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Development of ecofriendly bionanocomposite: Whey protein isolate/pullulan films with nano-SiO_2

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ABSTRACT

During the past decade, the limitation of petroleum based polymers, the high price of oil, and the environmental concern were attracted the attention of researchers to develop biobased polymers. The composition of different biopolymers and the reinforcement with nano filler are common methods to improve the drawbacks of biopolymers. In this study whey protein isolate/pullulan (WPI/PUL) films contain 1%, 3%, and 5% (w/w) nano-SiO_2 (NS) were prepared by a casting method. Tensile strength of nanocomposite films increased after increasing NS content, but elongation at break decreased, simultaneously. Water absorption, moisture content, solubility in water improved in the wake of increasing NS content because NS increase the cohesiveness of the polymer matrix and improved the barrier and water resistance properties of the films. water vapor permeability of film specimens decreased by increasing NS content. Uniform distribution of NS into polymer matrix was confirmed by scanning electron microscopy (SEM). XRD pattern and thermal analysis revealed increasing crystallinity and increasing T_g of film specimens with increasing NS content, respectively. According to our result WPI/PUL/NS films possess potential to be used as environment friendly packaging films to improve shelf life of food and can be used as promising alternative to petroleum based packaging films.

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1. Introduction

Development of biopolymers has been increased over the past decade. This development is attributed to environmental concerns, the limitation of petroleum resources, and the oil price [1,2]. Although the complete substitution of petroleum-based polymers is impossible, but partial substitution of petroleum-based polymers by biopolymers is conceivable. The sensitivity of the biopolymers to water, the incommensurate barrier properties, and the mechanical weakness of the biopolymers are the main reasons for paying attention to biopolymer modification. Combination of polysaccharides-proteins [3,4], adding plasticizers [5,6], development of bionanocomposites [1], and γ-irradiation [7] are the common modification methods of biopolymers.

Biopolymers are considered as promising materials because they are abundant cost effective end products. Whey protein isolate (WPI) is one of the abundant by-products in dairy factories and it causes environmental obstacles. WPI contains highly bio available essential amino acids [8]. WPI based biopolymers form a transparent and flexible film with fairly good functional properties [9,10].

Pullulan (PUL) is a microbial polysaccharide which is produces by *Aureobasidium pullulans* in starch and sugar cultures. PUL is utilized in low calorie foods, adhesive binders, thickeners, and encapsulating agents [11]. Recently, PUL has attracted an interest to be used as filmogenic material in food packaging. PUL film is transparent, low oil, and oxygen permeable [12,13].

The common disadvantages of most of biopolymers are sensitivity to water, poor mechanical properties, and water vapor barrier properties [1,14]. One of the recommended modification methods is blending polysaccharides and proteins [3,15]. Furthermore, mixing the biopolymer with nano particles has been the center of attention as a modification method of biopolymers [1,3,16]. High surface area and homogenous dispersion of the nano particles in polymer matrix bring about outstanding properties compared to conventional micro composites [17].

Nano-SiO_2 (NS), amorphous powder with tridimensional structure, was produced from silicon oxide with SiO_{2-x} formulation that x ranges from 0.4 to 0.8 [18]. The striking feature of NS i.e. small size,

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large specific surface area, high surface energy, unsaturated chemical bonds, and hydroxyl group on the surface bring about easy dispersion of NS in polymer matrix and attention to NS as nano filler in nanocomposites [18,19,20]. In many parts of the world, SiO_2 is the major constituent of sand [21]. Almost all of nanoparticles show some side effects on humane body. Nevertheless, there is less concern about eco-pollution of NS [21].

The main purpose of the current study was to improve water sensibility, permeability, visual drawback, and mechanical properties of WPI and PUL by composition. Furthermore, the functional property of WPI/PUL films was intensified with NS. The characterization of WPI/PUL/NS films was investigated by XRD, SEM, and Differential scanning calorimetry (DSC).

2. Materials and methods

2.1. Material

WPI contain over 85% protein was purchased from (Arla Food Ingredient, Denmark), PUL with molecular weight 10,000–14,000 was provided from Hayashibara (Hayashibara Co., Ltd., Japan), and NS was purchased from TECNAN (tecnologia Navarra de Nanoproductos S.L, Spain). The analytical grade chemicals, including sodium chloride (NaCl), calcium chloride ($CaCl_2$) and calcium nitrite ($Ca(NO_2)_2$), were purchased from Merck (Merck Co., Germany).

