Celal Bayar University Journal of Science
Volume 16, Issue 4, 2020, p 373-378
Doi:10.18466/cbayarfbe.761144

Celal Bayar University Journal of Science

Polyvinyl Alcohol-Corn Starch-Lemon Peel Biocomposite Films as Potential Food Packaging

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Received: 6 July 2020
Accepted: 19 December 2020
DOI: 10.18466/cbayarfbe.761144

Abstract

In this study, novel biocomposite films were prepared by incorporating lemon peel to a polyvinyl alcohol-starch matrix. The influence of the lemon peel content on the structural, mechanical, and optical properties of biocomposite films was discussed. The FT-IR studies revealed the formation of a strong interaction between the lemon peel and polymer matrix. Blending with lemon peel led to an improvement in the UV-light barrier properties of the polyvinyl alcohol-starch films. The incorporation of 1 wt% lemon peel further enhanced the mechanical properties of the films due to good compatibility and bonding between peel particles and polymer matrix. The results showed that lemon by-product has great potential to be evaluated into added-value products for packaging applications.

Keywords: Biodegradable polymer, *citrus* by-product, composite films, organic filler.

1. Introduction

Food packaging sector plays a significant role in the global polymer market. The most widely used polymer packaging materials are produced from non-renewable petroleum sources. Therefore, sustainable production requirements are increasing day by day due to growing consumption and environmental concerns [1].

Polyvinyl alcohol (PVA) is one of the most used biodegradable polymers in the packaging industry. It has unique properties as film-forming ability, biodegradability, high resistance to oil and solvents, and gas (O_2) barrier properties [2]. However, PVA has a relatively high price [3]. PVA is convenient for blending with natural polymers due to being highly polar and soluble in water [4]. Polyvinyl alcohol and starch blends are stand out due to being more economical, showing excellent compatibility with each other, and enhanced biodegradability features [5]. The reinforcement of the PVA/starch matrix with natural fibers or powders can further improve the mechanical properties of these materials [6]. Therefore, the investigations towards the development of green polymer composites via efficiently evaluating natural lignocellulosic by-products because of their renewable, environmentally-friendly, inexpensive, and non-toxic nature have shown an increasing trend [6]. Previous studies reported the utilization of different food industry by-products as organic fillers or reinforcing agents in packaging systems. Apple pomace [7], banana peel [8], mangosteen rind [9], pomegranate peel [10], tomato pomace [11] has been successfully used to develop biocomposite films. Lemon (*Citrus limon* L.) peel is also an agro-industrial solid residue resulting from the lemon juice processing industry. Annual worldwide lemon production is estimated at over 4 200 000 tons [12]. The peels generate about 50-65 % of the fresh lemon weight [13,14]. Only a small quantity of lemon peel has been used as food additives [15], while most are usually discarded. Lemon peel contains many crude fibers as well as pectin, bioactive compounds, and carbohydrates [16]. The use of lemon peel in packaging appears as an interesting inexpensive alternative.

Currently, there is no report about the incorporation of lemon peel as filler in PVA/starch-based biocomposite films. Additionally, glycerol and citric acid were chosen as plasticizers that are widely used compounds in the food industry. Recently, there is an increasing trend to use citric acid in polymer film preparation which naturally occurs in fruits as the main organic acid [17]. Citric acid can effectively enable esterification reaction with hydroxyl groups of the PVA/starch blend due to having three carboxylic groups and one hydroxyl per monomer. It provides both crosslinking and plasticizing effect to enhance the features of the composite film. Furthermore, the use of glycerol together with citric acid can enhance the film flexibility [18]. In this way, PVA/starch biocomposite films with better functional properties can be developed with the incorporation of citric acid and glycerol.

The principal goal of this work was the evaluation of the structural, mechanical, and optical properties of PVA/starch based packaging materials containing waste lemon peels. The films were characterized by Fourier transform infrared spectroscopy (FT-IR) and Ultraviolet-Visible (UV-VIS) spectroscopy. Mechanical properties of the composite films were evaluated in terms of tensile strength, Young's modulus, and p 2.3.2. elongation. 2.3.3.

2. Materials and Methods

2.1. Materials

Polyvinyl alcohol (average molecular weight; 30.000, 95.4% purity, 87.16% hydrolysis degree, 24.9 r 2.3.4. viscosity) was obtained from Zag Kimya (TURKEY). Corn starch and citric acid were obtained from Güneş Company and Aksu Company (TURKEY), respectively. Glycerol was purchased from Merck (Darmstadt, GERMANY). Lemon peel was purchased from Naturelka (Aydın, TURKEY). The lemon peel was sieved using a steel mesh sieve (<200 µm).

