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Fabrication and characterization of chitosan-titanium dioxide nanocomposite film as ethylene scavenging and antimicrobial active food packaging

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ABSTRACT

Active packaging from nanocomposite of chitosan and nanosized titanium dioxide (TiO₂) to be used as an ethylene scavenging and antimicrobial film was developed. Chitosan films containing different TiO₂ concentrations (0, 0.25, 0.5, 1 and 2% w/w) were fabricated and characterized for structural, mechanical, optical and barrier properties. The scanning electron microscope images showed that TiO₂ nanoparticles evenly distributed in the chitosan film matrix. However, the spontaneous agglomeration of the TiO₂ was observed at high TiO₂ concentrations. The X–Ray diffraction patterns presented that crystallinity of TiO₂ in the nanocomposite films increased with TiO₂ concentration. The Fourier transform infrared spectra indicated that there were hydrogen bonding and O-Ti-O bonding between TiO₂ and chitosan, which affected the tensile strength, elongation at break, and water vapor barrier properties of the films. The chitosan-TiO₂ nanocomposites exhibited ethylene photodegradation which increased with increasing TiO₂ concentration. Based on tensile strength, water barrier and ethylene photocatalytic degradation properties, chitosan film containing 1% TiO₂ (CT1) was found to be optimal and hence was selected for antimicrobial evaluation. The results suggested that the CT1 exhibited antimicrobial activity against Gram-positive (*Staphylococus aureus*) and Gram-negative (*Escherichia coli, Salmonella* Typhimurium, and *Pseudomonas aeruginosa*) bacteria and fungi (*Aspergillus* and *Penicillium*).

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1. Introduction

Postharvest losses and quality deterioration of fresh produce are generally caused by ripening process induced by ethylene and contamination of spoilage and/or pathogenic microorganisms (de Chiara, Pal, Licciulli, Amodio, & Colelli, 2015; Maneerat et al., 2003). Active packaging which can prevent the negative effects of ethylene and inhibit microbial growth is desirable to extend shelf life of fresh produce and might be a good alternative to the use of chemical treatments and disinfectants (Kaewklin, Siripatrawan, Suwanagul, & Lee, 2018; Maneerat et al., 2003).

Titanium dioxide (TiO₂) is an efficient photocatalyst and is useful as an ethylene scavenger and an antimicrobial agent (Lin et al., 2015; Zhang et al., 2017). Titanium dioxide is considered as

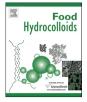
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an attractive photocatalyst because it is chemically stable, nontoxic, inexpensive and Generally Recognized as Safe (GRAS) (Lin et al., 2015). When exposed to ultraviolet (UV) light, TiO₂ exhibits ethylene photodegradation by generating strong reactive oxygen species (ROS) including hydroxyl radicals (OH•) and superoxide ions (O₂⁻) on its surface which further react with ethylene and/or polyunsaturated phospholipid in the cell membrane of microorganisms (Hussain, Bensaid, Geobaldo, Saracco, & Russo, 2011; Lin et al., 2015; de Chiara et al., 2015).

The nanosized TiO_2 particles, due to their high surface area to volume ratio, have higher photocatalytic activity than those in bulk form. The performance of TiO_2 nanoparticles is enhanced because the nanosized TiO_2 owes greater band gap energy when exposed to UV light and generates electron—hole pairs on its surface (Lian, Zhang, & Zhao, 2016; Maneerat et al., 2003; Onda et al., 2005). However, the efficiency of the nanosized TiO_2 is reduced by its tendency to agglomerate (Lin et al., 2015; Qian, Su, & Tan, 2011). The spontaneous agglomeration of TiO_2 nanoparticles results in a decrease in specific surface area, and consequently lessening their







photocatalytic activity (Li et al., 2010; Lin et al., 2015). However, Al-Sagheer and Merchant (2011) and Qian et al. (2011) have reported that interactions between nanosized TiO_2 and organic polymers can help to reduce the spontaneous agglomeration of the inorganic TiO_2 in the polymer matrix which can also have positive effect on the properties of the composite films.

A number of biopolymeric materials have recently been employed to incorporate with nanosized TiO₂ to enhance the functional properties of the biopolymers and to exhibit the particular photocatalytic activity of TiO₂ (Kavitha, Sutha, Prabhu, Rajendran, & Jayakumar, 2013; Lian et al., 2016; Qian et al., 2011). Among them, chitosan, a linear polysaccharide of randomly distributed β -(1–4)-linked p-glucosamine and N-acetyl-p-glucosamine, has gained most interest for active food packaging applications due to its good film-forming ability and intrinsic antimicrobial and antioxidant properties (Cazón, Velazquez, Ramírez, & Vázquez, 2017; Crizel et al., 2018; Shankar, Wang, & Rhim, 2017; Yuceer & Caner, 2014).

Nanocomposites prepared from TiO_2 and chitosan or other biopolymers have been mostly studied for their antimicrobial applications (Bodaghi et al., 2012; Lin et al., 2015; Zhang et al., 2017), while the studies of chitosan and TiO_2 nanocomposite which can simultaneously reduce the negative effects of ethylene and inhibit microbial growth have never been reported. Therefore, this study was aimed to fabricate and characterize multifunctional active food packaging from chitosan- TiO_2 nanocomposites to be used as an ethylene scavenging and antimicrobial film aimed for postharvest applications of fresh produce.

