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The effects of UV treatment on the properties of sago and mung bean films

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Abstract: Starch blend films made from sago and mung bean were prepared by casting with glycerol as the plasticizer and subsequently exposed to ultraviolet (UV) irradiation for 2 h. The films were characterized by thickness, moisture sorption isotherms, X-ray diffraction and Fourier transform infrared (FTIR) spectroscopy. All films produced were colorless while incorporation of glycerol resulted in more flexible and manageable films. Moisture sorption isotherms for all films showed sigmoidal shape and the control films showed slightly higher curve than treated films. While for X-ray analysis, the control and treated films for all formulations showed similar pattern, however for treated films showed more crystalline character. UV radiation showed affect on X-ray diffraction and sorption isotherms; however the UV radiation did not affect the spectra pattern of FTIR.

Keywords: Sago, mung bean, starch films, moisture sorption isotherms, FTIR

Introduction

Biopolymer films have been regarded as potential replacements for synthetic films in food packaging applications in response to a strong marketing trend towards more environmentally friendly materials (López *et al*., 2008). Starch is the most commonly used biopolymer for manufacturing thermoplastic films. Starch is composed of a mixture of amylose and amylopectin macromolecules both consisting of 1, 4 α-D glucopyranose repeating units (Pushpadass *et al*., 2008). Amylose is primarily responsible for its film forming ability and functional properties of starch-based films due to its recrystallization behavior after its processing. Amylose forms strong and flexible films whereas amylopectin produces weak and brittle ones (Zobel, 1988; Myllärinen *et al*., 2002b).

Eventhough starch films are excellent O_2 barrier (Mali *et al*., 2005); there are some limitations in mechanical properties due to high moisture sensitivity (Veiga-Santos *et al*., 2005). Consequently, improving the properties of starch films through various treatments is desirable (enzyme, physical and chemical). Generally, the modification techniques are often complex and time consuming, however irradiation can provide a simple and environmental-friendly to change the physical, chemical and / or biological characteristics of a product. This treatment is fast, does not require catalyst and does not induce major temperature increase (Woods and Pikaev, 1994). Ultraviolet radiation covers small part of the electromagnetic spectrum with wavelength shorter than that visible light but longer than X-rays (Diffey, 2002). It is not only used in industrial applications but also has been used in medical and pharmaceutical research to crosslink collagen and gelatin films (Fujimori, 1965; Weadock *et al*., 1984; Tomihata *et al*, 1992). Based on previous studies, UV radiation (wavelength 200-300 nm) also used to reduce microbial loads on foods (Wallner-Pendleton *et al*., 1994). Similar to ionizing radiation like gamma rays, UV light is also known to bring about the photodegradation of starch, resulting in the cleavage of glycosidic bonds accompanied by the shortening of the amylose chain and a debranching of the amylopectin chain due to the formation of free radicals (Merlin and Fouassier, 1981).

Pure starch film is brittle due to its strong intermolecular and intramolecular hydrogen bonding in the amylose and amylopectin molecular chains; it requires and needs plasticization to make it flexible. Plasticizers overcome the brittleness in starch films by softening the structure and increasing the mobility of the macromolecular chains (McHugh and Krochta, 1994; Myllärinen *et al*., 2002b; Ke and Sun, 2001; Mali *et al*., 2005; Chang *et al*., 2006).

Starch is present in semicrystalline granules that vary in composition, size, shape and functionality when obtained from different botanical sources (Tharanathan, 1995; Tharanathan, 2003). Several different starches are available on the market, including corn, cassava, wheat, rice, etc. and each type has specific properties and, hence, special application (Riaz, 1999).

Starch from sago palm (*Metroxylon sagu Rottb*.) is an increasingly important socioeconomic crop in Southeast Asia. Sago starch is the only example of industrial starch (food and nonfood) derived from

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different sources in contrast to common starches e.g. cereals (rice, corn), tubers (potato), roots (tapioca) and legumes (pea, mung bean) (Abdorreza *et al*., 2011). Sago starch has own its unique characteristics but some of its physicochemical properties are quite similar to common starch such as cassava and potato (Karim *et al*., 2008a; Karim *et a*l., 2008b; Tie *et al*., 2008).