2.2. Preparation of biocomposite films

The five different composite films were prepared by solution casting method according to the ratios given in Table 1. The film solutions were prepared by dissolving 8% (w/v) PVA in distilled water under magnetic stirring (700 rpm) overnight at 90°C. Separately, 2% (w/v) corn starch was gelatinized at 95°C for 15 minutes. Next, gelatinized corn starch was added to the PVA solution and continuously stirred at 70°C for an hour. The solution was then cooled to 50°C followed by the addition of citric acid (10%, w/w of total po 2.3.5. weight) as a plasticizer and stirred for 10 mi 2.3.6. Following the addition of glycerol as a plasticizer at a constant concentration (20%, w/w of total polymer weight), the stirring was continued for another 10 minutes. Then, Tween 80 (50µL) was added as a surfactant to the mixture and stirred for 10 minutes. Finally, the lemon peel with concentrations from 1% to 8% (w/w of total polymer weight) was added to the film solutions. The prepared film solution (35 mL) was cast onto the glass petri dishes (12 cm) and dried in an oven at 40°C for 24 hours. PVA/Corn starch film-forming solution without lemon peel was used to produce a control film.

2.3. Characterization of biocomposite films

2.3.1. Thickness of films

The film thickness was measured by a digital caliper (ABS ASİMETO, Turkey). Measurements were randomly taken at three locations on each specimen. The results were expressed as the mean of measurements.

2.3.2. Fourier-transform infrared spectroscopy

Fourier-transform infrared **(**FT-IR) spectra of the composite films were recorded on a Thermo Scientific Nicolet i550 spectrometer between 4000 and 500 cm^{-1} wavelengths.

2.3.3. UV-VIS spectrophotometric analysis

The optical properties of the PVA/starch/lemon peel films were measured by UV-VIS-NIR Shimadzu UV-3600 device (Shimadzu, Japan). Each film sample cut into square and placed into the spectrophotometer cell. Percent transmittance was taken at the selected wavelength between 200 and 800 nm. Three replicates of each sample were applied.
The transparency and opacity of the films calculated using equation 2.1 and 2.2, respectively as given by Cazon et al. (2020) [19]:

$$\text{Transparency}=\frac{\log(\%T_{600})}{x} \quad \textbf{(2.1)}$$

$$\text{Opacity}=\frac{Abs_{500}}{x} \quad \textbf{(2.2)}$$

where $\%T_{600}$=the percent transmittance, Abs_{500}=the absorbance at 500 nm, and x=film thickness (mm).

2.3.4. Mechanical properties

The mechanical properties of neat PVA/starch and PVA/starch/lemon peel composite films were measured by Universal Tensile Test Machine (Shimadzu-AGS-X, Japan) and the tests were performed according to ASTM D882, rectangular strips were prepared for each film, the samples were clamped and deformed under 1 kN load cell and crossheads peed set at 15 mm/min.

Table 1. The formulations of composite films.

Sample Code	PVA (w/v %)	Starch (w/v %)	Lemon peel (wt.% of total polymer)	Glycerol (wt.% of total polymer)	Citric acid (wt.% of total polymer)
L0	8	2	0	20	10
L1	8	2	1	20	10
L2	8	2	2	20	10
L3	8	2	4	20	10
L4	8	2	8	20	10

3. Results and Discussion

3.1. Film thickness

Film thickness is an important parameter that affects the barrier, optical, and mechanical properties of films [4]. The thickness of films varied from 0.29 to 0.35 mm (Table 2). In general, the films containing 4 wt. % and 8 wt. % lemon peel were slightly thicker than the other samples. The increment of film thickness with the incorporation of lemon peel could be because of the increase in solid content in films. A similar result was reported by Gaikwad et al. (2016) [7] that the addition of apple pomace increased the thickness of polyvinyl alcohol bio-composite films. The thicknesses of the prepared PVA/Corn starch/Lemon peel composite films were homogeneous indicating the good compatibility of the selected matrix materials.

Table 2. Thicknesses of PVA/Corn starch/Lemon peel biocomposite films.

