



Optical and thermo-mechanical properties of composite films based on fish gelatin/rice flour fabricated by casting technique



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ABSTRACT

Packaging films based on fish gelatin-rice flour (FG-RF) at different blend ratios (FG-RF \approx 10:0, 8:2, 6:4, 5:5 and 0:10, w/w) using 30% (w/w) glycerol as plasticiser were prepared and characterised. FG-RF composite films exhibited lower tensile strength (TS) and elongation at break (EAB), compared to FG film ($P < 0.05$). Higher water vapour permeability (WVP), but lower water solubility (WS) was obtained for FG-RF composite films having the increased proportion of RF ($P < 0.05$). Light transmission in ultraviolet (UV) and visible regions (200–800 nm) was lowered in all FG-RF composite films, indicating excellent light barrier characteristics. Based on FTIR spectra, significant changes in molecular structure and lower intermolecular interactions between FG and RF molecules were found in FG-RF (8:2) composite film. Thermogravimetric analysis indicated that FG-RF (8:2) composite film had only 7.61% (w/w) heat-stable mass residues in the temperature range of 50–600 °C. DSC thermograms suggested that FG-RF (8:2) composite film consisting of amorphous/microcrystalline layers of partially miscible aggregated junction zones and the coexisting two different order phases of unbound domains. SEM micrographs elucidated that FG-RF (8:2) composite film was rougher than FG film, but no signs of phase separation between film components were observed, thereby confirming their potential use as packaging material.

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1. Introduction

Packaging is one of the largest market sectors of polymer industry and is currently dominated by crude oil-derived, non-biodegradable polyolefin and polyesters based packaging materials. Due to their environmental persistence (non-biodegradability) leading to accumulation in land and water, plastic packaging materials have come under intense scrutiny. As a consequence, packaging films from eco-friendly materials have become the focus of worldwide attention in recent years due to their good film-forming ability, relative abundance, non-toxic nature, cost-effective, biodegradability and recyclability. Amongst biopolymer materials, fish gelatin and rice flour offer an alternative packaging material for the development of composite packaging films

that could maintain the food quality and extend the shelf-life of perishable items, due to their adequate mechanical properties and excellent barrier to moisture transfer, oxygen uptake, lipid oxidation, and losses of volatile aroma compounds at intermediate and low relative humidity (RH) [1].

Gelatin is a natural hydrocolloidal macromolecule derived from thermal hydrolysis or physicochemical degradation of fibrous insoluble collagen present in the bones and skin [2]. Based on the method in which the collagenous raw material is pre-treated, gelatins are classified into type A ($pI \sim 6-9$) produced from acid-treated collagen, and type B ($pI \sim 5$) produced from alkali-treated collagen [3]. In general, gelatin exists as a copolymer build up from triads of α -amino acids with glycine at every third position (soft blocks) and triads of hydroxyproline, proline and glycine (rigid blocks) [4]. The α -, β -, γ -chains and other peptide fragments of gelatin structure hold the native hydrophilic and hydrophobic domains, which enable gelatin to be an ideal dispersing and film-forming agent [5,6]. In addition, the unique structure of gelatin based on 20 different monomers (amino acids) provides a wider range of potential functionalities via different intermolecular

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bondings [7]. Due to the existence of reactive groups including $-\text{NH}_2$, $-\text{COOH}$, and $-\text{OH}$, gelatin can be further blended with other functional polymeric molecules viz., rice flour, for the preparation of composite films [8].

Rice flour is a starchy material derived from rice (*Oryza sativa* L.). Starch is the major chemical component of rice, comprising around 90% of its dry weight, while protein and lipid constitute about 6.5% and 0.8%, respectively. Due to its abundance, inherent biodegradability, low-cost and desirable performance, rice flour generates a greater interest and is considered as a promising alternative to synthetic polymers for packaging applications [9]. Its structure consists of concentric alternating amorphous and crystalline lamellae. The starch granules of rice flour are composed of two structurally distinct α -D glucan components, amylose and amylopectin. Amylose is a linear polymer of glucose units linked by α -(1,4) linkage; whereas amylopectin is a highly branched polymer of glucose units with ramifications in α -(1,6) linkage [10]. The amylose–amylopectin ratio ranges from 15:85 to 35:65 depending on the source of starch [11]. Films with high amylose content showed higher tensile performance (stronger), elasticity and oxygen barrier properties, but lower stretchability, retrogradation temperature and water solubility than those of low amylose starch films at high RHs [10,12,13]. Based on this, the rice flour used in our study has a higher amylose content of 30.40% in its composition [10]. In addition, amylose and amylopectin are responsible for the starch crystallisation which in turn modify the mechanical response (increased stiffness) of rice flour films [14].

