maintain the relative humidity gradient (75%). The difference in relative humidity on both sides of the film at 25 °C creates a heating pressure equal to 1753.55 Pa. In this way, the changes in the weight of falcons over time were measured using a digital scale. The weight gain curve of falcons was drawn with the passage of time and the linear regression was calculated. The slope of the line was calculated and by dividing the slope of the weight loss line to the surface of the film, the Water Vapor Transmission Rate (WVTR) was calculated. Finally, WVP was calculated by dividing the Water Vapor Transmission Rate (WVTR) by the vapor pressure difference on both sides of the film and multiplying it by the thickness of permeability to water vapor (equation (3)).

$$\mathrm{WVP} = \frac{\mathrm{WVTR}\ .\ \mathrm{X}}{\mathrm{P}(\mathrm{R1}\ - \mathrm{R2})} \tag{3}$$

X: Film thickness (m)

P: Vapor pressure of pure water at 25 °C.

R_1: Relative humidity in the desiccator (RH = 97%)

R_2: Relative humidity inside the vial (RH = 0%)

2.3.6. Antioxidant property

Diphenylpicrylhydrazyl (DPPH) free radical inhibitory method was used for determination of the antioxidant activity of the films. For this test, 6 cm^2 of films were immersed in methanol and vortexed vigorously for 10 min. During this time, the film was digested and antioxidant compounds were released to the solution. The solution was centrifuged for 10 min at 3000 rpm. Then, to determine the antioxidant activity, the methanol supernatant was used. 1 mL of sample supernatant and 1 mL of methanol was mixed with 2 mL of DPPH ethanol solution (0.06 mM). 2 mL of methanol (without the presence of the film sample solution) was used as control sample. After stirring for 1 min, the mixture was placed in a dark place (at 25 °C for 30 min). Finally, the absorption of mixture was measured at 517 nm. A spectrophotometer (Dr-4000.TOG/TPH) was used for this test. equation (4) was used for calculation of the antioxidant property.

$$\text{Antioxidant activity } (\%) = \frac{A_{c-A_s}}{A_c} \times 100 \tag{4}$$

where A_b is equal to the absorbance of the control and A_s is equal to the absorbance of the film sample.

2.3.7. Antimicrobial property

With a slight change, the agar diffusion method was used to determine the antibacterial properties of the films. For this purpose, the films were cut into discs (with a diameter of 15 mm) and then placed on Mueller Hinton agar plates containing *Staphylococcus aureus* ATCC6538 and *Escherichia Coli* ATCC13706, with a concentration of (10^7 CFU/mL). The plates were incubated for 24 h at 37 °C. After 24 h, the radius of the halos of bacterial growth around the films (in millimeters) was measured with a precise caliper [6].

2.3.8. Scanning electron microscope (SEM)

The SEM instrument (ZEISS, SIGMA, Germany) was used to study the morphology, particle distribution, porosity, polymer structure and surface characteristics of the prepared films. The surface of the films was coated with a thin layer of gold. For the setting device, the accelerator voltage of 20 kV was used. The films were placed in the special position of the device and taking pictures of the films with different magnifications was done [3].

2.3.9. Fourier transform infrared (FTIR)

FTIR technique was used as a powerful method to investigate chemical/physical interactions between composite film components. FTIR device)Tensor 27, Bruker(made in Germany, was used for this work. For this purpose, first the films were dried and completely powdered by a grinder. The powder obtained from the film was mixed with KBr at a ratio of 1:20 and made uniform. The uniform mixture was made into a thin tablet with a special press. The FTIR spectrum of each composite film was recorded by the special software of the device. The spectrum of the samples was recorded with a resolution of 4 cm^{-1}. The wavenumber range of 400–4000 cm^{-1} was used to record the spectra [3].

2.3.10. X-ray diffraction (XRD)

XRD technique using X-ray device (Kristalloflex D500, Siemens) made in Germany was used to investigate the crystalline/amorphous structure of the composites. In this test, the film samples are placed in the special position of the sample cell. Radiant rays were irradiated to the sample at ambient temperature and reflected rays were detected in the range of angle $2\theta = 0$–80. The XRD spectrum of the samples was recorded by the device and plotted with the special software of the device. The Cu Ka radiation source was used at a wavelength of 0.154 nm. The X-ray generator was used at a voltage of 40 kV and 40 mA [3].

2.3.11. Thermogravimetric analysis (TGA)

The TGA thermal analyzer (Linseis–L81A1750, Germany) was used for TGA test. For this purpose, the film samples were heated of 50 mL/min in aluminum cups. The nitrogen atmosphere was used for heating and temperature range was of 30–600 °C. The heating rate of 10 °C/min of was used. As a reference, the empty aluminum cup was used. The TGA curve was drawn and recorded by the device [6].