Mung bean starch has a considerably lower degree of syneresis than other legume starches. Moreover, gel consistency decreases as starch concentration increases. Viscosity of mung bean starch is high, indicating that it had a higher resistance to swell and rupture than cereal starches. This indicated that the swelling power and solubility of the starch increases with increasing temperature. In addition, the solubility percentage increases non-linearly with increasing swelling power (Abdel-Rahman *et al*., 2008). Mung bean is similar in composition to other members of the legume family with 24% protein, 1% fat, 63% carbohydrate and 16% dietary fiber (Liu and Shen, 2007). Mung bean has been reported as being the best raw material for starch noodle preparation owing to its high amylose content and restricted swelling (Ohwada *et al*., 2003). The purpose of this study is to investigate the effects of crosslinking via UV treatment on physical properties of films.

Materials and Methods

Sago and mung bean starches were purchased from YKL Multi Enterprise, Penang, Malaysia whereas glycerol from Qrec (Auckland, New Zealand).

Film preparation

Films were obtained by using different ratio of sago (S) and mung bean (M) starches (S100, M 100, SM75:25, SM50:50 and SM25:75) based on total weight basis (7 g) in 200 mL distilled water including 2 g glycerol. The mixtures of starches were heated to 85 °C while continually stirring for 1 h followed by cooling down to room temperature. The films were prepared by casting; the cooled solutions (~ 95 g) were immediately poured on rectangular polyacrylic plates (16 cm x 16 cm). UV treatments were applied to films (253.7 nm; 20W) in a laminar flow cabinet (Esco® PCR Vertical, Laminar Flow Cabinet, Singapore) at a distance of 20 cm from the surface for 2 h. Finally, the films were dried by oven drying at 400 °C for 24h and the dried films were peeled and stored for further analysis.

Thickness measurements

Thickness of the films was determined using a micrometer (dial thickness gauge 7301; Mitutoyo Co., Tokyo, Japan) at five random positions of the film and the average was calculated.

Moisture sorption isotherm

Sorption isotherms of starch films were determined at 30 °C according to the procedure described by Spiess and Wolf (1983) with some modifications. Films were cut into small pieces (1.2 x 2.0 cm) and pre-dried in a vacuum dessicator over P_2O_5 at room temperature (25-28°C) for 7 days to obtain 'zero' water content. The dried films (200 mg each) were weighed to the nearest 0.0001 g into pre-weighed weighing bottles. The dried samples in triplicate were equilibrated in air-tight 1 L Kilner jars containing different saturated salt solutions of known relative vapour pressure (RVP) at 30°C (Greenspan, 1977). The saturated salt solutions used are shown in Table 1. Samples were weighed daily. 'Equilibrium' was assumed to have been achieved when the change in weight did not exceed 0.1% for 3 consecutive weighing. Moisture content (dry basis) was later calculated from the weight gained.

Table 1. RVP for saturated salt solutions used at 30°C

Saturated salt solution	RVP at 30°C
Lithium chloride (LiCl)	0.11
Potassium acetate (CH_3COOK)	0.22
Magnesium chloride ($MgCl_2$)	0.32
Potassium carbonate (K_2CO_3)	0.43
Magnesium nitrate ($MgNO_3$)	0.52
Strontium chloride ($SrCl_2$)	0.69
Sodium chloride (NaCl)	0.75
Potassium chloride (KCl)	0.84
Potassium nitrate (KNO_3)	0.92

X-ray diffraction

Films were cut into 5 x 5 cm strips and conditioned in a dessicator at RH 52% using a saturated salt solution of magnesium nitrate for 7 days. Films were analyzed between $2\theta = 2°$ and $2\theta = 60°$ with a step size $2\theta = 0.02°$ in a X-ray diffractometer (D5000, Siemens Diffractometer, Karlsruhe, Germany) using Cu Kα radiation ($\lambda = 1.543$), 50 kV and 30 mA (Mali *et al*., 2002).

