Egyptian Journal of Chemistry

http://ejchem.journals.ekb.eg/

Preparation and Characterization of Biodegradable Pectin/Polyvinyl Alcohol Composite Membranes

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Abstract

People are becoming increasingly concerned about the environmental issues that arise due to the excessive usage of plastic and are starting to search for alternative biodegradable materials for food packaging. So, in the present work, biodegradable plastic films were prepared using pectin and polyvinyl alcohol (PVA) composites at the levels 1:2, 1:1 and 2:1 compared to pure PVA film. The prepared films were characterized using FT-IR, SEM and tensile techniques. The obtained results showed that PVA membrane doesn't have biodegradability rate, while the pectin membrane show very speed degradation. PVA/Pectin membranes at the ratio of 2:1, 1:1, and 1:2 loosed 9.4, 12.2, and 15.2% of their weight, respectively. FT-IR spectra of PVA/Pectin membranes showed good interactions between PVA and pectin through freeze-thawing process. Smooth surface structure of PVA membrane with no or few pores appeared under the SEM, while the pectin membrane was rough with many pores. PVA/Pectin membrane surfaces showed intermediate characteristics. Tensile test showed that the maximum stress increased from 16.25± 0.79 for PVA membrane and 12.54± 0.81 for pectin membrane to become more than 31 for PVA/Pectin membranes. Also, the maximum force increased from 14.63 ± 0.71 for PVA membrane and 7.72 ± 0.68 for pectin membrane to become 26.15 ± 0.80, 25.27 ± 1.51, and 48.00 ± 1.82 for PVA/Pectin membrane at the levels of 1:1, 2:1, 1:2, respectively, indicating enhanced mechanical properties with the increase of pectin concentration.
Keywords: Pectin; polyvinyl alcohol (PVA); biodegradable packaging membranes; microstructure; mechanical properties

1. Introduction

Biodegradable plastics are one of the segments of the global plastics industry that are expanding rapidly. The capacity for biodegradable plastics was 1.2 million tons annually in 2020, and it is anticipated to increase significantly **[1]**. It is challenging to switch from traditional plastics to eco-friendly polymers **[2]**. (bacteria and fungal enzymes) are capable of decomposing biodegradable polymers. The ability of a plastic to mineralize into gaseous end products in an environment with temperature, moisture, and microbial populations that meet biodegradability parameters is the definition of a biodegradable plastic **[1]**. Using both aerobic and anaerobic organisms, plastics should decompose into CO_2, methane, water, and nutrient-rich biomass, or compost. For biodegradable polymers, the generated organic matter needs to be safe for plants and animals **[3]**.

Plastic packaging is a major problem as it is an environmental pollutant **[4]**. Plastic packaging generates huge amounts of solid waste. It is good to look for ways to develop new packaging systems to reduce the risk of this waste **[5]**. Recently, a large number of packaging films using biopolymers, such as proteins and polysaccharides, have been developed. Polysaccharides (e.g., chitosan, starch, cellulose, pectin, and alginate) have gained attention due to their good film-forming ability **[6, 7].**

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Receive Date: 23 July 2023 Revise Date: 12 September 2023 Accept Date: 09 October 2023
DOI: 10.21608/EJCHEM.2023.224571.8300
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Numerous studies have documented the formation of pectin films with the addition of various substances **[8, 9]**. The majority of the pectin chain structure is made up of long homogalacturonic chains of a-(1-4)-D-galacturonic acid units, which are spaced out by sections of rhamnogalacturonan that alternately contain rhamnose and galacturonic acid residues **[10]**. The degree of methylation (DM) separates the two types of pectin by determining how much of the carboxylic groups in the galacturonic chain are in the form of methyl ester. More than 50% of the carboxyl groups are methylated in the high-methoxyl (HM) form, whereas less than 50% are methylated in the low-methoxyl (LM) form. The degree of methylation affects the gelling properties of pectin, with HM pectin requiring high sugar concentrations and a low pH to form a gel, while LM pectin can gel at lower sugar concentrations and a higher pH. This makes LM pectin more suitable for use in products such as jams and jellies that require a softer texture **[11]**.