2.4. Statistical analysis

In the first stage, the effect of the amount of independent variables including the amount of quercetin pigment and the amount of V_2O_5 nanoparticles (each in three levels) on the physicochemical properties of the composite film, including thickness, mechanical characteristics (percentage of elasticity and resistance to breaking point), moisture percentage, solubility in water and WVP was studied by the central composite design (CCD) (Table 1–A). In this section, Design Expert-10 software was used to design experiments and study mathematical relationships between independent and dependent variables. In this study, the probability level of 95% was considered. In the second part, the factorial design was used to investigate the effect of independent variables including the amount of quercetin pigment and the amount of V_2O_5 nanoparticles on antioxidant properties, antibacterial properties (against *E. coli* and *S. aureus*), morphology, FTIR spectroscopy, thermal resistance and crystalline characteristics (Table 1–B). In this section, Mini Tab-16 software was used to design experiments and analyze results and compare averages. In this section, the probability level of 95% was considered.

3. Result and discussion

3.1. Thickness, tensile strength (TS) and elongation at break (EAB)

Biodegradable polymers, despite having many advantages in terms of keeping the environment clean, have disadvantages such as weak mechanical and physical properties. Therefore, mechanical properties are of special importance in biodegradable polymers, and the thickness of the film is one of the factors that affect the mechanical properties of the film. Fig. 2 shows the three-dimensional and perturbation curves of the effect of quercetin and V_2O_5 nanoparticles on the thickness, TS and EAB of the BS/Pec films. In these curves, mathematical models of the relationship between independent and dependent variables, as well as regression coefficients and adjusted regression coefficients are also reported. By examining the obtained curves, it is clear that the thickness and tensile strength of the films increased with the increase of V_2O_5 nanoparticles and quercetin pigment. In this way, the thickness and tensile strength of the control film (BS/Pec) were 120 μm and 2.4 MPa, respectively, and with the addition of V_2O_5 nanoparticles and quercetin pigment, these values increased to 220 μm and 6 MPa. Also the effect of V_2O_5 in increasing the thickness was greater than the effect of quercetin. Considering that nanoparticles increase the dry matter of the composite, the increase in thickness in the presence of V_2O_5 has been predictable. On the other hand, quercetin pigment, by being placed in the pores of the film, in addition to making the structure of the film coherent and increasing the mechanical resistance of the film, can also increase the thickness of the film. The elasticity and flexibility of the film increased with the increase of quercetin, but the increase of nanoparticles decreased the flexibility of the film. EAB in control film (BS/Pec) was 30% that with the increase of quercetin the EAB it was increased to 50% and with the increase of nanoparticles it was decreased to 20%. Due to the structure of quercetin, which has many O and OH groups, and these groups can establish electrostatic interactions with the active groups of pectin and barley sprout powder and increase the consistency of polymer chains, and while increasing the tensile strength, they can increase the elongation. On the other hand, the O groups present in V_2O_5 nanoparticles probably hinder the easy movement of the polymer chains and reduce the flexibility of the film by involving the polymer chains.

Zhang et al. (2022) prepared chitosan/zein degradable composite film and studied the effect of curcumin pigment and nanoparticles on the mechanical properties of the film. Their results showed that adding of curcumin pigment and nanoparticles to the film significantly increased the elongation at break from 63.2 to 86.8% and increased tensile strength from 32.4 to 45.1 MPa. The results of their research show good agreement with the results of the current research [15]. Also, in another similar study, Santana et al. (2019)

Table 1-A
List of prepared films based on central composite design.

Run	A: Quercetin (mg)	B: V2O5 (mg)
1	40	0
2	20	50
3	40	100
4	0	50
5	20	50
6	0	0
7	40	50
8	20	50
9	20	0
10	0	100
11	20	100
12	20	50
13	20	50

Table 1-B
List of prepared films based on factorial design.

Film type	A: Quercetin (mg)	B: V_2O_5 (mg)
BS/Pec	40	0
BS/Pec/Qu	20	50
BS/Pec/V_2O_5	40	100
BS/Pec/Qu/V_2O_5	0	50

investigated the morphological and mechanical properties of cassava starch film modified with different nanoparticles. They have reported that nanoparticles increase the mechanical strength and improve the mechanical properties of the biodegradable composite films, and their research results partially confirm the results of the current research [16].