Sample	Thickness (mm)
L0	0.29±0.01
L1	0.29±0.01
L2	0.29±0.01
L3	0.30±0.01
L4	0.35±0.02

3.2. FT-IR analysis of the biocomposite films

FT-IR spectra of the composite films are shown in Figure 1. A wide absorption centered around 3280 cm^{-1} represents the stretching vibration of O–H as hydrogen-bonded hydroxyl groups related to the absorbed moisture in the films. The presence of asymmetric CH_2 methyl group stretch/stretching vibration is observed at 2938 cm^{-1} [20]. The band at 1712 cm^{-1} is the characteristic peak of the C=O stretching vibration peak due to the ester bond and carboxyl groups in citric acid [20]. The peak at 1420 cm^{-1} is attributed to the CH_2 bending with deformation [21]. The mixed bending modes of CH and OH groups and attributed to the associated alcohols were observed at 1373 cm^{-1} [22]. The deformation vibration of the CO groups occurred at 1330 cm^{-1} [21]. The peak at 1241 cm^{-1} due to the wagging vibration of CH groups [22]. The stretching vibration of CO bond in C-O-H was observed at 1088 cm^{-1} [23]. The peak at 1024 cm^{-1} assigned to C–O stretching of the glucose ring [24]. The rocking vibration of CH_2 groups appeared at 919 cm^{-1} [22]. The band occurs at 844 cm^{-1} related to C-C groups stretching vibrations and out of plane bending vibration of OH groups [22].

The addition of lemon peel slightly weakened the intensities of 2938, 1342, 1024, 944 and, 919 cm^{-1} absorption bands. The peak at 919 and 842 cm^{-1} in the pure PVA-starch film also shifted to 922 and 845 cm^{-1} in the PVA/starch/lemon peel composite films, respectively. This was attributed to the formation of new hydrogen bonds between the PVA, starch, and lemon peel.

3.3. Optical analysis of the biocomposite films

The optical characteristics including color, transparency, opacity, and desired light-blocking ability of film materials are significant quality parameters [25]. These factors are important in view of consumer's acceptance and expanding the shelf life of packed products [19].

The light barrier properties of the biocomposite films by the percentage transmittance in the UV region (200-400 nm), and the VIS region (400-800 nm) are given in Figure 3. It is clear that the transmittance decreased with increasing lemon peel concentration in the polymer matrix in the UV-A (320-400 nm), UV-B (280-320 nm), and UV-C (190-280 nm) region, as shown in Figure 3. The lemon peel incorporated films were slightly yellowish. The L4 sample (8 wt. % lemon peel) significantly lowered the transmittance of the