Pure pectin films, however, are weak and rigid **[12, 13]**. Consequently, synthetic polymers like polyvinyl alcohol (PVA) or polyethylene glycol (PEG) can be combined with polysaccharide films to improve their mechanical properties. The addition of synthetic polymers to polysaccharide films also enhances their water resistance and biodegradability, making them suitable for various applications in the food and packaging industries **[14]**. However, the optimal ratio of polysaccharides to synthetic polymers must be carefully determined to achieve the desired properties **[15, 16]**. It has been shown by Coffin et al. (1996) that PVA was added to pectin to increase the films' toughness and decrease their brittleness **[17]**. One of the best-known synthetic hydrophilic polymers with controlled physicochemical properties and biodegradation time is PVA, which is both biodegradable and nontoxic to humans. The addition of PVA to pectin has been shown to improve the mechanical properties of the resulting films, making them more suitable for food packaging applications **[18]**. Moreover, PVA has a wide range of potential applications in the biomedical field due to its biocompatibility and biodegradability **[19, 20]**.

Thus, by fusing the distinctive qualities of the two co mponents (PVA and pectin), the combined PVA/Pectin films can have advanced properties.Addi tionally, the development of materials using PVA and pectin is consistent with the idea of sustainable d evelopment. This is because both PVA and pectin are biodegradable and can be obtained from renewable sources **[21]**. Therefore, the use of these materials in film production can contribute to reducing environmental impact and promoting a circular economy **[22]**.

Therefore, the goal of the current work is to create a suitable method for producing biodegradable films that can be used for food packaging. The method should be cost-effective and environmentally friendly. This will help to reduce the negative impact of non-biodegradable packaging materials on the environment.

2. Materials and Methods

2.1. Materials

Poly vinyl alcohol (PVA) (molecular mass of 146–186 kDa and 98–99% hydrolysis degree) was purchased from Sigma-Aldrich, Saint Louis, MO. Pectin from citrus peel (galaturonic acid ≥ 74%) was purchased from Sigma-Aldrich, Saint Louis, MO. sodium chloride, potassium chloride, di sodium hydrogen phosphate, potassium di hydrogen phosphate, and other chemical reagents were of analytical grade.

2.2. Methods

2.2.1. Preparation of polyvinyl alcohol (PVA) membrane

Poly (vinyl alcohol) was dissolved under stirring at 90 °C to obtain aqueous solutions of 2 wt% PVA concentrations. After complete dissolution, PVA solution was left to cool to room temperature. Then, the final solution was casted into a clean Petri dish. The casted PVA membranes were subjected to 6 cycles of freezing and thawing between -20 and 20 °C with a rate of freezing and thawing of 0.1 C min1 and a holding time of 6 h, following the procedure implemented by Wan et al. (2002) **[23]**. The dry membranes were taken off the Petri dish and then washed with distilled water.

2.2.2. Preparation of polyvinyl alcohol (PVA)/Pectin composite membrane

Poly (vinyl alcohol) was dissolved under stirring at 90 °C to obtain aqueous solutions of 2 wt% PVA concentrations. After complete dissolution, PVA solution was left to cool to room temperature. 2 wt% of pectin was added. Then, the final solution was casted into a clean Petri dish. The casted PVA membranes were subjected to 6 cycles of freezing and thawing between -20 and 20 °C with a rate of freezing and thawing of 0.1 C min1 and a holding time of 6 h, following the procedure implemented by Wan et al. (2002) **[23]**. The dry membranes were taken off the Petri dish and then washed with distilled water.

2.2.3. Membrane Characterization

2.2.3.1. Determination of hydrolytic degradation

Dried membranes were initially weighed and then immersed into 10 ml of 0.1 M phosphate buffer saline (PBS, pH 7.4) at 37 °C for time intervals. Afterwards, the samples were taken out and gently blotted by soft filter papers to abolish the water over

the surfaces. Finally, the membrane samples were dried under vacuum condition at room temperature and reweighed. All investigations were carried out in six replicates, and the mean and standard deviation (SD) were calculated **[24]**.

2.2.3.2. Light Transmittance

A UV–visible spectrometer (Spectrophotometer model Jasco V-630 - made in Europe) was applied to test the regular light transmittance of PVA membrane, pectin membrane, and PVA/Pectin composite membranes. The wavelength range is 300–800 nm.

