



Pectin-polyvinylpyrrolidone films: A sustainable approach to the development of biobased packaging materials



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ABSTRACT

In this work, the new biodegradable blend films composed of natural polymer pectin and synthetic polymer polyvinylpyrrolidone were obtained through casting process. The concentration of glycerol in all formulations was constant (2% v/v), while the amount of polyvinylpyrrolidone varied from 20 to 60% by mass of pectin. The molecular interactions between two components were elucidated by FTIR-ATR spectroscopy, while functional properties of these films were carried out by determination of puncture resistance, tensile strength, thermal stability, water vapor barrier, wettability and printability. The blending of pectin with PVP led to an improvement of mechanical resistance, barrier properties and hydrophobicity of these films for 57%, 58 and 24%, respectively, remaining adequate thermal stability. Since pectin and PVP are nontoxic, food-grade and biodegradable materials, this research present an eco-sustainable approach to provide films with satisfied physical properties for use as packaging material of non-food products, such as water-treatment products (detergents, biocides, agrochemical compounds).

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1. Introduction

Packaging is one of the largest market segments for the polymer industry. Petroleum-derived materials such as polyolefin and polyesters have been increasingly used as packaging materials due to low cost and favorable mechanical, gas barrier and heat sealing characteristics. However, the petroleum-derived materials are resistant to biodegradation and their accumulation in landfills and marine affects human health, eco and aqua system. Environmental awareness among a growing population of consumers is demand for sustainability and the minimization of harmful effect of synthetic packages on the environment. Thus, development and implementation of biobased packages, as alternative environmental-friendly materials to the conventional fossil-based materials, have been highlighted. The biobased materials are highly beneficial due to their derivation from renewable feedstock (plants, trees, algae, bacteria), which offers a range of advantages:

lower carbon footprint, higher energy-efficient process and reduce in raw material use.

The aim of this work is to use apple pectin as a matrix for preparation of biobased bags intended for packaging of various products. The choice is related to the fact that pectins are renewable, nontoxic and biodegradable resource present in fruits, mostly apples and citrus. Pectins are family of natural polysaccharides comprising chains of α -(1–4)-linked D-galacturonic acid [1]. In nature, around 80% of carboxylic groups of galacturonic acid are methylesterified. The ratio of esterified to non-esterified galacturonic acid determines the behavior of pectin in solutions, influencing the properties of pectin-based materials, as well [2]. Its unique property to gel at acidic conditions or in presence of divalent cations, allows the widen use in food and beverage technology as colloidal stabilizer, thickening and gelling agent [3]. Furthermore, pectin has been increasingly investigated as a carrier material in colon specific drug delivery system [4–7], as edible film to prolong shelf-life of food products [8,9] and in biomedicine [10,11].

The neat pectin films are too brittle and easy hand-breakable, which prevent their wide application. In order to improve its mechanical properties, pectin has been blended with various polymers [12–14]. In this work, polyvinylpyrrolidone (PVP) was chosen as blending component, due to approved safety from U.S. Food and

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Drug Administration. PVP is a nontoxic and biocompatible synthetic polymer with excellent film-forming ability and have widespread use in medicine, pharmaceuticals, cosmetics, foods, printing inks, detergents, adhesives, paints, coatings and plastics production. The films formed by blending of two polymers may result in modified physical and mechanical properties compared to films made of the neat pectin. In addition, since PVP is easily obtained and has low production cost, the blending with pectin may improve the cost-performance ratio of the resulting films. The objective of this research was to evaluate and compare the mechanical, thermal and barrier properties and functional performance of pectin films prepared with various levels of PVP for potential application in packaging industry.

2. Materials and methods

Apple pectin having an average molecular weight of 75,000 and degree of esterification in a range of 70–75% was purchased Sigma Aldrich (Switzerland) and used as received. PVP of molecular weight 25,000 was purchased from Sigma Aldrich, as well. Glycerol used as a plasticization agent was obtained from Merck Co., (Darmstadt, Germany).

2.1. Preparation of pectin/PVP films

The 2 wt% solution of pectin in distilled water was prepared at room temperature. In order to enhance the elasticity of films, glycerol was added (2% v/v) in pectin solution and this solution was further used to dissolve PVP. PVP was added to the pectin/glycerol solutions at different levels (20, 40 and 60 wt% by mass of pectin) and the samples were marked as pectin/PVP 20, pectin/PVP 40 and pectin/PVP 60. Pectin film plasticized with glycerol, without any concentration of PVP, was used as control sample. The film-forming solutions were spread onto a Plexiglas plate by spreading device 30 cm width, which allowed controlling the thickness of final-forming material. The films were dried at room temperature. The thickness of films was approximately 20 μm . Final step was sealing of films into bags.