Polymer blending is a well-used technique to endow multi-purpose materials with desired tailor-made properties [8]. The blending of compatible/miscible biopolymers could modify the properties of resulting biomaterial in effortless, economical and eco-friendly procedure [15]. Since, fish gelatin (FG) possess diverse functional groups distributed along the backbone and side chains, and thus is an ideal candidate to form a well mixed hybrid with rice flour (RF) upon blending in self organisation process [16]. Therefore, the objective of this investigation was to prepare FG-RF composite films at different blend ratios, and to analyse their physico-mechanical and optical properties. The intermolecular interactions and phase compatibility of composite films were determined by FTIR and SEM, respectively. The study was also focused to examine the heat flow and thermal stability using DSC and TGA, respectively.

2. Materials and methods

2.1. Materials

Type B commercial fish gelatin (FG) extracted from tilapia skin (~240 bloom strength, $pI \sim 5.08$) containing 85.78% (w/w) of protein as determined by the Kjeldhal method [17], was purchased from Lapi Gelatine S.p.a (Empoli, Firenze, Italy). Rice flour {RF; 90.83% (w/w) carbohydrate} was purchased from local market in Palau, Penang (Malaysia). Sodium azide was obtained from Sigma Chemical Co. (St. Louis, MO, USA). Glycerol (98% purity) and sodium hydroxide (NaOH) were procured from Merck (Merck Chemicals, Darmstadt, Germany). All chemicals used in this study were of analytical grade.

2.2. Preparation of composite film forming solutions

Briefly, FG was hydrated in distilled water to obtain the protein concentration of 2% (w/v), followed by heating at 60 °C for 30 min using a hot plate magnetic stirrer (IKA® C-MAG HS-7, Selangor, Malaysia). After dissolution, glycerol at 30% (w/w) based on protein content was added as plasticiser and the mixture was stirred gently for 30 min at room temperature. Similarly, RF was solubilised in

distilled water with continuous stirring to obtain the final carbohydrate concentration of 2% (w/v). The RF dispersion was gelatinised by heating at 85 °C for 30 min to ensure disintegration of starch granules and formation of a homogeneous dispersion. The mixture was cooled down at ambient temperature (28–30 °C) and then added with glycerol at 30% (w/w) based on carbohydrate content and left stirring overnight. Subsequently, composite film-forming solutions (FFSs) were prepared by mixing the above solutions at different FG-RF ratios (10:0, 8:2, 6:4, 5:5 and 0:10, w/w). The FFS was homogenised at a speed of 11,000 rpm for 2 min using a homogeniser (Model T25 basic, IKA® 18, 19 Labortechnik, Selangor, Malaysia). Prior to film casting, the viscous FFS was degassed for 10 min using ultra-sonicator water bath (Elmasonic EH075EL, New Jersey, USA) until homogenous solution was obtained.

2.3. Film casting, drying and conditioning of films

FFSs (4 ± 0.01 g) were cast onto a rimmed silicone resin plate (5×5 cm²), air-blown for 12 h at room temperature, followed by drying in an environmental chamber (Binder, KBF 115 # 00-19735, D-78532, Tuttlingen, Germany) at 25 ± 0.5 °C and $50 \pm 5\%$ RH for 24 h. Dried films were manually peeled off and subjected to further analyses.

Prior to testing, films were conditioned for 48 h at $\sim 50 \pm 5\%$ relative humidity (RH) and 25 ± 0.5 °C. For ATR-FTIR, DSC, TGA and SEM studies, films were conditioned in a desiccator containing dried silica gel for 1 week to minimise the plasticising effect of water, followed by drying in a desiccator containing P₂O₅ gel for 2 weeks at room temperature (28–30 °C) to obtain the most dehydrated films.

2.4. Analyses

2.4.1. Thickness

The thickness of films was measured using a digital electronic micrometer (Model ID-C112PM, Serial No. 00320, Mituyoto Corp., Kawasaki-shi, Japan). Ten random locations around each film sample were used for determination of thickness. Mean thickness values for each sample were taken and used in the calculation of WVP and TS.

2.4.2. Measurement of stress–strain properties

The stress–strain properties, such as TS and EAB of films were determined as described by Iwata et al. [18] using the Universal Testing Machine (Lloyd Instrument, Hampshire, UK). The test was performed in the controlled room at 25–28 °C and $\sim 50 \pm 5\%$ RH. Ten films (2×5 cm²) with the initial grip length of 3 cm were used for testing. The films were clamped and deformed under tensile loading using a 100 N load cell with the cross-head speed of 30 mm/min until the samples were broken. The maximum load and the final extension at break were used for calculation of TS and EAB, respectively.

2.4.3. Water vapour permeability (WVP)

WVP was measured using gravimetric modified cup method based on ASTM method (American Society for Testing and Materials) as described by Shiku et al. [19]. The films were sealed on an aluminum permeation cup containing dried silica gel (0% RH) with silicone vacuum grease and rubber gasket, and held with four screws around the cup's circumference. After taking the initial weight of the test cups, they were placed in a desiccator containing the distilled water (30 °C, $\sim 50 \pm 2\%$ RH), followed by weighing the test cup to the nearest 0.0001 g with an electronic balance (Model CPA225D, Sartorius Corp., Goettingen, Germany) after every 1 h interval for up to 8 h. A plot of weight gained versus time was used to determine the WVP and the slope of the linear portion of this

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