3.2. Moisture, solubility in water and WVP

Considering that biodegradable polymers are used in the packaging of food products and considering the effect of moisture and water on the spoilage of food products, the characteristics of biodegradable films in terms of percentage of moisture and resistance to water and preventing water penetration are extremely important. Fig. 3 shows the three-dimensional and perturbation curves of the effect of quercetin and V_2O_5 nanoparticles on the moisture, solubility and WVP of BS/Pec films. In these curves, mathematical models of the relationship between independent and dependent variables, as well as regression coefficients and adjusted regression coefficients are also reported. According to the moisture percentage results, both V_2O_5 nanoparticles and quercetin reduced the moisture content of the film and both had almost the same effect that when they were added to the film at the same time, they increasingly reduced the moisture content and in other words have had a synergistic effect on each other. The O and OH groups related to quercetin, which are abundant in the structure of this compound, as well as the O groups in the V_2O_5 nanoparticles can cover the O and OH groups in the polymer chains of pectin and barley sprout powder, and thus prevent the interaction between water molecules and O/OH groups in the polymer chains of pectin and barley sprout powder and ultimately reduce the water holding capacity or the moisture content of the film. Also, the water solubility of the films has decreased with the increase of V_2O_5 and quercetin nanoparticles. The reduction of soluble in water in the presence of V_2O_5 nanoparticles and quercetin was significant ($p < 0.05$). As mentioned in the discussion of mechanical properties, V_2O_5 and quercetin nanoparticles by creating electrostatic interactions with polymer chains and creating a coherent structure increase the mechanical resistance of the film, which makes the film have high resistance against water molecules. The curves related to water vapor permeability also show that quercetin had little effect in reducing water vapor permeability, but V_2O_5 nanoparticles had a significant effect in reducing WVP. Due to the fact that V_2O_5 nanoparticles greatly increase the thickness of the film and increasing the thickness of the film causes water molecules to take a longer path to pass through the width of the film, so the WVP decreases.

Ngo et al. (2020) have studied the properties of water resistance and water vapor permeability of pectin-based films modified with nanoparticles. They reported that nanoparticles increase the resistance of films to water solubility, which confirms the results of the present research [17]. Jayakumar et al. (2019) investigated the effect of combining polyvinyl alcohol and starch films as well as the effect of nanoparticles on aqueous properties and film resistance to dissolution in water. They showed that nanoparticles significantly increase the water resistance of films, and their results are in good agreement with the results of the present research [18].

3.3. Antioxidant and antibacterial properties

Antioxidant and antimicrobial properties of films are very important from the point of view of active food packaging to increase the shelf life and maintain the freshness and quality of food products. On the other hand, the release of antioxidant substances in the structure of the active film into the food can make the food more useful and as an anti-cancer substance, it can also apply medicinal and therapeutic properties to the food. Table 2 shows the antioxidant and antimicrobial properties (against *Escherichia coli* and *Staphylococcus aureus*) of composite films.

As it is clear from the results of the antioxidant property, the pure BS/Pec film also has about 30% antioxidant property, and this antioxidant property can be caused by the physical removal of DPPH radicals that are trapped in the structure of the composite polymer. Also this can be related to vitamins and minerals in the structure of barley sprout powder. It was also observed that with the increase of quercetin, the antioxidant property increased strongly. Quercetin as an antioxidant polyphenol in the structure of the composite film also has a very good effect in deactivating cancer-causing radicals. According to the results, V_2O_5 nanoparticles also had a small effect on increasing the antioxidant property, which is probably related to the physical removal of DPPH radicals by these nanoparticles. BS/Pec/Qu/V_2O_5 film, which contains both quercetin and nanoparticles, has shown the highest antioxidant properties. Examining the antimicrobial properties of the produced films showed that the pure BS/Pec film does not have significant antimicrobial properties against *Escherichia coli* and *Staphylococcus aureus*. Adding quercetin and vanadium oxide nanoparticles to the film has increased the antimicrobial properties of the film towards both bacteria. The effect of nanoparticles in creating antimicrobial properties in the film has been greater than that of quercetin. The previous research that has been done on quercetin and V_2O_5 nanoparticles has confirmed the antimicrobial properties of these two materials, and in this study, when these two materials were used in the composite film structure, they were able to show their antimicrobial properties. Also, the antimicrobial effect of quercetin and V_2O_5 nanoparticles on *Staphylococcus aureus* as a Gram positive bacterium was more than *Escherichia coli* as a Gram negative bacterium. Due

