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Thermo-mechanical behaviors of thermoplastic starch derived from sugar palm tree (*Arenga pinnata*)

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ABSTRACT

In recent years, increasing environmental concerns focused greater attention on the development of biodegradable materials. A thermoplastic starch derived from bioresources, sugar palm tree was successfully developed in the presence of biodegradable glycerol as a plasticizer. Sugar palm starch (SPS) was added with 15–40 w/w% of glycerol to prepare workable bioplastics and coded as SPS/G15, SPS/G20, SPS/G30 and SPS/G40. The samples were characterized for thermal properties, mechanical properties and moisture absorption on exposure to humidity were evaluated. Morphological studies through scanning electron microscopy (SEM) were used to explain the observed mechanical properties. Generally, the addition of glycerol decrease the transition temperature of plasticized SPS. The mechanical properties of plasticized SPS increase with the increasing of glycerol but up to 30 w/w%. Meanwhile, the water absorption of plasticized SPS decrease with increasing of glycerol.

1. Introduction

Plastics are extremely stable and commonly used in various rapidly growing areas industries including packaging, electric and electronic devices and automotive. Convenience, lightweight, low price and esthetically pleasing are the most important features that make them high priority materials in the plastics production. However, the plastics produced from petroleum were not biodegradable and brought negative impact not only for human being, but also unfortunately create the serious environmental problems. In addition, the shortage and high cost of fossil resources require alternative resources that are sustainable for our future generation. Therefore, green materials are the wave of the future which can lead to solve non degradable polymer waste disposal problems.

Nowadays, much effort emphasized on the biodegradable polymers derived from natural resources for the aim of the replacement of petroleum based polymers (Averous & Boquillon, 2004). Among all biodegradable polymers, starch has been considered as one of the most promising one due to its easy availability, biodegradability, lower cost and renewability (Xu, Kim, Hanna, & Nag, 2005). Usually, 6–30% of starch was used as filler to increase the biodegradability of reinfoced synthetic polymer (Bagheri, 1999; Evangelista, Nikolov, Sung, Jane, & Gelina, 1991) and possibility of transforming starch into a thermoplastic material has spurred considerable interest in the last decade (Roper & Koch, 1990; Stepto, 2003). The starch is not a real thermoplastic, but, it will be act as thermoplastic material in presence of a plasticizer at high condition of temperature. Plasticizers are the most important materials that can increase the flexibility and processibility of thermoplastic starch. Besides, use of plasticizer would lead to change in properties of starch, which is depend on the amount of plasticizer used. For example, glass transition temperature (Tg) of cassava starch is reported as decreased with the increasing amount of glycerol (Bergo, Sobral, & Prison, 2009). Tensile properties of cassava starch are also reported to change accordingly. Significant research has been performed on the plasticization of thermoplastic starch using glycerol (Fishman, Coffin, Konstance, & Onwulata, 2000; Forssell, Mikkia, Moates, & Parker, 1997; Forssell, Mikkilti, Moates, & Parker, 1997), sorbitol (Gaudin, Lourdin, Le Botlan, Ilari, & Colonna, 1999), urea, formamide (Ma, Yu, & Jin, 2004), dimethyl sulfoxide (Nakamura & Tobolsky, 1967) and low molecular weight sugars (Kalichevsky, Jaroszkiewicz, & Blanshard, 1993).

Sugar palm tree is one of multipurpose trees grown in Malaysia. It is a member of the *Palmae* family and naturally a forest species. Besides yielding *neera* sugar, it also provides a great variety of products such as ropes, filters, brooms and roof materials. Sugar palm tree also can produce juices from its fruits. The fruits are white in colors which are taken from its fruit bunch. They can be

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preserved in heavy syrup for canned product and the fruits can be cooked with sugary syrup for desserts. The outer part of the stem consists of wood which is extremely hard and durable. It can be processed for flooring, furniture and hand grips of tools. It is also reported that the sugar palm's roots are useful for medicine (Ishak, Leman, Sapuan, Rahman, & Anwar, 2011; Mogea, Seibert, & Smits, 1991). Nowadays, researchers are more focusing on production of bioethanol that can be derived from *neera* sugar via fermentation process (Sahari, Sapuan, Ismarrubie, & Rahman, 2011a,b).