2.2.3.3. Fourier Transform Infrared Spectroscopy (FTIR)

In order to find out the modifications of functional groups inside the formulated membranes, Attenuated total reflection Fourier transform infrared spectroscopy (ATR-FT-IR) was used to identify main chemical features of the PVA, Pectin, and PVA/Pectin composite membranes. ATR-FT-IR analysis of the membranes was conducted using FT-IR spectrophotometer (Shimadzu FTIR-8400S, Japan). Examined samples were scanned in the range from 4000 to 400 cm-1 **[25]**.

2.2.3.4. Scanning electron microscopy

Scanning electron microscopy (Scanning electron microscope / FEI Quanta 3D 200i Edx / thermo fisher pathfinder Operated under conditions of high vacuum for acceleration voltage 5.0 ~ 10.0 kv using Secondary Electron detector with working distance 15 ~17 mm) was used to image the fracture surfaces of the PVA, Pectin, and PVA/Pectin composite membranes.

2.2.3.5. Mechanical properties

The tensile mechanical properties of membranes were investigated according to ASTM D-882 standard methods by an electronic universal material-testing machine (SHIMADZU 5 KN, AUTOGRAPH AG_X PLUS, made in Japan) at room temperature. The samples were cut into 50 mm long, 10 mm wide with a crosshead speed of 12.5 mm/min. These properties included the maximum stress and strain to failure. The thickness of membranes was measured using an electronic digital micrometer. All analyses were carried out in triplicate.

3. Results and Discussion

3.1. Determination of hydrolytic degradation

In order to entirely eliminate plastic items, biodegradability is a plastic end-of-life alternative that leverages the microbes existing in a specific environment to mineralize them into CO_2, water, and biomass. The polymer's chemical composition, the additives it contains, and the surrounding environment all play a role in this process. The hydrolysis or enzymatic breakage of the polymer backbone caused by microorganisms that can digest the polymer is typically how plastics biodegrade. With regard to in hydrolytic degradation phenomenon, the degrading property of the membranes used in packaging application (food preservation) is critical to the efficient execution of their biological functions. Thus, the weight loss of PVA membrane, Pectin membrane, and PVA/Pectin membranes was estimated in vitro using phosphate buffer saline (PBS, pH 7.4) at 37 °C for different time points. As shown in Figure (1) PVA membrane doesn't has biodegradability rate as the weight loss was very little (it was only 1.39 % after 6 hours), while the pectin membrane show very speed degradation rate (it degraded completely after only 5 minutes), on the other hand, the PVA/Pectin membranes show biodegradation rate and it increase with the increase of pectin concentrations. After 6 hrs of incubation in PBS, pH 7.4, PVA/Pectin membranes had weight loss of 9.4%, 12.2%, and 15.2% for PVA/Pectin in ratio 2:1, 1:1, and 1:2, respectively, as displayed in Figure (1). These data reveal the good biodegradation features of the prepared PVA/Pectin membranes. From the aforementioned, it is obvious that adding pectin to PVA causes the rate of decomposition to increase, and that rate also increases with an increase in the percentage of pectin. This may be because pectin features transferred to the PVA when they combined to create one film **[26]**.

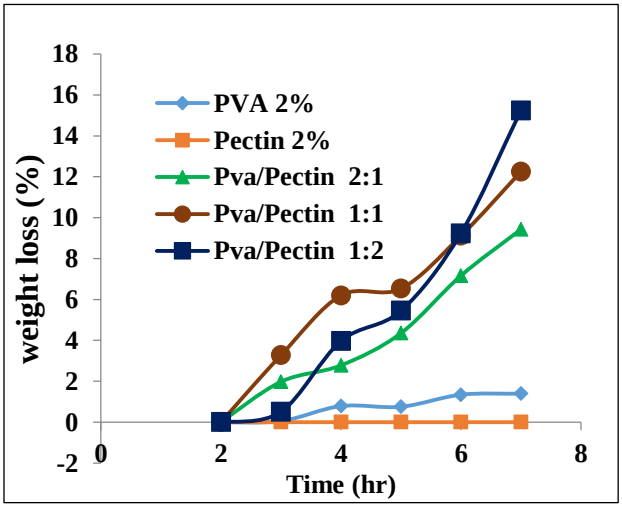

Figure (1): Hydrolytic degradation phenomena for the PVA membrane, Pectin membrane, and PVA/Pectin membranes

3.2. Light Transmittance

Materials transparency is also required in many applications, especially in packaging applications. The light transmittance spectra of the PVA membrane, pectin membrane, and PVA/Pectin

composite membranes were shown in Figure (2). At a visible wavelength of 600 nm, the visible light transmittance of pectin membrane, and PVA/Pectin composite membranes was higher than that of PVA membrane; also pectin membrane and PVA/Pectin composite membranes show a characteristic peak at wavelength of 350 nm **[27]**.