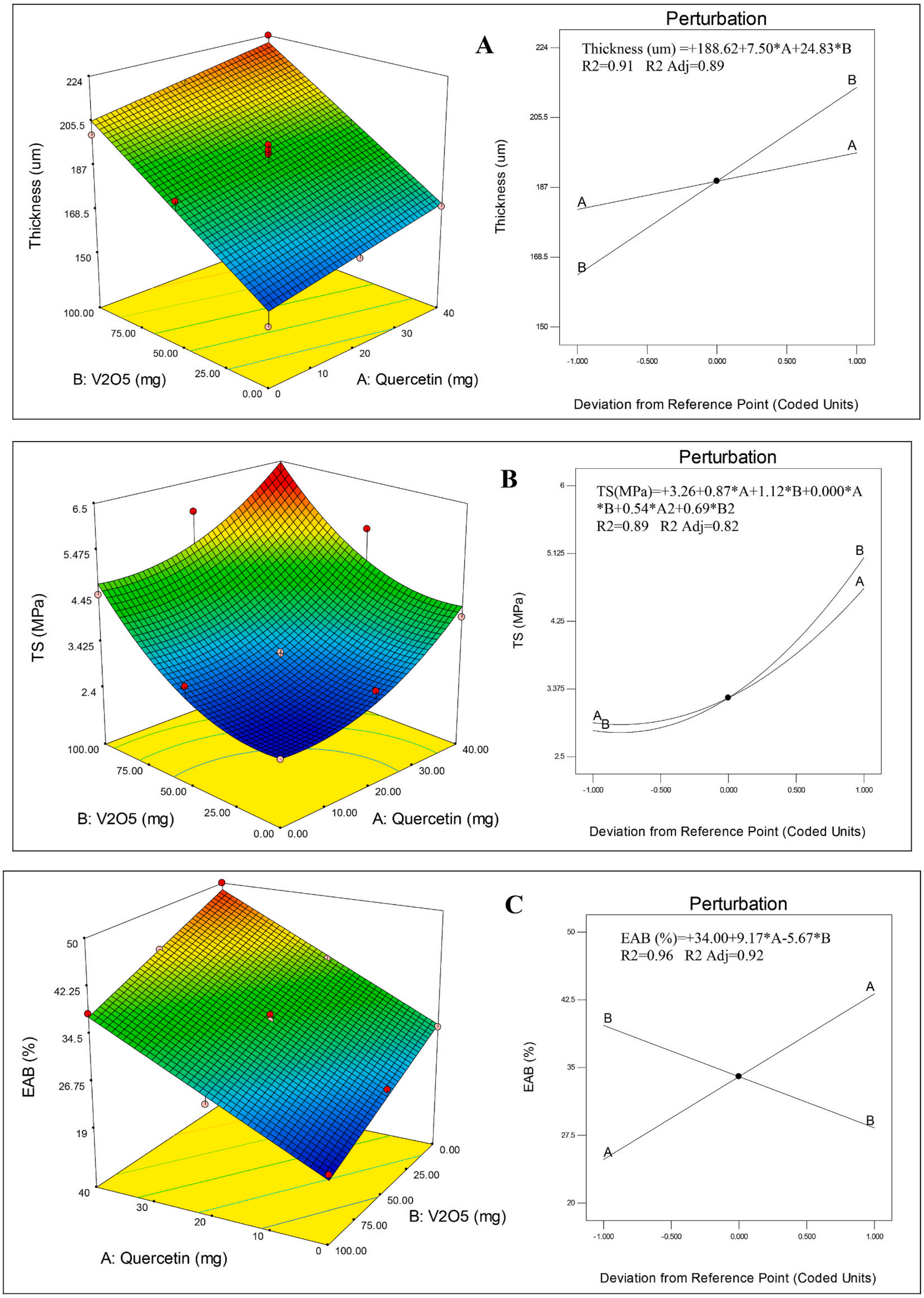

Fig. 2. Three-dimensional and perturbation curves of the effect of quercetin and V_2O_5 nanoparticles on film thickness (A), TS (B) and EAB (C).

to their strong and complex structure, Gram-negative bacteria are more resistant to antimicrobial agents than Gram-positive bacteria (with a weak and permeable cell wall), so the low effect of antimicrobial agents on *Escherichia coli* bacteria is acceptable.

Manso et al. (2021) have investigated and reported the antioxidant and antibacterial properties of plant extracts, cardamom polyphenol and different flavonoids in a review article. According to their report, quercetin has strong antioxidant properties and has