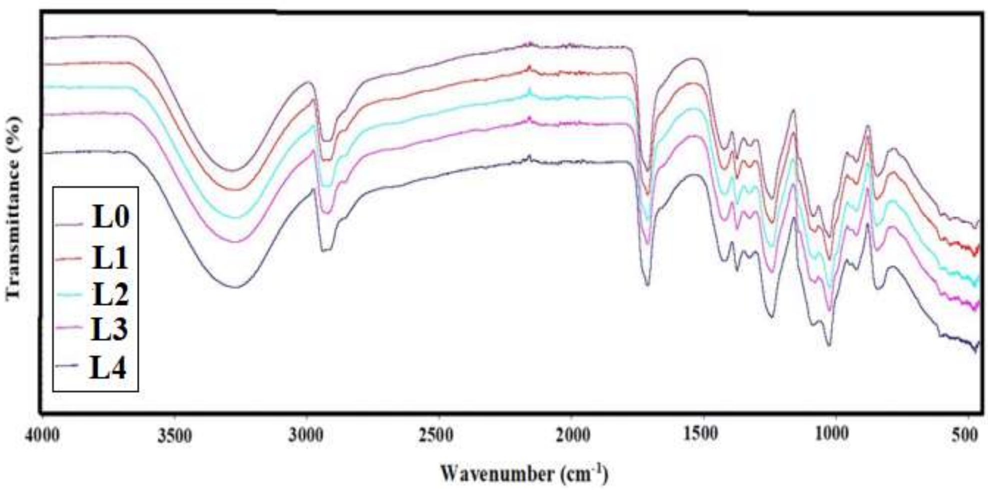

Figure 1. FT-IR spectra of PVA/Corn starch/Lemon peel biocomposite films

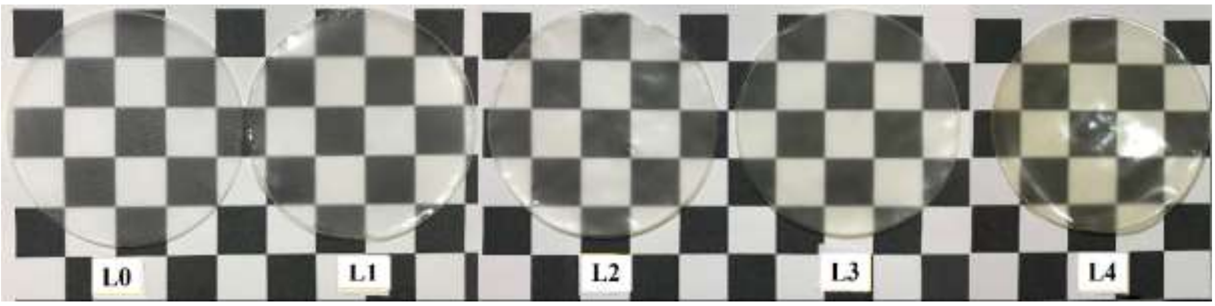

Figure 2. Visual appearances of PVA/Corn starch films containing different lemon peel contents.

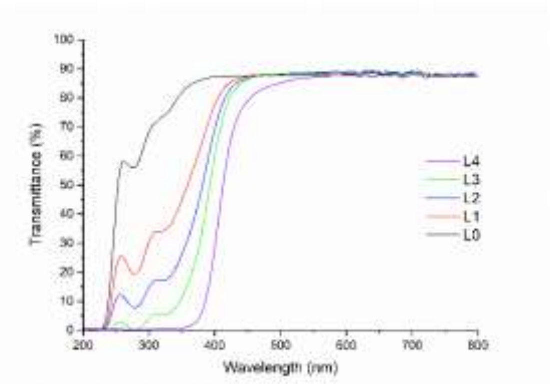

Figure 3. UV–VIS spectra profiles of the biocomposite films.

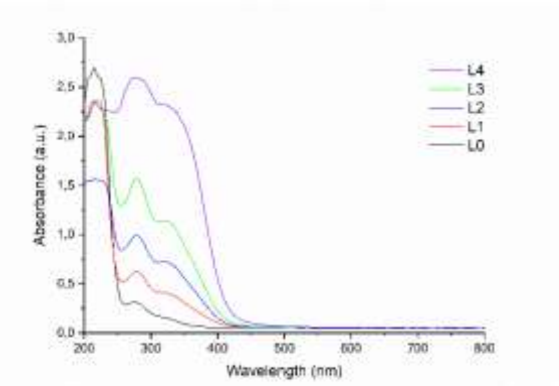

Figure 4. Absorbance spectra of PVA/Corn starch/Lemon peel biocomposite films.

PVA/starch film which was also apparent in the digital photographs of the composite films (Figure 2). The results showed that produced films would help to prohibit oxidative deterioration of packed food due to exposure to visible light. This result was supported by the study of Hanani et al. (2019) [10] who reported the effect of pomegranate (Punicagranatum L.) peel powder on the color of fish gelatin films.

The light absorbance of composite films is shown in Figure 4. The biocomposite films exhibited a strong absorption peak around 280 nm. The absorbance values of films were increased with the increase of the lemon peel content in UV-A (320-400 nm) and UV-B (280-320 nm) region. According to the absorbance results, less light could pass through the lemon peel incorporated biocomposite films.

Table 3 presents the transparency and opacity results of the lemon peel incorporated biocomposite films with the effect of the blend ratio.

Table 3. Transparency and opacity values of the biocomposite films.

Sample	Transparency	Opacity
L0	6.71±0.01	0.18±0.01
L1	6.71±0.01	0.18±0.01
L2	6.71±0.01	0.18±0.01
L3	6.54±0.02	0.20±0.01
L4	5.55±0.01	0.19±0.01

The transparency and opacity results of the films ranged from 5.55 to 6.71 and 0.18 to 0.20, respectively. The 4 wt. % and 8 wt. % lemon peel incorporated PVA/starch films had lower transparency and higher opacity when compared to other films. Therefore, it can be concluded that the addition of the lemon peel at these concentrations into the PVA/starch matrix significantly enhanced the light barrier properties.