The inner part of sugar palm stem contains starch and for the production of one ton of starch, ten to twenty trees are needed which suggests that one tree can produce fifty to one hundred kg of starch (Muhtadi, 1991; Sahari, Sapuan, Zainudin, & Maleque, 2012). This unutilized starch can be developed as a thermoplastic starch and the research been carried out to evaluate the potential of sugar palm starch (SPS) as a thermoplastic starch with the present of glycerol. The present investigation deals with the effect of glycerol to the plasticized SPS. The properties of plasticized SPS such as thermal, physical and mechanical properties were studied and presented.

2. Materials and methods

2.1. Preparation of sugar palm starch

The sugar palm starch (SPS) was collected from sugar palm tree at Jempol, Negeri Sembilan, Malaysia. Firstly, the matured sugar palm tree was cut using chainsaw and the mixture (woody fibers and starch powder) was obtained from the interior part of the stem. This mixture was then carried out for washing process to obtain the starch. In washing process, water was added to the mixture and then the mixture was kneaded by hand. The fibers remain on the top of the sieve while the water carrying the starch granules in suspension passes through the sieve and was caught in container. The starch settles on the bottom and the excess water flows over the sides. After washing process, the fibrous remnants were discarded and the wet starch was taken out from the container. Then starch was kept in an open air for 30 min and dried in an air circulating oven at 120 °C for 24 h.

2.2. Fabrication of plasticized SPS

Sugar palm starch (SPS) was prepared by the addition of glycerol in a specific ratio (15 w/w%, 20 w/w%, 30 w/w% and 40 w/w%). The mixture stirred and maintained at 40 °C for 30 min. The mixture was cast in an iron die and was kept for pre-curing at room temperature (28 °C) for 24 h. Finally, the sample was cured by hot pressing in a Carver hydraulic hot press at 130 °C for 30 min under the load of 10 ton. The final products were in the form of plates with dimensions 150 cm × 150 cm × 0.3 cm.

2.3. Thermogravimetric analysis (TGA)

Plasticized SPS was cut into small pieces for TG analysis. The thermogravimetric analysis (TGA) conditions were: a nitrogen atmosphere, a flow rate of 20 mL min^{-1}, a heating rate of 10 °C min^{-1} and a temperature range from ambient to 800 °C.

2.4. Differential scanning calorimetry (DSC)

DSC measurements were carried out in a Shimadzu TA-50WSI Thermal Analyzer equipped with DSC-50 modules. The scans were carried out from room temperature to 500 °C at a heating rate of 10 °C min^{-1}, using a nitrogen atmosphere with a flow of 20 mL min^{-1}.

2.5. Mechanical properties of plasticized SPS

Plasticized SPS specimens were cut from the plates by using vertical saw with the size of specimens for tensile test was 150 mm (length, L) × 25 mm (width, W) × 3 mm (thickness, T). The tensile test was done based on ASTM D638 using the Instron 3365 machine with a crosshead-speed of 2 mm/min.

2.6. Water absorption

Plasticized SPS was stored in closed humidity chamber at 75% relative humidity (RH) for 3 days in order to analyze the effect of environmental humidity. Prior to the water absorption measurements, the plasticized SPS with the dimension of 20 mm × 20 mm × 3 mm was dried at 80 °C overnight to constant weight. The water content of plasticized SPS was determined gravimetrically based on original weight, current weight and its original water content.

2.7. Fourier transform infrared (FT-IR) spectroscopy

Fourier transform infrared (FT-IR) spectroscopy was used in order to detect the presence of the functional groups existed in plasticized SPS. The spectra were obtained using an IR spectrometer (100 Series type, Perkin-Elmer). About 2 mg of sample which is in powder form was mixed with potassium bromide (KBr) and pressed into a disk of about 1 mm thick. The FT-IR spectra of the sample was collected within the range of 4000–200 cm^{-1}.