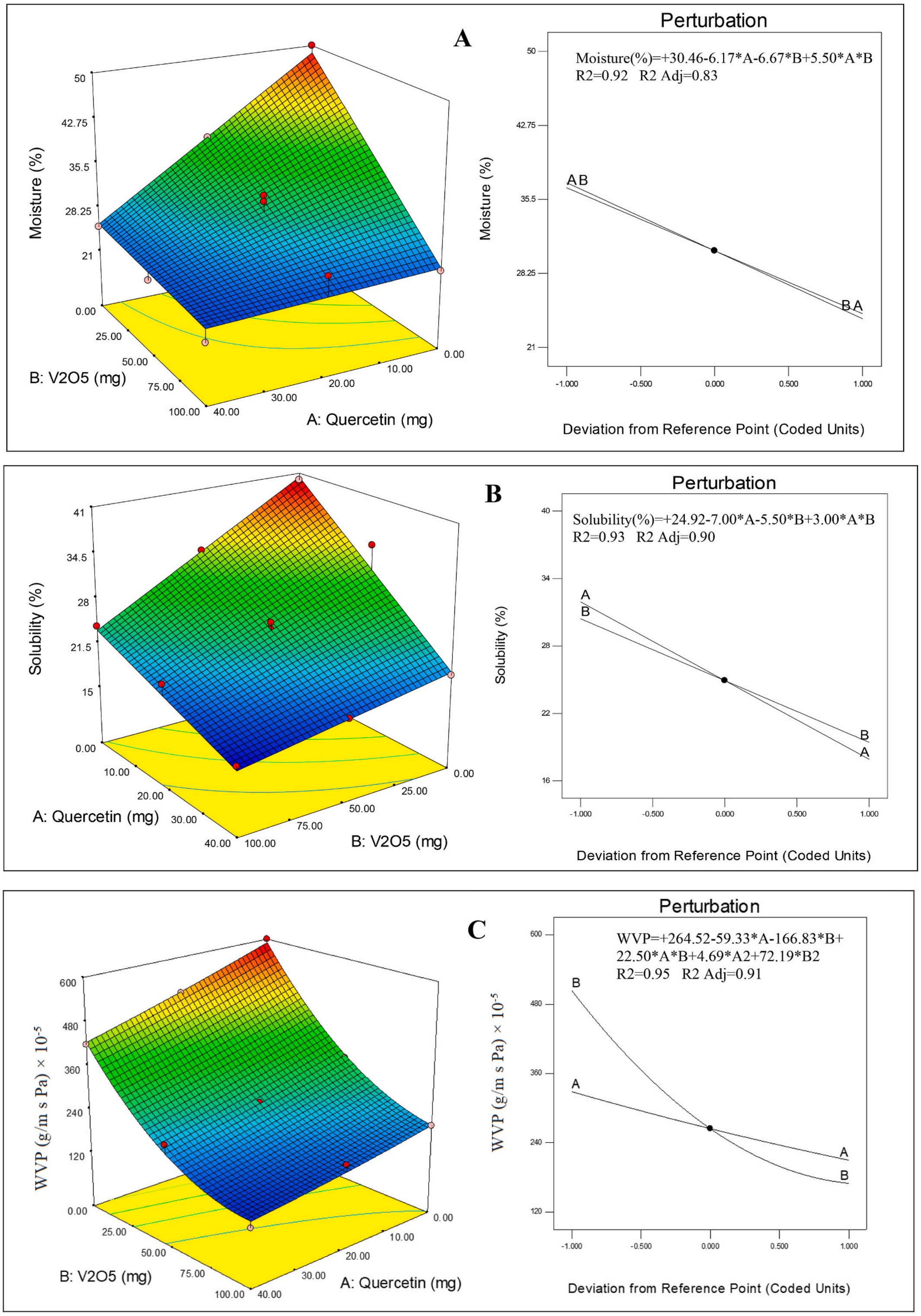

Fig. 3. Three-dimensional and perturbation curves of the effect of quercetin and V_2O_5 nanoparticles on the moisture (A), solubility (B) and WVP (C).

Table 2
Antioxidant and antibacterial properties of BS/Pec film and its composites.

Film	Antibacterial activity: Inhibition zone diameter (mm)		Antioxidant activity (%)
	Escherichia coli (G-)	*Staphylococcus aureus* (G+)	
BS/Pec	0*	0	31 ± 2^{a}
BS/Pec/Qu	8.8 ± 0.3^{b}	12.5 ± 0.4^{b}	75 ± 3^{c}
BS/Pec/V_2O_5	12.3 ± 0.4^{c}	14.0 ± 0.4^{c}	36 ± 1^{b}
BS/Pec/Qu/V_2O_5	14.7 ± 0.5^{c}	18.2 ± 0.3^{c}	81 ± 2^{d}

*Different letters in each column indicate the significance of the difference in means. The probability level of 95% was considered.

good antimicrobial properties [19]. Aljadaan et al. (2020) have investigated and confirmed the antimicrobial and antioxidant properties of quercetin and its derivatives, and the results of their report confirm the results of the current research [20]. Wang et al. (2016) have investigated and confirmed the antimicrobial property of V_2O_5. Their research results confirm the results of the current research [21].

3.4. SEM and FTIR

Fig. 4-A shows the SEM images of different BS/Pec films and its composites. SEM images show the inhomogeneous surface of the barley sprout/pectin film. This film contains fibers of barley sprout powder, which are clearly visible on the surface of the film. The existence of nanoparticles on the surface of the film is not visible in the barley sprout powder/pectin film containing V_2O_5 nanoparticles. Due to the fact that the surface of these films is very heterogeneous, the presence of nanoparticles on this surface is invisible. Also, in films containing quercetin pigment, the surface is somewhat inhomogeneity covered by this pigment.