Fourier Transform Infrared spectroscopy

The spectra of control and UV-treated (2 h) films were determined using Fourier Transform Infrared (FTIR) spectrometry (System 2000, Perkin Elmer Wellesley, MD, USA) in the frequency range of 4000-650 cm-1. The films were pressed against the objective to directly analyzed the samples The spectra were used to determine any possible changes induced on treated samples by UV.

Statistical analysis

All tests were performed in triplicates. Analysis of variance (ANOVA) was performed using the using the Duncan's multiple range tests ($p < 0.05$) was used to determine the significant differences between means

Results and Discussion

Based on the observations, it was found that the films produced with an average thickness of 0.130 mm are transparent, odorless and tasteless. Other researchers reported that the starch films are generally transparent (Myllärinen *et al*., 2002b; Parra *et al*., 2004) or translucent (Rindlav *et al*., 1997), odorless, tasteless and colorless. According to Guilbert (1986), the addition of a plasticizer agent, such as glycerol, effectively reduces internal hydrogen bonding while increasing intermolecular spacing, thereby decreasing brittleness and increasing permeability of the film.

The sorption isotherms of S 100, M 100, SM 7525, SM 5050 and SM 2575 films, control and UV-treated (2 h) are presented in Figure 1 and Figure 2. The results have shown that the moisture content of all films increases with increasing water activity (Aw). The sigmoidal shape found has been typically reported as Type II isotherm (Al-Muhtaseb *et al*., 2002). This is in agreement with some other researchers, who found this behavior for other starchy materials, such as potato starch (Boki and Ohno, 1991; Chatakanonda *et al*., 2003; Al-Muhtaseb *et al*., 2004; Mishra and Rai, 2006), wheat and rice starches (Boki and Ohno, 1991). This type II isotherm occurs when more water condenses on the surface over the 1st water monolayer (Ruthven 1984).

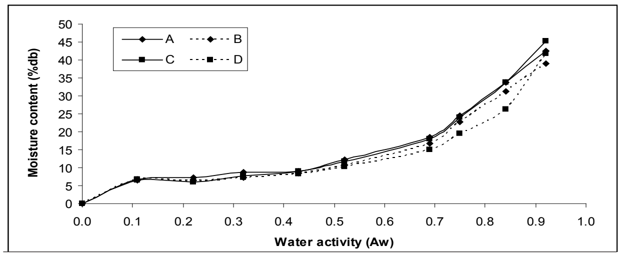

Figure 1. Moisture sorption isotherms for starch films, control and UV-treated for 2 h, A) S 100 0h, B) S 100 2h, C) M 100 0h and D) M 100 2h

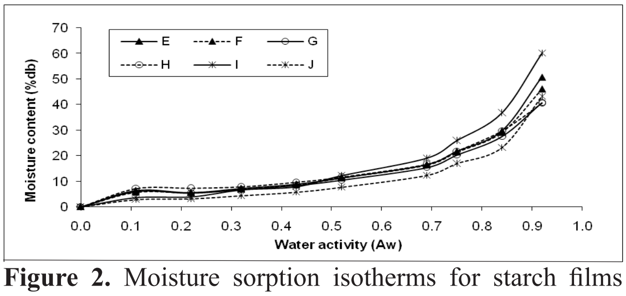

Figure 2. Moisture sorption isotherms for starch films control and UV-treated for 2 h, E) SM 75:25 0h, F) SM 75:25 2h, G) SM 50:50 0 h, H) SM 50:50 2h, I) SM 25:75 0h, J) SM 25:75 2 h

Moisture content of all films increased slowly from 0 to 0.5 of aw but increased drastically from 0.6 to 0.9 of aw. This result is inline with Zhang and Han (2008), who found that the weak interactions between the water and starch matrix led to low uptakes at low relative humidity. However, once a water molecule had been adsorbed at a primary adsorption site on the surface of the starch film, the water-water interaction becomes the driving force of the adsorption process, resulting in accelerated uptakes at higher relative pressures. Zhang and Han (2008) also stated that the 1st water molecules that are adsorbed loosen and swell the starch polymer structure locally (i.e. lower the cohesion of starch polymers), attract other water molecules in the atmosphere and allow subsequent water molecules to easily enter the neighborhood of the 1st molecules. Thus, making the adsorbed water molecules, effective plasticizing agents of the starch polymer.