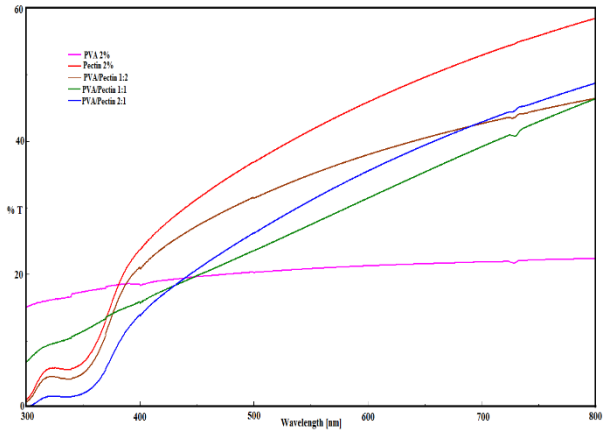

Figure (2): UV–vis spectra of the PVA membrane, pectin membrane, and PVA/pectin composite membranes.

3.3. Fourier Transform Infrared Spectroscopy (FT-IR)

FT-IR spectra of the PVA membrane, pectin membrane, and PVA/Pectin composite membranes were presented in Figure (3). From the spectrum of PVA membrane, the characteristic band at 3250 cm-1 associated with the presence of O-H groups from the intermolecular and intramolecular hydrogen bonds, while the C-H stretching band appeared at 2918 cm-1 from alkyl groups and the peaks between 1732–1657 cm−1 are due to the stretching C=O and C–O from acetate group remaining from PVA, and C-O-C stretching band appeared at 1087 cm-1 **[28, 29]**. While the spectra of pectin membrane shows characteristic peaks of pectin as the broad band at 3346 cm-1 that refers to O-H group, a band at 2923 cm-1 that refers to sp3 C-H, a band at 1735 cm-1 that refers to stretching vibration of C=O, broad band at 1350 to 1440 cm-1 that refers to sp3 CH2 of methylene bridge, band from 1014 cm-1 to 1141 cm-1 that refers to C-O group **[30, 31]**. Form the PVA/Pectin spectra; it is clear that all characteristic beaks of PVA and pectin (O-H, C=H, C=O, C-O, and C-O-C groups) are represented in the PVA/Pectin spectra that mean the good interaction between PVA and pectin through freeze-thawing process.

3.4. Scanning electron microscopy

Scanning electron microscopy analysis was performed in order to characterize the morphological changes of the PVA membrane, pectin membrane, and PVA/Pectin composite membranes. Figure (4) depicts the porosity structures of prepared membranes. It is clear that the surface structure of PVA membrane is somewhat soft with no or few pores on the surface, on the other side; the pectin membrane is rough with many pores. Based on that, the PVA/Pectin membrane surface become rougher and has more pores than the PVA membrane and also less than pectin membrane alone. From the figure, the roughness and pores increase with the increase of pectin concentration and decrease with the increase of PVA concentration. In PVA/Pectin membranes, PVA and pectin are associated through hydrogen bond during freeze-thawing process, leading to the formation of compact structure between the pores **[32, 33]**.