FTIR spectra have been used to investigate the structure of composite films and the effect of quercetin pigment and V_2O_5 nanoparticles on the chemical structure of the composite film. Fig. 4-B shows the FTIR spectra of different BS/Pec films and its composites. Examining the spectrum related to the pure BS/Pec film shows the presence of functional groups related to pectin and barley sprout in this film. In this spectrum, the peak in wave number 3263 cm^{-1} is related to stretching vibrations of OH groups. Phytochemical compounds in barley sprouts have abundant OH groups. In addition, OH groups are abundant in pectin structure. The peak in 2924 cm^{-1} is related to the vibrations of R–CH_2-CH_3, R–CO–OH, C=C–CO–OH and –CH_2 groups, which are also present in the structure of pectin and barley sprout and confirm the structure film. Peak of 1589 is related to C–O stretching vibrations. Peak of 1411 is related to C–C vibrations and peaks of 1018, 1107 and 1319 are related to the stretching of C–O groups related to carboxylic acid. Peaks of 648 and 918 also show the bending vibrations of C–H and OH groups, respectively. By examining and comparing the spectra of BS/Pec/Qu, BS/Pec/V_2O_5 and BS/Pec/Qu/V_2O_5 with the pure BS/Pec spectrum, it was found that all the peaks related to pure BS/Pec film spectrum are seen also in these spectra. In the films containing nanoparticle and quercetin, the peaks related to different functional groups have shifted to different wavenumbers. This change in the wavenumbers shows the electrostatic interactions between BS/Pec with V_2O_5 and quercetin. Also, in the BS/Pec/V_2O_5 film, a new peak has appeared at wave number of 1022, which is probably related to V–O vibrations. In the spectrum of BS/Pec/Qu film, two new peaks have appeared at wave numbers of 1608 and 1662, which are related to C=C trans stretching vibrations in the quercetin structure. In the BS/Pec/Qu/V_2O_5 composite film, which has all the components of the composite film, all the peaks related to BS/Pec, V_2O_5 and quercetin are observed, but the wave numbers of the observed peaks have changed. Changing of peak numbers confirms the electrostatic interactions between composite components. Xu et al. (2022), Surya et al. (2016) and Catauro et al. (2015) have investigated and analyzed the FTIR spectra of barley sprout, V_2O_5 and quercetin, respectively [22–24]. The results obtained in this study are in good agreement with the results of similar researches.

3.5. Thermal resistance and crystalline properties

Fig. 5-A shows the thermal decomposition curves of different BS/Pec films and its composites with quercetin and V_2O_5 nanoparticles. According to the thermal decomposition curves, the pure film of barley sprout powder/pectin has been thermally decomposed in three stages. In the first stage, thermal decomposition occurred in the temperature range of 80–130 °C and approximately 15% of the film was thermally decomposed, probably related to the evaporation of water molecules and other volatile compounds with low boiling temperatures. In the second stage, thermal decomposition took place at a temperature of 140–200 °C, and at this stage, about 10% of the weight of the polymer was destroyed. This temperature decomposition is probably related to the evaporation of glycerol film or the decomposition of the compounds in the barley sprout powder that have a low decomposition temperature. The third stage of destruction, which happened between 220 and 300 °C, is related to the destruction of the main structure of the film. As can be seen, the entire film is not 100% destroyed and some film remains, which was predictable due to the presence of non-degradable mineral compounds in the structure of barley sprout powder. By examining the films containing V_2O_5 nanoparticles (BS/Pec/V_2O_5 and BS/Pec/Qu/V_2O_5), it was observed that in these films, the temperature of the film destruction has increased by about 10 °C, which indicates the positive effect of nanoparticles in thermal stability of the film. Also, in these films, the amount of destruction of the main structure of the film has also decreased by several percent, which is also due to the high thermal stability of V_2O_5 nanoparticles. Also, by examining the films containing quercetin (BS/Pec/Qu and BS/Pec/Qu/V_2O_5), it was observed that the destruction stage of the main structure of the film happened in two stages. Maybe the last stage of destruction in these films is related to the destruction of quercetin, which

