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Effects of electron beam irradiation on properties of corn starch undergone periodate oxidation mechanism blended with polyvinyl alcohol

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ABSTRACT

This work was performed to examine the properties of pristine PVOH and PVOH-starch blends under exposure of different irradiation dosages. The periodate oxidation method was used to produce dialdehyde starch. The application of low dosages of electron beam irradiation (≤10 kGy) has improved the tensile strength by forming crosslinking networks. However, the tensile strength drastically declined when radiated at 30 kGy due to the reduction of available hydroxyl groups inside polymer matrix for intermolecular interaction. Also, the incorporation of corn starch and dialdehyde starch has significantly reduced the melting temperature and enthalpy of melting of PVOH blends due to cessation of the hydrogen bonding between PVOH and starch molecules. The crystallite size for deflection planes (101), (101) and (200) for all PVOH blends was significant reduced when irradiated. The electron beam irradiation has also weakened the hydrophilic characteristic of all PVOH blends as evidenced in infrared and microscopy analysis.

1. Introduction

Nowadays, the synthetic and non-biodegradable polymer materials have been widely applied in human daily life and various applications in industry. However, the increasing of synthetic and non-degradable polymer materials usage in human daily life have caused serious environmental problem due to accumulation of non-degradable polymer solid wastes [1–3]. Hence, biodegradable polymer materials such as polyvinyl alcohol (PVOH), polylactic acid (PLA), blends of starch (polysaccharides) and biodegradable polymer etc. are considered as promising alternatives to replace the usage of non-biodegradable polymer materials [4–5]. Currently, the development of biodegradable polymer materials such as polyvinyl alcohol (PVOH), starch (polysaccharides) based materials, polylactic acid, etc. have rapidly increased due to its environmental friendly characteristic can reduce the pollution problem caused by accumulation of polymer solid waste [1,6–8]. PVOH is a one of the most commonly used synthetic biodegradable polymers in food packaging and biomedical industries. The high melting temperature of PVOH is mainly attributed to the high crystallinity of polymer matrix and also the existence of strong hydrogen bonding among hydroxyl groups (O—H) in polymer matrix PVOH [9]. PVOH has been widely used in food packaging application due to its excellent performances in mechanical, optical and physical properties [4,10–11]. However, the applications of PVOH in various industries are limited due to its expensive price [4].

On the other hand, starch is a multi-hydroxyl polymer with the presence of vast intermolecular and intramolecular hydrogen bonds in the structure of granule [12]. Starch is widely used to blend with synthetic biodegradable polymers such as PVOH, polylactic acid, chitosan [13,14] to reduce the cost of polymer blends. Various modification methods of native starch have been carried out by many researchers to improve the irregularity problem of starch particles in the polymer matrix such as PLA/starch blends and PVOH-starch blends, etc. [12,15]. Periodate oxidation method is one of the modification methods used to improve the regularity of starch particles in polymer matrix by providing the reinforcement of polymer matrix [12,16–18]. During the periodate oxidation reaction, the C-2 and C-3 bonds of the anhydroglucose rings of native starch polysaccharide would breakdown into aldehyde groups (C=O) from the hydroxyl groups (C—OH) to form dialdehyde starch (DAS) [12,18]. In decades, dialdehyde starch attracts considerable potential market as biodegradable polymer materials in the food industries for packaging purpose. Whilst, electron beam irradiation is widely applied in polymer engineering field to improve the mechanical properties of polymer blends. Numerous of researches have

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been conducted to investigate the enhancement effect of electron beam irradiation on physical–mechanical properties of polymer blends added with types of filler [19–22]. Previous research by Bee et al. [20] reported that the application of electron beam irradiation is able to gradually improve the mechanical properties of montmorillonite (MMT) added PVOH. Hence, the current work aims to investigate the effect of electron beam irradiation on physical, mechanical and thermal properties of native corn starch and dialdehyde starch (DAS) added PVOH compounds.

2. Experimental

2.1. Materials

In this study, fully hydrolyzed polyvinyl alcohol (PVOH) with grade of Denka Poval, K-17C was used as the primary polymer base. The PVOH used in this study was manufactured by Denki Kagaku Kougyo Kabushiki Kaisya (DENKI). The hydrolysis and viscosity of the Denka Poval, K-17C graded PVOH are 87–89 mol% and 45–55 mPa.s, respectively. The food grade native corn starch was used as secondary polymer base in this study. The native corn starch was supplied by Thye Huat Chan Sdn Bhd., Malaysia. Sodium periodate with Fisher Scientific brand was used to prepare the dialdehyde starch from native corn starch using periodate oxidation reaction. The Fisher Scientific brand sodium periodate with the specification of 0.2 mL N% of acidity, 0.003% of insoluble matter in water and 0.025% of loss of drying at 110 °C, Assay > 99% was supplied by Warisan Alam Enterprise Sdn. Bhd., Malaysia. Concentrated sulphuric acid with purity of 95–98% was used to control the acidity of dialdehyde starch solution during the preparation of dialdehyde starch. The concentrated sulphuric acid was purchased from Sigma-Aldrich (M) Sdn Bhd., Malaysia.