3.4. Mechanical analysis of the composite films

Mechanical properties are important parameters to obtain information about the stiffness, brittleness, and ductility of the biocomposites [26]. Tensile testing was conducted to examine the effect of blending with lemon peel on the mechanical properties of PVA/starch-based biocomposite films. The results are presented in Table 4 and Figure 5. The tensile strength of pure PVA/starch film (L0) increased from 24.35 ± 1.2 to 25.11 ± 1.3 MPa with the incorporation of 1 wt% lemon peel (L1). However, the tensile strengths of the films were decreased with further increment of lemon peel concentration. This might be attributed to the slight aggregation of lemon peel at higher concentrations and voids formation within the polymer matrix resulted in cracks at the biocomposite interface [27]. It could be due to the availability of surface hydroxyl groups of this lignocellulosic waste which tended to agglomerate easily by forming hydrogen bonds. In further studies, the surface modification of lemon peel should be evaluated to enhance dispersibility in the polymer

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matrix and interfacial interaction of the matrix/filler at higher concentrations [28]. Similar to the tensile strength result, the elongation at break of pure PVA/starch film was 260.18 ± 13.00 % and has been increased to 275.19 ± 13.8 % with blending 1 wt. % lemon peel. The good dispersion of lemon peels and strong interfacial interaction might improve the ability to deliver external stress transfer from the PVA/starch matrix to lemon peel particles which leads to enhancement of both elongation at break and tensile strength [29]. With the increase of lemon peel concentration in the range of 2-8 wt. % in the polymer matrix, the elongation at break of the films was decreased because of the improvement in the stiffness of three-dimensional network by incorporation of filler [26]. In another study, the tensile strength and % elongation of PVA/starch/barley husk films were also reduced with the increase in barley husk content from 1 to 2% [30].

When compared with pure PVA/starch film, the biocomposite with lower lemon peel contents (1 and 2 wt. %) indicated a slightly decreased Young's modulus.

Table 4. Mechanical properties of biocomposite films.

Sample	Tensile strength (MPa)	Elongation at break (%)	Young'smodulus (MPa)
L0	24.35 ± 1.2	260.18± 13.0	56.33 ± 2.8
L1	25.11 ± 1.3	275.19 ± 13.8	52.70 ± 2.6
L2	22.52 ± 1.1	215.85± 10.8	51.26 ± 2.6
L3	20.03 ± 0.9	199.91 ± 10.0	59.46± 2.9
L4	17.72 ± 0.9	211.78 ± 10.6	71.82 ± 3.6

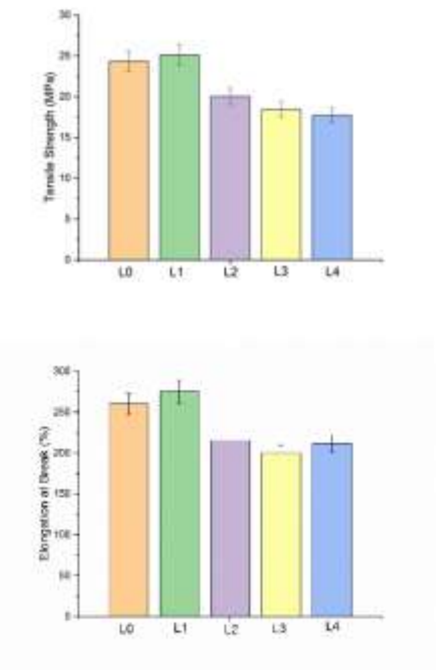

Figure 5. Tensile strength and elongation at break of biocomposite films.

However, when lemon peel content increased from 2 to 4 wt. %, Young's modulus was increased from 51.26 ± 2.6 to 59.46 ± 2.9 MPa. The highest Young's modulus was observed for the L4 sample which was containing the highest amount of lemon peel (8 wt. %).

4. Conclusion

A new approach was developed to add value to lemon peel by a cost-effective way to obtain functional packaging materials. A series of PVA/corn starch/lemon peel biocomposite films were successfully prepared using a solution blending method in the presence of glycerol and citric acid. UV-VIS results showed that the optical barrier properties of lemon peel incorporated PVA/starch films against UV-radiation enhanced with the increase of the lemon peel concentration. The mechanical properties of the films improved at the appropriate incorporation of lemon peel. Reduction in tensile strength and elongation at break were observed with the addition of lemon peel in the range of 2-8 wt. %. The lemon peel incorporated PVA/corn starch films with desirable performance can be taken into consideration as a sustainable and environmentally-friendly material alternative to the traditional packaging applications.

Author's Contributions

Pınar Terzioğlu: Drafted and wrote the manuscript, performed the experiment, supervised the characterization progress and interpreted the results.

Fatma Nur Parın: Performed the characterization studies and helped in manuscript preparation.

Ethics

There are no ethical issues after the publication of this manuscript.

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