Figure 1 and Figure 2 also showed the effect of UV irradiation on the moisture sorption characteristics of starch blend films. The sorption isotherms of control films adsorb more moisture under the same conditions of relative humidity and temperature, therefore the curve of control films were slightly higher compared to treated UV films. A 22% difference was observed at 0.84 of aw between the UV treated and control M100 films. Our observation was supported by Zhou *et al.* (2008), who found that moisture sorption isotherms of corn starch sheet treated by UV were lower than the controlled sheet with 17% difference between the UV and control starch sheet.

X-ray diffraction patterns of control and UV-treated starch blend films are presented in Figure 3 to 7. The pattern shown the starch films could be assigned to a B-type. Based on previous studies, starch films normally show a B-type x-ray diffraction, regardless of the original polymorphs of starch from which the films are made (Bader and Göritz, 1994; Rindlav *et al*., 1997; Rindlav-Westling *et al*., 1998; Mali *et al*., 2002; Myllärinen *et al*., 2002a). The films show an important contribution of the amorphous phase containing a small fraction, in agreement with previous studies (Angellier *et al*., 2006; Famá *et al*., 2006; Famá *et al*., 2007; Flores *et al*., 2007; Kristo and Biliaderis, 2007).

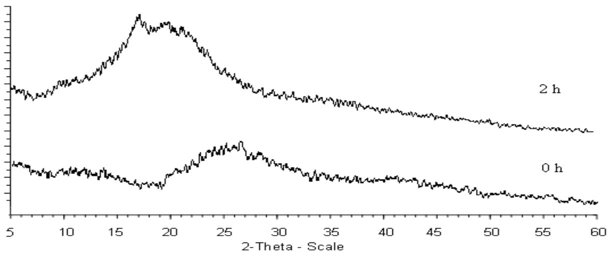

Figure 3. X-ray chromatograms of S 100 control and UV-treated for 2 h exposure time

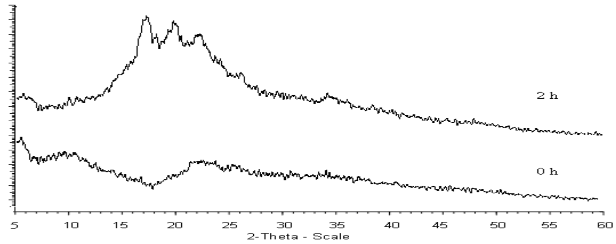

Figure 4. X-ray chromatograms of M 100 control and UV-treated for 2 h exposure time

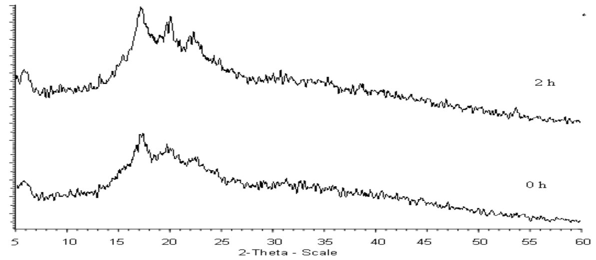

Figure 5. X-ray chromatograms of SM 75:25 control and UV-treated for 2 h exposure time

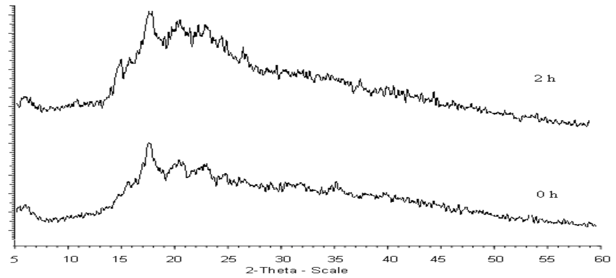

Figure 6. X-ray chromatograms of SM 50:50 control and UV-treated for 2 h exposure time