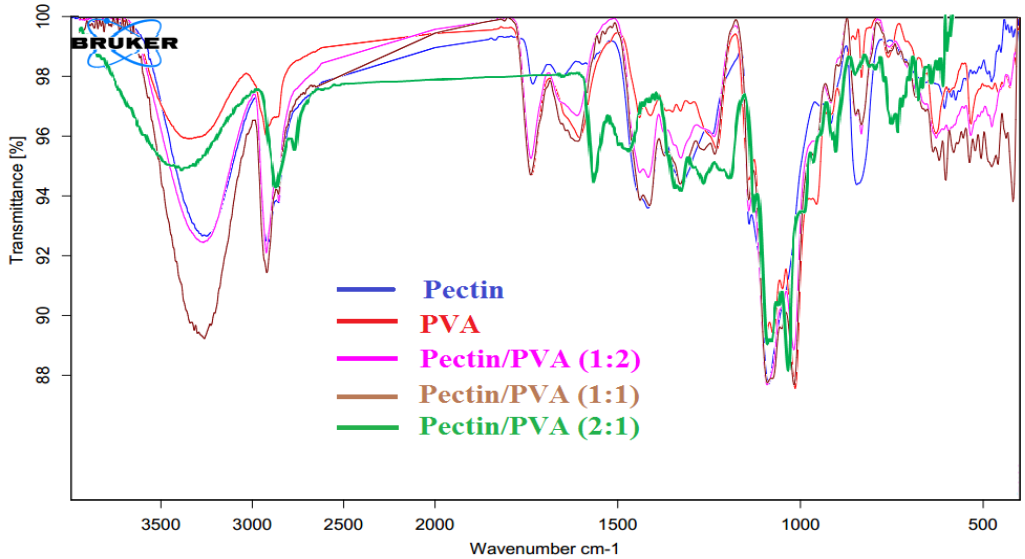

Figure (3): FT-IR of the PVA membrane, pectin membrane, and PVA/pectin composite membranes.