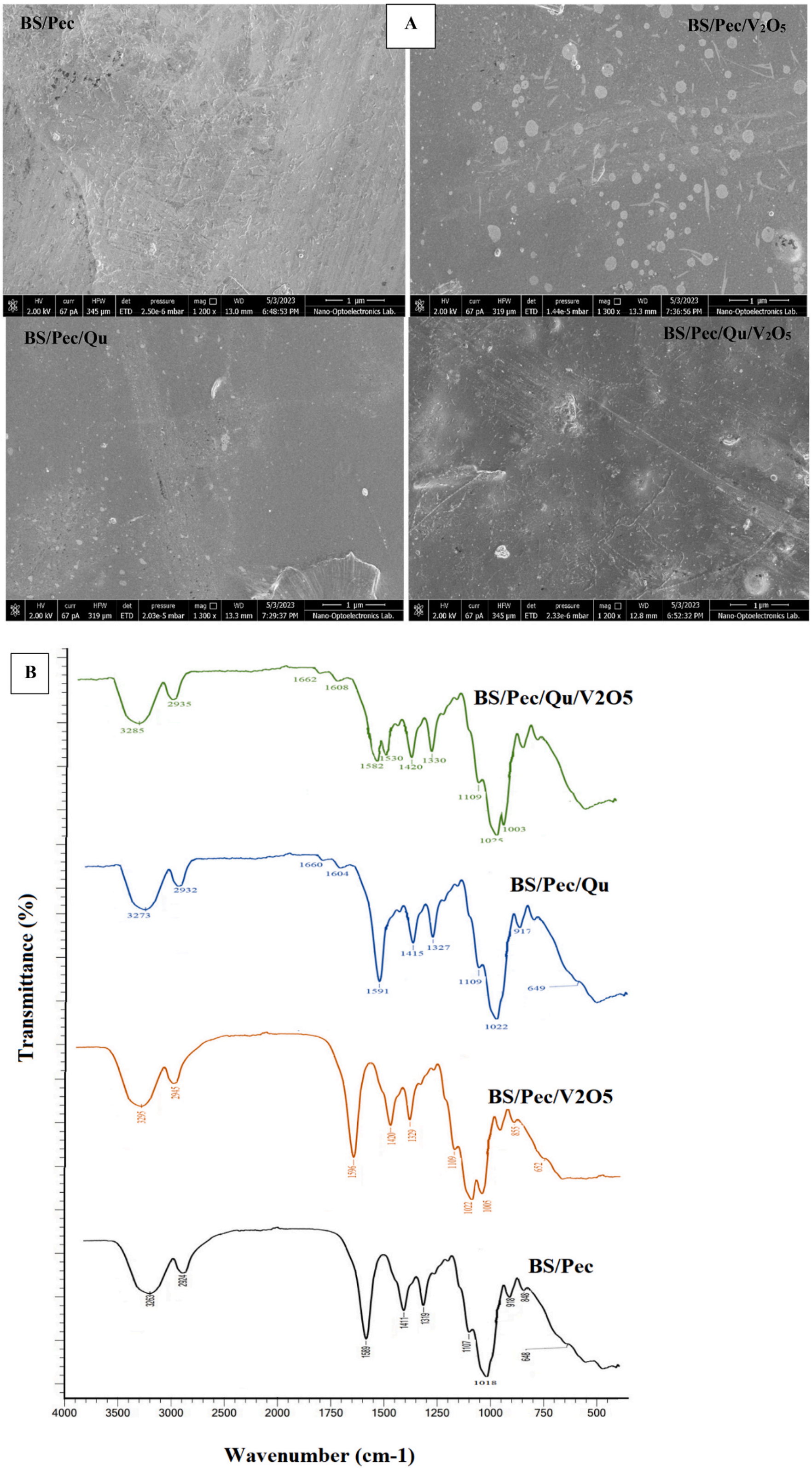

Fig. 4. SEM images (A) and FTIR spectra (B) of different BS/Pec films.