2.2. Characterization of pectin/PVP films

2.2.1. FTIR-ATR spectroscopy

The interactions between pectin and PVP were investigated by FTIR-ATR spectroscopy. The spectra were recorded on Perkin Elmer Spectrum 100 spectrometer, equipped with a Universal ATR diamond crystal sampling accessory. A series of 16 scans were collected for each sample over the 4000–400 cm^{-1} spectral range with a 4 cm^{-1} resolution. Spectra were analyzed with the Omnic 8.1 software.

2.2.2. Tensile and puncture test

Instron M 1185 universal testing machine was used to obtain the stress-strain curves for the tensile tests of the films. Crosshead speed was 2 mm min^{-1} for all testing samples. From these curves, Young modulus (E, MPa), tensile strength (TS, MPa) and elongation at break (ϵ , %) of the films were evaluated. Prior to testing, the films were conditioned in an environmental chamber at 25 $^{\circ}\text{C}$ and 50% relative humidity (RH) for 24 h. The measurements were done at room temperature and six samples (mold $4.92 \times 26.73 \times 0.05$ mm) for each formulation were tested. The obtained values of the Young's modulus were within $\pm 10\%$, while the stress and the elongation at break fluctuated in the range of $\pm 15\%$.

In order to predict the behavior of material under sharp stress, the puncture tests were performed. For each film formulation, 5 round samples with diameter of 34.9 mm were taken. The samples

were fixed in a measurement cell with a round hole in its middle. A spherical probe of 3.2 mm in diameter was moved down, perpendicularly to the film surface, at a constant speed of 2 mm/min , until the probe passed through the film. The puncture force was determined directly from the curves of strength versus time, energy at break from the peak area and puncture deformation (D, %) was calculated according to the following equation:

$$D = \frac{\sqrt{d^2 + l_0^2} - l_0}{l_0} \times 100 \quad (1)$$

where d is displacement of spherical probe in the puncture moment (mm) and l_0 is initial radius of film (mm). The puncture deformation was calculated considering that stress was perfectly distributed along the film at breaking point, as it is shown in Fig. 1. For each composition, 6 samples were tested. The obtained values of the force to break and energy to break were within $\pm 10\%$, while the deformation values were within $\pm 15\%$.

2.2.3. Thermal analysis

Mettler-Toledo TG-SDTA 851 thermobalance equipped with a differential thermal analyzer instrument was utilized to measure the thermal weight loss (TG) and derivate (DTG) of the pectin/PVP films in the temperature range between 25 $^{\circ}\text{C}$ and 600 $^{\circ}\text{C}$, with a heating rate of 10 $^{\circ}\text{C min}^{-1}$ under nitrogen stream with the flow rate of 30 mL min^{-1} .

Differential scanning calorimetry (DSC) measurements were performed on a DSC Q1000-TA Instrument. All samples were firstly equilibrated at 20 $^{\circ}\text{C}$ and heated to 110 $^{\circ}\text{C}$ at 10 $^{\circ}\text{C min}^{-1}$, cooled to 20 $^{\circ}\text{C}$ and reheated to 200 $^{\circ}\text{C}$ at 10 $^{\circ}\text{C min}^{-1}$. All analyses were carried out under nitrogen atmosphere with the flow rate of 50 mL min^{-1} . The data were collected from the second heating run.

2.2.4. Water vapor permeability and contact angle

Water vapor permeability (WVP) was determined according to the ASTM E96-95 gravimetric method (1995). Film samples (six per formulation) with exposed area of 6.154 cm^2 were sealed over a circular opening of permeation cells filled with distilled water. Afterword, the permeability cups were placed in a pre-equilibrated chamber with constant temperature at 25 $^{\circ}\text{C}$ and constant relative humidity of 50%. The cups were weighed periodically after steady state was reached. Water vapor permeability rate (WVTR) ($\text{g (h m}^2)^{-1}$) was determined by slope (of the weight loss vs. time plot) divided with the exposed film area. Water vapor permeability (WVP) was calculating according to the following equation:

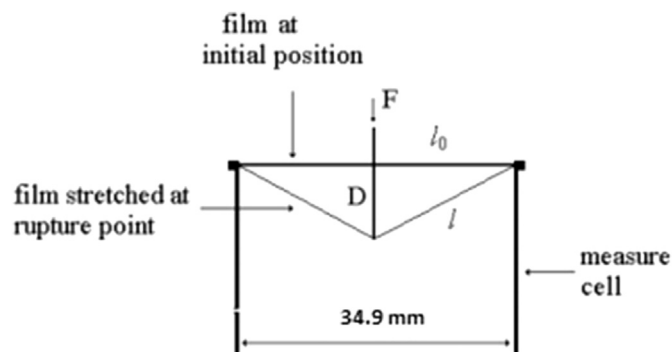


Figure 1. Puncture test scheme.

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