2.8. Scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) Hitachi S-3400N operating at an acceleration voltage of 0.3–30 kV was used to study the fracture surfaces morphology of the samples.

3. Results and discussion

3.1. Thermal properties

The glass transition temperature, Tg is a very important parameter for determining the mechanical properties. Fig. 1 shows typical curves of plasticized SPS determined by DSC. For dry SPS the Tg reaches 242.14 °C and decreased with addition of glycerol. This value was higher than Indica rice starch where Tg values were 237 °C (Vasudeva, Hiroshi, Hidechika, Seiichiro, & Ken'ichi, 2000). Meanwhile, Myllärinen, Partanen, Seppälä, and Forsella (2002) claimed that for dry starch the Tg reaches 227 °C. The sample with high glycerol concentrations showed lower Tg values and Tg of starch without plasticizer were higher than those of samples with glycerol (Table 1). This behavior was also observed by Mali, Grossmann, Garcia, Martino, and Zaritzky (2002) for yam starch and by Forssell, Mikkia, et al. (1997) and Forssell, Mikkilti, et al. (1997), for films based on barley starch, in both cases with glycerol as plasticizer. According to Guilbert and Gontard (1995), plasticization decreases the intermolecular forces between polymer chains,

Table 1
Glass transition temperature (Tg) of plasticized SPS.

Sample	Glass transition temperature, Tg (onset)	Glass transition temperature, Tg (midpoint)
Native SPS	237.91 °C	242.14 °C
SPS/G15	225.68 °C	229.26 °C
SPS/G20	206.44 °C	217.90 °C
SPS/G30	189.57 °C	187.65 °C
SPS/G40	176.71 °C	177.03 °C

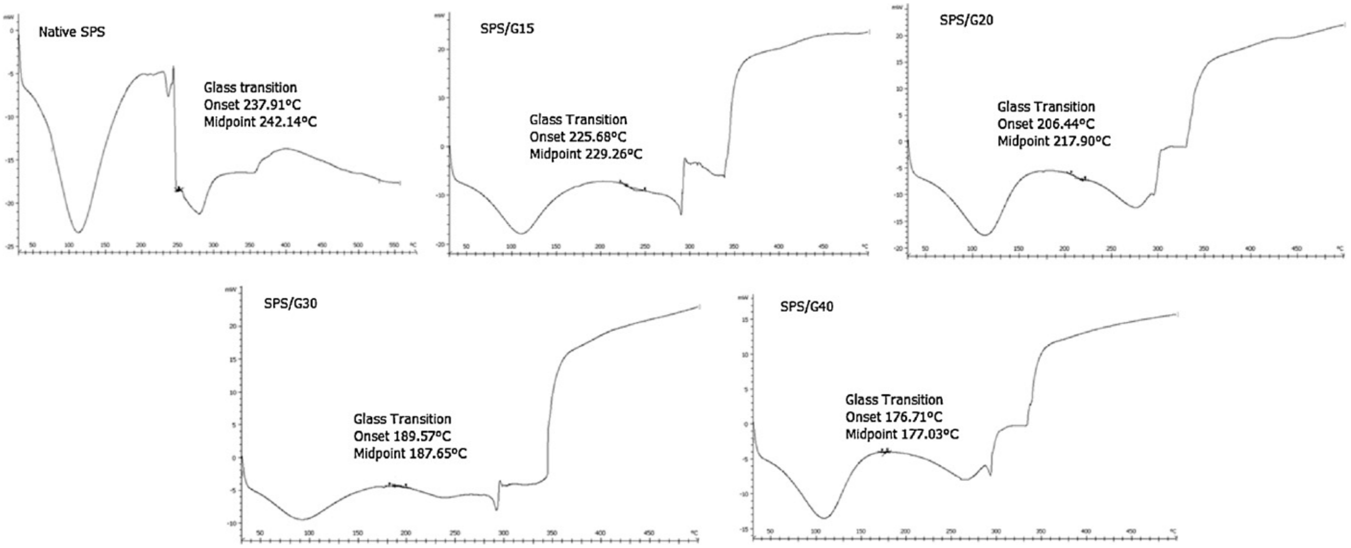

Fig. 1. DSC curves of plasticized SPS.