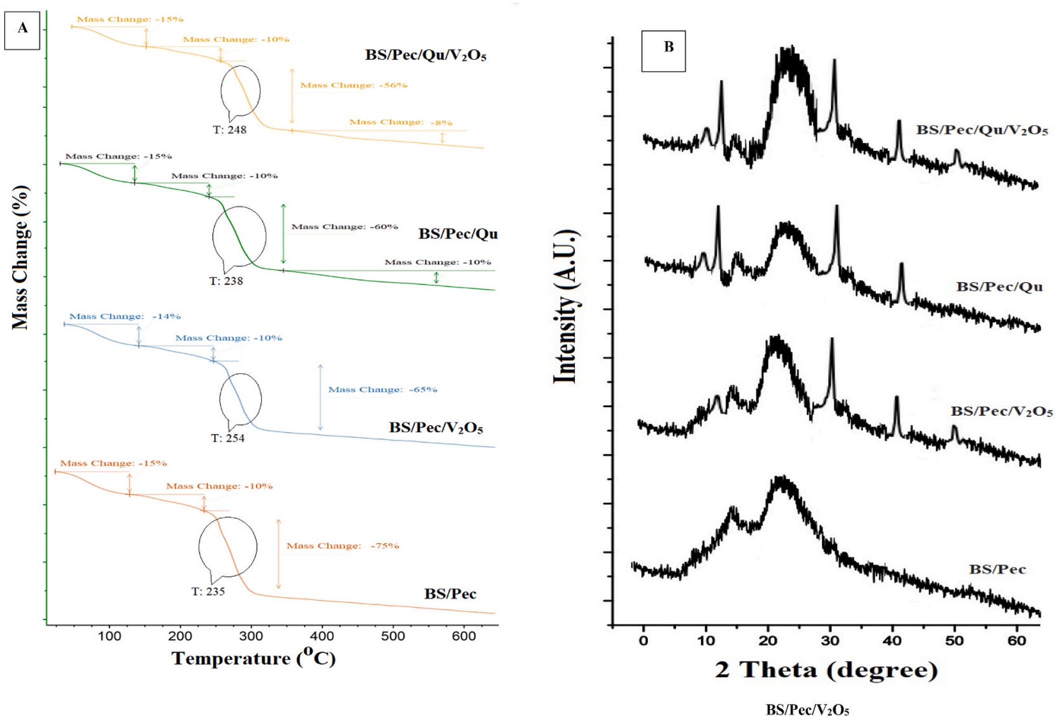

Fig. 5. TGA curves (A) and XRD spectra (B) of different BS/Pec films.

occurred at a temperature of 390–430 °C. The general conclusion that can be drawn from examining the thermal degradation curves is that both quercetin pigment and V_2O_5 nanoparticles have increased the thermal stability of the film.

Gorrasi et al. (2021) have investigated the structure and thermal stability of pectin film and pectin modified with polycaprolactone film. Their results confirm the results of the current research in terms of degradation temperature and degradation rate, as well as the effect of additives on film thermal stability [25]. Bruno et al. (2010) have investigated the thermal degradation of quercetin. The results of their research confirm the final stage of degradation in the structure of films containing quercetin [26].

Fig. 5-B shows the XRD spectra of different BS/Pec composite films. Examination of the XRD spectra of different films showed that in the pure film of BS/Pec, a relatively sharp peak at 2-theta of about 18° is observed, which is probably related to the crystalline celluloses present in barley sprout powder, and a relatively broad peak at 2-theta of about 22° is observed, which is related to the semi-amorphous structure of pectin, and previous researches have also confirmed the amorphous structure of pectin films [27]. So it can be concluded that the BS/Pec film has an amorphous/crystalline or semi-crystalline composite structure. Examination of films containing V_2O_5 nanoparticles (BS/Pec/V_2O_5 and BS/Pec/Qu/V_2O_5) showed that these crystalline nanoparticles are present in the film structure. The peaks in 2-theta of 12, 32, 43 and 50° show the existence of these nanoparticles in the film structure. Also, by examining the films containing quercetin (BS/Pec/Qu and (BS/Pec/Qu/V_2O_5) it was also found that firstly quercetin has a crystalline structure and secondly it has improved the overall crystalline structure of the film. The peaks in 2-theta of 12, 30 and 40° also indicate the presence of quercetin in the film structure. Of course, it should be mentioned that the intensity of peaks related to V_2O_5 and quercetin observed in the structure of the films compared to their pure state as reported in references are lower [28,29]. The reason for this result is that, firstly, the amount of these substances used is small, and secondly, due to the electrostatic interactions between these substances with barley sprout powder and pectin, some weak peaks related to these substances do not appear. The general conclusion obtained from examining the XRD spectrum is that the observation of peaks related to V_2O_5 and quercetin indicates the presence of these nanoparticles in the film structure and the induction of crystalline properties to the film. Hastuti et al. (2019) investigated the crystal structure of pectin and Carciofi et al. (2012) investigated the crystal structure of barley sprout powder and reported the results of the corresponding XRD spectra, which show a good agreement with the results of this research [27,30].

4. Conclusion

Biodegradable BS/Pec/Qu/V_2O_5 composite film was prepared and analyzed. According to the results, the thickness and tensile strength of the films increased with the increase of V_2O_5 nanoparticles and quercetin. Both V_2O_5 nanoparticles and quercetin decreased the moisture content of the film. Quercetin had little effect in reducing WVP, but V_2O_5 nanoparticles had a significant effect in reducing WVP. The control film (BS/Pec) had about 30% antioxidant property that with the increase of quercetin, the antioxidant property increased strongly. Adding quercetin and V_2O_5 nanoparticles to the film increased the antimicrobial properties of the film against both Gram-positive and Gram-negative bacteria. SEM images showed the inhomogeneous surface of the BS/Pec film. This film contains fibers of barley sprout powder, which were visible on the surface of the film. The FTIR results confirmed the electrostatic interactions between the composite components in the films. The degradation temperature of the overall structure of the film in the presence of nanoparticles increased about 10 °C. The XRD results showed that the composite film of BS/Pec has semi-crystalline structure.

Data availability statement

Data will be made available on request.

CRediT authorship contribution statement

Alaa Jasem odhaib: Investigation, Data curation. **Sajad Pirsa:** Writing – original draft, Validation, Software, Project administration. **Forogh Mohtarami:** Validation, Software.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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