2. Materials and method

2.1. Fabrication of nanocomposite film

Chitosan with 85% degree of deacetylation was obtained from Seafresh Industry Public Co., Ltd., Thailand. Chitosan film forming solution (2% w/v) was prepared using the procedure modified from the method of Siripatrawan and Vitchayakitti (2016) by dissolving chitosan powder into 1% acetic acid solution, and glycerol was used as plasticizer. The TiO₂ nanopowders of 21 nm particle size (>99.5%, Sigma-Aldrich Co., St. Louis, MO, USA) were added to the chitosan solution to obtain the chitosan-TiO₂ (CT) films with final TiO₂ concentrations of 0, 0.25, 0.5, 1 and 2% w/w (designated as CS, CT0.25, CT0.5, CT1 and CT2, respectively). The resulting solution was shaken at 90 rpm in a controlled-temperature water bath shaker (1083 GFL, Burgwedel, Germany) at 90 °C for 6 h. The solution was homogenized using a homogenizer (D-79282, Ystral GmbH, Ballrechten-Dottingen, Germany) and subsequently degassed using a sonicator (Ultrasonic Processor, Cole-Parmer, Vernon Hills, Illinois, USA). The film-forming solution (190 mL) was casted on a 30×12 cm² ceramic plate and dried. The obtained films were conditioned in an environmental chamber at 25 °C and 50% relative humidity (RH) for 48 h before further analysis.

2.2. Scanning electron microscope

The TiO₂ and cross sectional morphology of chitosan film, and chitosan-TiO₂ nanocomposites containing 0.25, 0.5, 1 and 2% TiO₂ were analyzed using scanning electron microscopy (JSM-5800 LV, JEOL, Tokyo, Japan). The film samples were fractured in liquid Nitrogen and were sputtered with gold for conductivity by a sputter coater before SEM analysis.

2.3. X-ray diffraction analysis

Crystal structures of TiO₂, chitosan, and chitosan-TiO₂

nanocomposite films were analyzed using an X–Ray diffractometer (D8-Discover, Bruker AXS Inc., Madison, WI, USA) in the range of $2\theta = 10^{\circ} - 80^{\circ}$ with a Cu K α radiation source at 0.15 nm. The working parameters were voltage of 40 kV, current of 40 mA, and scanning rate of 2 min⁻¹.

2.4. Fourier Transform Infrared spectroscopy

Fourier Transform Infrared (FTIR) spectroscopy was carried out to observe the interactions between chitosan and TiO_2 . The FTIR spectra of chitosan- TiO_2 nanocomposite films were recorded in the wavenumber range of 4000-400 cm⁻¹ with 4.0 cm⁻¹ resolution of 64 scans using a FTIR spectrometer (PerkinElmer 1760X, PerkinElmer Life And Analytical Sciences, Inc., Waltham, Massachusetts, USA) equipped with the Universal Attenuated Total Reflectance (ATR).

2.5. Mechanical properties

Mechanical properties including tensile strength (TS) and elongation at break (EAB) were measured with an Instron[®] universal materials testing machine (Instron Corporation, Canton, MA, USA) following the ASTM Standard Test Method D 882 (ASTM, 2010). The film samples, conditioned in an environmental chamber at 25 °C and 50% relative humidity (RH) for 48 h, were cut into strips with a test dimension of 15 × 2.5 cm². The film was mounted between grip heads with initial grip separation of 5 cm. The test was performed at a crosshead speed of 20 cm-min⁻¹. TS (Eq. (1)) was calculated by dividing the maximum load (F_{max}) by the initial cross-sectional area (ϕ) of the film sample and expressed as MPa. EAB (Eq. (2)) was calculated as the ratio of the film extension (Δl) at the point of sample rupture to the initial length (l_0) of a sample and expressed as a percentage. The measurements represent an average of at least nine samples.

$$TS = \frac{F_{\text{max}}}{\Phi} \tag{1}$$

$$%EAB = \frac{\Delta l}{l_0} 100 \tag{2}$$

2.6. Optical transmittance

Optical transmittance of the chitosan-TiO₂ nanocomposite film was measured at 800 nm using an UV-Vis spectrophotometer (Thermo Scientific GENESYS 20, Thermo Fisher Scientific, Inc., Rochester, NY, USA) following the method of Wang, Du, Luo, Lin, and Kennedy (2007). The films were cut into a rectangle piece and directly placed in a spectrophotometer test cell. Six replications were conducted for each sample treatment.

2.7. Water vapor permeability

Water vapor transmission rate (WVTR) of the films per unit area was determined following the ASTM Standard Test Method E96 (ASTM, 2010). Film samples, previously equilibrated at 25 °C and 50% RH for 48 h, were sealed to glass cups having 5 cm diameter containing silica gel. The film-covered cups were placed in an environmental chamber set at 25 °C and 75% RH using saturated solution of NaCl. The cups were weighed periodically until steady state was reached (\pm 0.0001 g). The water vapor permeability (WVP) of the films was calculated using Eq (3). At least six replications of each film treatment were tested for the WVP. Download English Version:

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