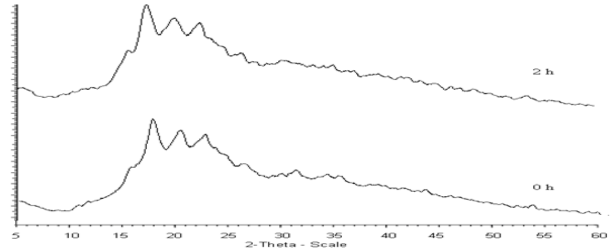

Figure 7. X-ray chromatograms of SM 25:75 control and UV-treated for 2 h exposure time

Even though the control and treated films show similar x-ray patterns, the treated films showed a more crystalline nature. The peaks appeared to be sharper and well defined as compared to control films. These results indicated that the crystallinity of the film increases when exposed to UV irradiation. This is in accordance with the results of Abd El-Rehim *et al.* (2004), who found that the UV irradiation increased the crystallinity the low density polyethylene (LDPE) / starch blends. According to Colonna *et al*. (1988), sharper x-ray diffraction peaks indicated that the amorphous parts of the starch granules had been disrupted. For M 100, SM 7525, SM 5050 and SM 2575 treated films exhibit strong peaks at 2θ about 15° and 17° were observed, while S 100 films showed lower and broader peak.

The spectra of control and UV treated starch blend films (Figure 8 and 9) show identical patterns, indicating insignificant changes among the functional groups. This spectrum represents a typical pattern of starch, in agreement with descriptions in literature (Pavlovic and Brandao, 2003). From the Figures 8 to 9, broad band was observed at 3600-3000 cm^{-1}, in which the main absorption band (3293.65 and 3325.39 cm^{-1}) is related to hydroxyl groups of starch. The absorption bands at 2928.57 cm^{-1} refer to the C-H stretching in the starch molecules, while in the region of 2000-400 cm^{-1}, the main absorption bands correspond to bound water at 1645.15 and 1653.08 cm^{-1}, C-H bending at 1411.23 cm^{-1} and bands associated with C-O, C-C and C-O-H in the region 1200-900 cm^{-1}. This similar to results obtained by Wolkers et al. (2004). The bands at 1153.52 and 1002.86 cm^{-1} can be assigned to the glycosidic linkage.

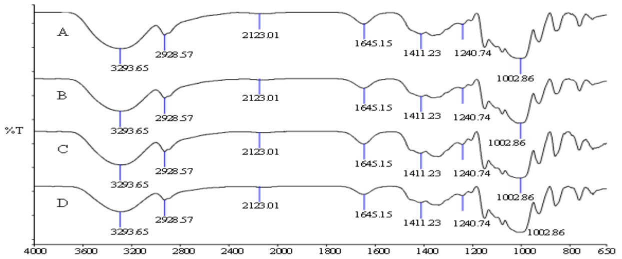

Figure 8. FTIR spectra for starch films (control and UV-treated) A) S 100 0h, B) S 100 2 h, C) M 100 0h and D) M 100 2h

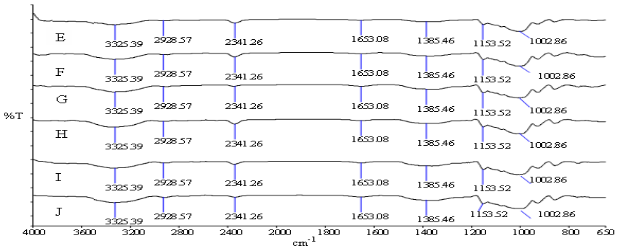

Figure 9. FTIR spectra for starch films (control and UV-treated) E) SM 75:25 0h, F) SM 75:25 2h, G) SM 50:50 0h, H)SM 50:50 2h, I) SM 25:75 0h and J) SM 25:75 2h

Results showed that all the films produced from different ratios have good flexibility and manageability. The addition of glycerol as a plasticizer has improved the extensibility of films. Although, the films were conditioned under the same RH and temperature, the curves of sorption isotherms for treated films are lower compared to control films. For X-ray diffraction, the pattern of starch films for control and treated are identical and showed a B-type pattern. However, the treated films showed more crystalline character. In contrast, for FTIR spectra there were no changes between films control and treated with UV radiation. Based on the outcome of the analysis, we can conclude that UV radiation has affect on X-ray diffraction and sorption isotherms but did not affect the spectra pattern of FTIR.

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