consequently change the overall cohesion, leading to the reduction of Tg. Mitrus (2005) has been claimed that the plasticizer decreased Tg because it facilitates chain mobility. Brittleness is one of the major problems connected with starchy material due to its high Tg (De Graaf, Karman, & Janssen, 2003). In the absence of plasticizers, starch is brittle. The addition of plasticizers overcomes starch brittleness and improves its flexibility and extensibility of the polymers.

Fig. 2 presents the thermogravimetric (TG) analysis results of plasticized SPS. The behavior of the mass loss curves was similar for all plasticized SPS. Degradation of the molecular structure occurs when plasticized SPS is heated. The extent of the changes induced depends on the temperature and under extreme conditions, may result in a complete loss of carbohydrate character. Because the boiling points of the plasticizer are more than 100 °C, the mass loss below 100 °C was mainly ascribed to water loss (Ma & Yu, 2004). Meanwhile, the mass loss below 290 °C indicated the vaporization of glycerol. Pure glycerol has a boiling point of 290 °C (Acros Organics, 2006) but in this case, starch possesses hydrophilic nature with helical structure of all hydroxyl groups that are directed outside of the ring (Imam, Gordon, & Greene, 1996). This structure promotes direct interactions of starch and glycerol by forming hydrogen bonds and subsequently lowers the volatility of glycerol in plasticized SPS. The large degradation at 310 °C appears to involve further elimination of the polyhyroxyl groups, accompanied by depolymerization and decomposition (Aggarwal & Dollimore, 1998). It has been documented earlier that at higher temperatures, depolymerization of the macromolecules takes place with the formation of b-(1,6) anhydro D-glucopyranose (levoglucosan), 2-furaldehyde (furfural) and a range of lower molecular-weight volatile and gaseous fragmentation products. A carbonaceous (substance rich in carbon) residue remains after all the volatile products have been driven off (Marchessault, Taylor, Fyfe, & Veregin, 1985).

3.2. Mechanical properties

Fig. 3 shows the tensile stress–strain of plasticized SPS which is SPS/G15, SPS/G20, SPS/G30 and SPS/G40. From the figure, it is found that SPS/G30 and SPS/G40 showed the typical pattern of rubbery starch plastic materials due to linearly increase in tensile stress at low strain and followed by the curved toward the strain axis until failure occurs (Soest & Knooren, 1997). It is also found that SPS/G15 and SPS/G20 exihibited the brittle properties compared to others due to failure occurs at the lower stress (<1.5 MPa) and lower strain (<2%). The addition of plasticizers at 30 wt% and 40 wt% overcomes the starch brittleness and improves their flexibility as shown in Fig. 3.

Fig. 4 shows the tensile strength of plasticized SPS. From the figure, it can be seen that the tensile strength of SPS/G30 showed the highest value 2.42 MPa compared to the other concentration of the plasticizer. The higher the concentration of the plasticizers, the higher the tensile strength of plasticized SPS and optimum concentration were 30 wt%. The tensile strength decreases to 0.5 MPa when concentrations of plasticizer were 40 wt%. As the

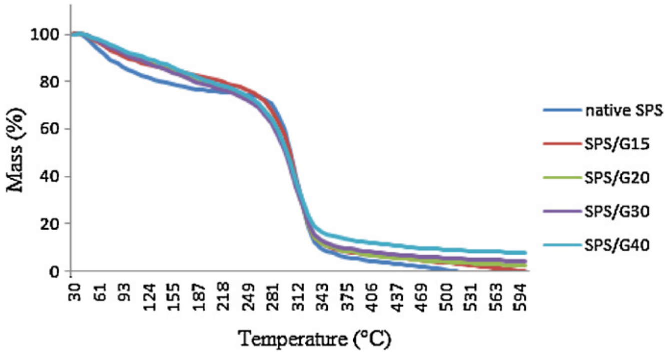

Fig. 2. TG curves of plasticized SPS.