2.2. Film preparation

5 g WPI was dissolved in 100 ml distilled water, heated at 90 °C for 30 min, and stirred with magnetic stirrer. Then 5 g PUL was dissolved in 100 ml distilled water and mixed with WPI solution (1:1) w/w. After wards 30% w/w (dry base) glycerol was added as plasticizer into the solution. Three NS suspensions were prepared by ultrasonic bath at ambient temperature for 1 h so that the concentration of NS in WPI/PUL solution was 1%, 3% and 5% w/w (dry base). The solution was subsequently casted onto Teflon plates (15 cm diameter) and dried for 48 h at 25 °C in oven and room relative humidity. All the film specimens were conditioned inside desiccators containing a saturated calcium nitrite solution to ensure a relative humidity of 55% at 25 ± 1 °C for 48 h. The conditioned films were used for other analysis. All samples were prepared in triplicates.

2.3. Thickness

The thickness of specimens was measured by a hand-held micrometer with an accuracy of 0.01 mm at least in 8 random positions for each film.

2.4. Mechanical properties

Mechanical properties of films were determined by material testing machine (Testometric – M350 – 10CT, England) according to ASTM D882 [22]. Initial grips separation and cross head speed were set to 50 mm and 10 mm/min, respectively. Tensile strength (TS) and elongation at break (EB) were calculated by Eqs. (1) and (2), respectively.

$$TS = \frac{F_{Max}}{A_{min}} \quad (1)$$

$$EB = \frac{L_{Max}}{L_0} \times 100 \quad (2)$$

where F_{max} is maximum load, A_{min} is minimum cross section area, L_{max} is extension at the moment of rupture, and L_0 is initial length of the specimen.

2.5. Moisture content

Moisture content (MC) of the film specimens were determined by the weight of the samples before (m_1) and after (m_2) oven drying at 105 °C (Eq. (3)).

$$MC = \frac{m_1 - m_2}{m_2} \times 100 \quad (3)$$

2.6. Solubility in water

Solubility in water (SW) was defined as the ratio of the water-soluble dry matter of film that is dissolved after immersion in distilled water. SW of the film specimens was measured according to the method of Shahabi-Ghahfarrokhi et al. [1]. The 20 mm×20 mm film specimens were dried at 105 °C, and weighed (m_3). Then dried samples were immersed into 50 ml of distilled water for 6 h. The remaining films were dried at 105 °C and weighed (m_4). SW was calculated by Eq. (4).

$$SW = \frac{m_3 - m_4}{m_4} \times 100 \quad (4)$$

2.7. Moisture absorption

Moisture absorption (MA) was measured according to the method of Almasi et al. [23]. In brief, the dried sheets of 20×20 mm^2 were first conditioned at 0% RH (prepared by dried calcium sulphate) for 24 h. After weighing (m_5), they were conditioned in a desiccators containing a saturated calcium-nitrite solution at 25 °C to ensure a relative humidity of 55%. The specimen was weighed at desired intervals until an equilibrium state was reached (m_6). The moisture absorption of the specimen was calculated via Eq. (5).

$$MA = \frac{m_6 - m_5}{m_5} \times 100 \quad (5)$$

2.8. Water vapor permeability

Water vapor permeability (WVP) was determined gravimetrically according to ASTM method E96-95 [24]. The films were fixed on the top of glass cup containing of dehydrated calcium chloride and sealed with paraffin. Cups were placed in a desiccators containing saturated solution of sodium chloride, and weighed with 1 hour interval for 24 h at 25 °C. The slope (S) of time–weight curve was calculated by linear regression model ($R^2 \geq 0.986$). Water vapor transition rate (WVTR) and WVP were calculated by Eqs. (6) and (7), respectively.

$$WVTR = \frac{S}{A} \quad (6)$$

$$WVP = \frac{WVTR \times X}{\Delta P} \quad (7)$$

where A is the effective film area (m^2), X is the average film thickness (m) and ΔP is the driving force (1753.55 Pa).

2.9. Transparency

Transparency of the films was determined using a UV–vis spectrophotometer (Model 8451A, Hewlett-Packard Co., USA) and measured by percentage of transmittance at 600 nm [25].

2.10. Color

Color of the film specimens was measured by Minolta colorimeter (Minolta CR 300 Series, Minolta Camera Co., Ltd., Osaka, Japan). White standard color plate ($L^* = 96.9$, $a^* = -0.33$, $b^* = 0.16$) was used as a background of all the samples. The samples' color was reported

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