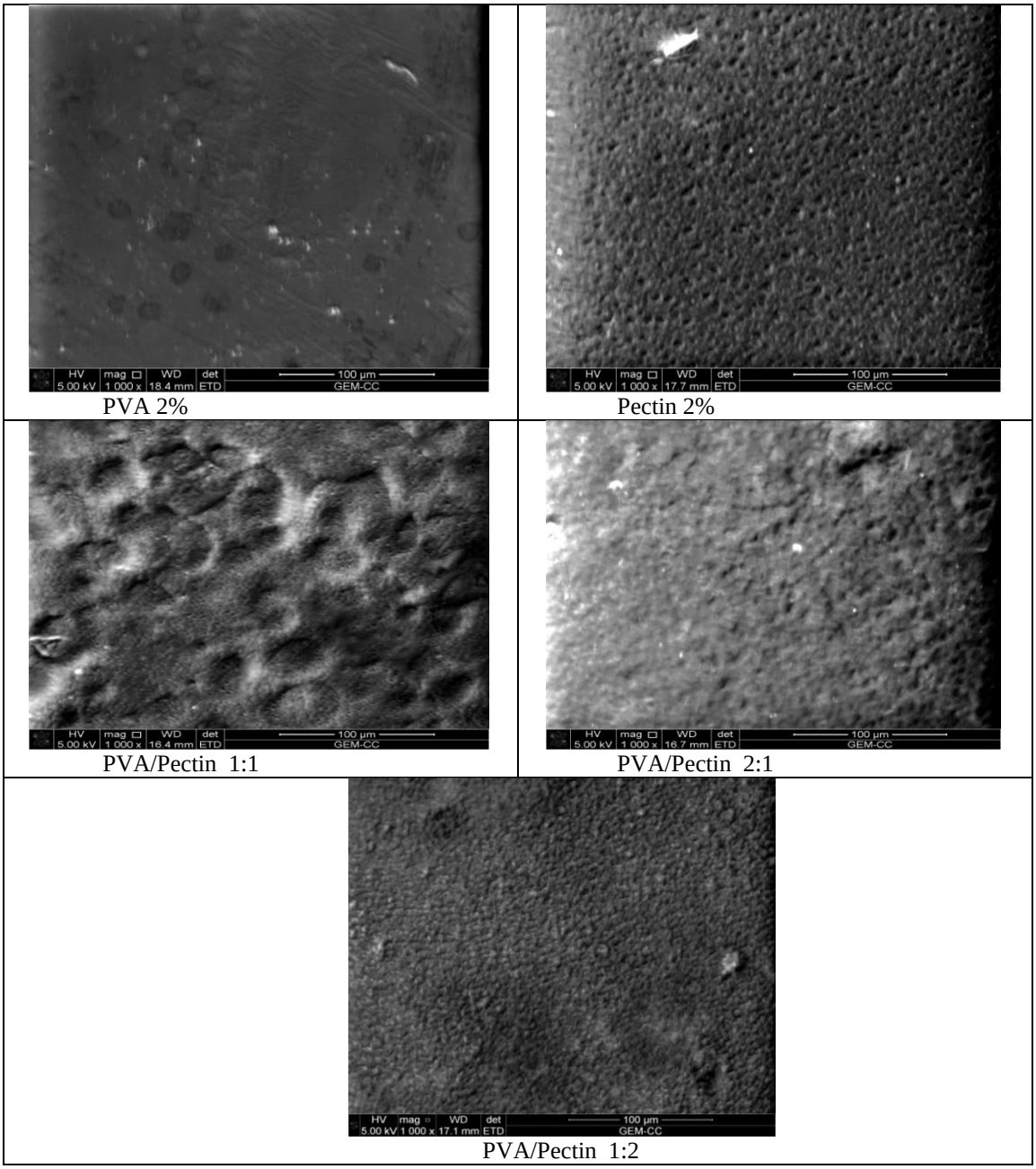

Figure (4): SEM images of the PVA membrane, pectin membrane, and PVA/pectin composite membranes

3.5. Mechanical properties

Mechanical properties are one of the factors that must be taken into consideration when talking about packaging membranes, so that; the mechanical properties of PVA membrane, pectin membrane, and PVA/Pectin composite membranes were measured as shown in Table (1). From the data in table, the average thickness of membrane was 0.09 ± 0.007, 0.06 ± 0.006, 0.08 ± 0.007, 0.08 ± 0.012, 0.08 ± 0.008 for PVA, Pectin , PVA/Pectin 1:1, PVA/Pectin 2:1, PVA/Pectin 1:2, respectively. Also maximum stress increased from 16.2525± 0.789 for PVA membrane and 12.5358± o.814 for pectin membrane to become more than 31 for PVA/Pectin membranes,

this means that the composite membrane can resist more force than the separately materials. Also, the maximum force (which is an important property of materials to determine their mechanical performance, It is the ability of a material to resist tearing due to tension) increased from 14.6272 ± 0.710 for PVA membrane and 7.72148 ± 0.680 for pectin membrane to become 26.156 ± 0.802, 25.27264 ± 1.506, and 48.0045 ± 1.823 for PVA/Pectin 1:1, PVA/Pectin 2:1, PVA/Pectin 1:2, respectively, meaning that there is enhancement of mechanical properties with the increase of pectin concentration in PVA membrane **[34, 35]**.

Table (1): Mechanical parameters of PVA membrane, pectin membrane, and PVA/pectin composite membranes

Film	Thickness (mm)	Max. Stress (N/mm^2)	Max. Strain (%)	Max. Stroke (mm)	Max. Force (N)
PVA	0.09 ± 0.007	16.2525 ± 0.789	25.27383 ± 1.300	12.6361 ± 0.650	14.6272 ± 0.710
Pectin	0.06 ± 0.006	12.5358 ± o.814	1.3918 ± 0.388	0.69592 ± 0.194	7.72148 ± 0.680
PVA/Pectin 1:1	0.08 ± 0.007	31.8621 ± 0.439	2.8404 ± 0.622	1.4202 ± 0.311	26.156 ± 0.802
PVA/Pectin 2:1	0.08 ± 0.012	34.9241 ± 1.697	1.6974 ± 0.460	0.84871 ± 0.230	25.27264 ± 1.506
PVA/Pectin 1:2	0.08 ± 0.008	35.2413 ± 1.444	1.0890 ± 0.164	0.54453 ± 0.082	48.0045 ± 1.823

4. Conclusions

Due to the importance of the environment and its rights, people must preserve it as much as they can, and due to the widespread use of plastic packaging, we must all use biodegradable plastic. So, in this work, we tried to prepare a biodegradable plastic membrane using a PVA/Pectin composite at the levels 1:2, 1:1, and 2:1. The obtained data revealed the possible use of a PVA/Pectin composite at a ratio of 1:1 for the preparation of biodegradable plastic films with improved mechanical and surface properties. Also, the biodegradability rate increased with the increase in pectin concentration in the composite membrane. But the prepared biodegradable films still need further investigations regarding their O2 and water vapor permeability as well as their antimicrobial activity.

Acknowledgements

This work was supported by National Research Center, Food Technology Department by grant No. 12050207.

Data availability

The data supporting this study's findings are available at the request of the corresponding author.

Author contributions

A.Y., A.S., H.A., F.M., A.M, and M.H. contributed to conceptualizing and planning experiments. A.Y., A.S., H.A., and F.M. designed the experiments. M.H.prepared the membrane. A. M and M.H. contributed equally to the interpretation of the results. All authors contributed to the writing of the manuscript.

Conflict of interest

The authors declare that there is no conflict of interest.

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