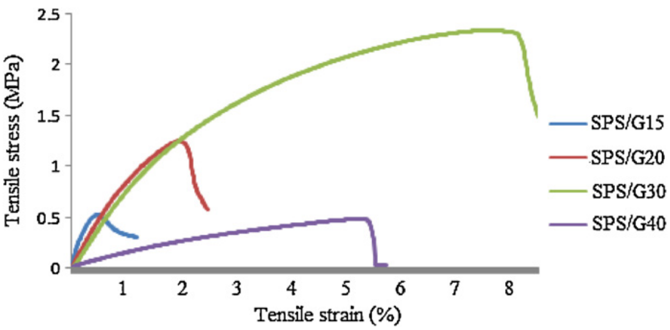

Fig. 3. Tensile stress–strain of plasticized SPS.

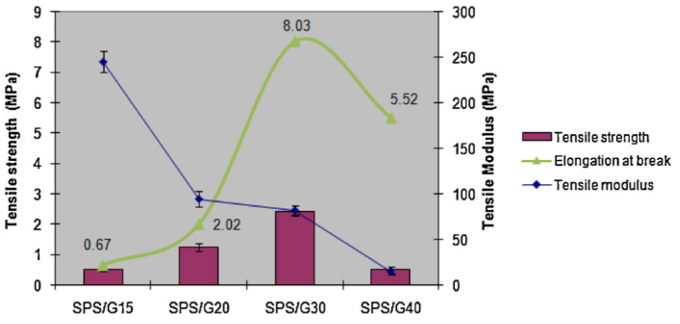

Fig. 4. Tensile strength of plasticized SPS.

plasticizer content increased to 40%, not enough SPS to be well bonded with glycerol and thus poor adhesion occurred which reduce the mechanical properties of SPS/G40. This result well agreed with the finding of Laohakunjit and Noomhorm (2004) who claimed that the films outside this range are either too brittle (<20 wt%) or too tacky (>45 wt%).

The tensile modulus or elastic modulus of plasticized SPS is also shown in Fig. 4. From the figure, it is clearly shows that the tensile modulus decreases from 245.24 MPa to 15 MPa with the increasing of plasticizer concentration from 15 to 40 wt%. Meanwhile, for elongation at break analysis, it is found that the relative % elongation increased significantly (0.67–8.03%) with increasing content of plasticizer from 15 wt% to 30 wt% as shown in Fig. 4. From this investigation, it is interesting to know that 30 wt% of glycerol made a very high elongation but the elongation dropped to 5.52% when the glycerol was used at 40%. Similar finding was reported by Laohakunjit and Noomhorm (2004) where they found that the relative % elongation increased significantly with the increasing concentration of plasticizer for both plasticizer (sorbitol and glycerol) and the elongation was dropped when the glycerol was used at 35%. The increasing percent elongation of plasticized SPS can be ascribed to the nature of the glycerol molecule, which is relatively hydrophilic due to large number of hydroxyl groups which can establish hydrogen bonding. Generally, as the plasticizer increase, the tensile strength and elongation of plasticized SPS increase, while the tensile modulus decreased. These phenomena indicate that the plasticized SPS is more flexible when subjected to tension or mechanical stress. The results from this study prove the finding done by Beerler and Finney (1983) whereby they reported that the plasticizers such as glycerol will interfere the arrangement of the polymer chains and the hydrogen bonding. It is also most likely affect the crystallinity of starch by decreasing the polymer interaction and cohesiveness. Thus, this make the plasticized SPS become more flexible with the increasing of glycerol.