2.2. Preparation of dialdehyde starch

Sodium periodate powder was initially dissolved into distillated water to prepare the sodium periodate solution with concentration of 0.35 mol/L. On the other hand, the native starch solution was prepared by dissolving 16 g of native corn starch into 100 mL of distillated water. After that, the sodium periodate solution and native starch solution were mixed together in a beaker and stirred slowly for five hours at room temperature to enable the occurrence of periodate oxidation reaction. During the mixing process, the concentrated sulphuric acid was used to control the pH of the mixture solution in the range of 3.5–4.0 by adding into the mixture solution. After five hours of reaction, dialdehyde starch was separated from the mixture solution in slurry form using centrifuge machine. The separation process was conducted under high stirring speed of 12,000 rpm for 30 min. The separated dialdehyde starch in slurry form was washed using distillated water for several times and then dried in the oven at 55 °C for 24 h.

2.3. Sample preparation

The samples of PVOH-corn starch blends and PVOH-DAS blends were prepared via solution casting method with the PVOH: starch ratios of 80%: 20%, 60%: 40%, 40%: 60% and 20%: 80%, respectively. Initially, the PVOH resin was dissolved in distilled water using a water bath at temperature of 97 ± 2 °C for 30 min. A driven motor was used to stir the mixtures of water and PVOH resin evenly at the rotating speed of 350 rpm until all the PVOH resin fully dissolved in distilled water. After that, native corn starch or dialdehyde starch (DAS) was added into PVOH solution and then stirred again in water bath at temperature of 97 ± 2 °C for another 30 min. The mixtures solution of PVOH-corn starch or PVOH-DAS was cast onto a petri dish into sheet form with approximately 1 mm in thickness. Then, the cast samples were dried in a vacuum oven at constant temperature of 65 °C until reach a constant weight. The dried PVOH samples were kept and stored in sealed plastic bags under room temperature of 25 °C at relative humidity of 65% for conditioning purpose.

The cast samples were further electron beam irradiated to irradiation doses of 10, 20 and 30 kGy under the EPS-3000 electron beam irradiation machine with the irradiation rate of 10 kGy per pass. The irradiation voltage, current and energy of the EPS-3000 electron were set at 15 kV, 1 mA and 1 MeV, respectively.

2.4. Gel content

The gel content test was conducted to evaluate the degree of crosslinking network induced by irradiation in accordance to ASTM D2765. Firstly, the cast samples of pristine PVOH, corn starch, dialdehyde starch and all PVOH-starch blends were cut into smaller pieces using sample cutter. After that, 0.2000 g of cut samples were weighed by using an analytical balance. The weighed samples were then gravimetrically immersed and heated hot water for two hours under a constant heating temperature of 100 °C. After two hours of extraction, the extracted samples were washed using clean water for several times to remove the stain of soluble materials. The washed samples were further dried to constant weight in a vacuum oven at temperature of 40 °C for 24 h. The dried samples were accurately weighed and recorded as remaining weight (W_f) using analytical balance. The gel content percentage of all the samples was calculated according to Eq. (1). Three specimens per sample were used to calculate the average value of gel content percentage.

$$\text{Gel content}(\%) = \frac{0.2000\ \text{g} - W_f}{0.2000\ \text{g}} \times 100\% \quad (1)$$

where W_f represents the remaining weight of samples after the extraction process.

2.5. Tensile test

Tensile test was carried out by using Instron 5848 Tensile Microtester in accordance to ASTM D882. Initially, the cast and electron beam irradiated samples were cut into the standard rectangular shape according to ASTM D882. After that, the cut samples were placed in the grips of the Tensile Microtester and tested with the crosshead speed of 50 mm/min. Five specimen were tested for each formulation and the obtained results were then averaged to calculate the mean value and standard deviation.

2.6. Differential scanning calorimetry (DSC) test

Differential scanning calorimetry test was conducted using Mettler Toledo DSC823 Differential Scanning Calorimeter. The samples of PVOH-corn starch and PVOH-DAS blends with weight of 2–5 mg was measured and loaded into a crucible. The nitrogen gas was purged into the testing column with flow rate of 2 L/min before the testing to eliminate the air inside the testing column. After that, the sample in crucible was heated from room temperature to 250 °C under nitrogen atmosphere with the heating rate of 20 °C/min.

2.7. X-ray diffraction (XRD) test

The crystalline structures of PVOH-corn starch and PVOH-DAS samples was investigated using X-ray Diffraction Shimadzu XRD 6000 diffractometer with Cu-Kα radiation (λ = 1.542 Å). The samples were scanned from 2θ = 0° to 2θ = 80° at a scanning rate of 1.0°/min. The operating acceleration voltage and current of the Cu-Kα radiation generator were set to 40 kV and 30 mA, respectively. The crystallite size, L of crystallites in polymer matrix of PVOH-corn starch and PVOH-DAS blends was calculated using the Scherrer equation as shown in Eq. (2). The d-spacing, d of crystallites in PVOH-corn starch and PVOH-DAS samples was calculated using the Bragg's equation as shown in Eq. (3).

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