3.3. *Water absorption*

Moisture absorption result of plasticized SPS is shown in Fig. 5. All specimens were stored in the humidity chamber at 75% RH for 72 h and data were collected at specific time interval. Moisture adsorption was more rapid at the initial stages of the storage and lower amounts of water were adsorbed as time increased. The moisture content of plasticized SPS reached a plateau indicating that they became equilibrated with storage RH, following a Fickian diffusion process. In general, the water absorption of the plasticized SPS decreased with the increase of glycerol. The decrease of the water absorption in plasticized SPS indicated that glycerol was a better water resistant material where less water gain was observed in the plasticized SPS. This can be attributed to the fact that the glycerol formed stronger hydrogen-bond with sugar palm starch which restrained the water molecule to combine to the plasticizer or to the sugar palm starch (Lieberman & Gilbert, 1973).

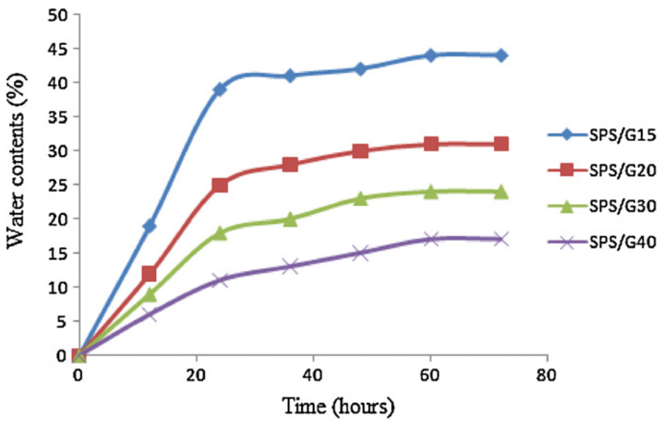

Fig. 5. Moisture absorption of plasticized SPS.

The result also showed that when the glycerol content was higher, stronger hydrogen-bond formed between plasticizer and the sugar palm starch. It was harder for water molecule to penetrate into the plasticized SPS and as a result, plasticizer–water interactions and starch–water interactions were very weak relatively, so that the plasticized SPS presented lower water absorption at the high glycerol concentration.

3.4. *Fourier transform infrared (FT-IR) spectroscopy*

FT-IR spectra for plasticized SPS with different concentration of glycerol are shown in Fig. 6. The intense peaks at 3200–3500 cm^{-1} shown in figure indicated the presence of O—H groups in all parts of plasticized SPS. This proves that the hydroxyl groups of the plasticized SPS decreased with the increase of glycerol. The strong peaks at 2850–3000 cm^{-1} assigned to the C—H stretching and peaks at 990–1030 cm^{-1} were characteristic of the anhydroglucose ring O—C stretch (Fang, Fowler, Tomkinson, & Hill, 2002). The peaks at around 1200 and 1450 cm^{-1} were characteristic of C—O—H. While, peaks at 1500–1600 cm^{-1} attributed to the bending of water in starch (Park, Im, Kim, & Kim, 2000).

3.5. *Scanning electron microscopy (SEM)*

The fracture surface analysis of plasticized SPS was done by SEM. The SEM images shown in Fig. 7 which clearly revealed that the smooth surface increase in plasticized SPS caused by the addition of glycerol. At very high concentrations glycerol (30 and 40 w/w%), the specimens were very soft, whereas at lower glycerol concentrations, the specimens were very brittle. The plasticizer effectively reduced internal hydrogen bonding while increasing

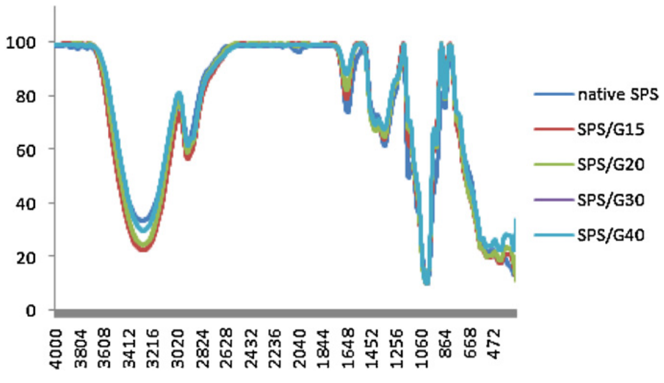

Fig. 6. FT-IR spectra of plasticized SPS.

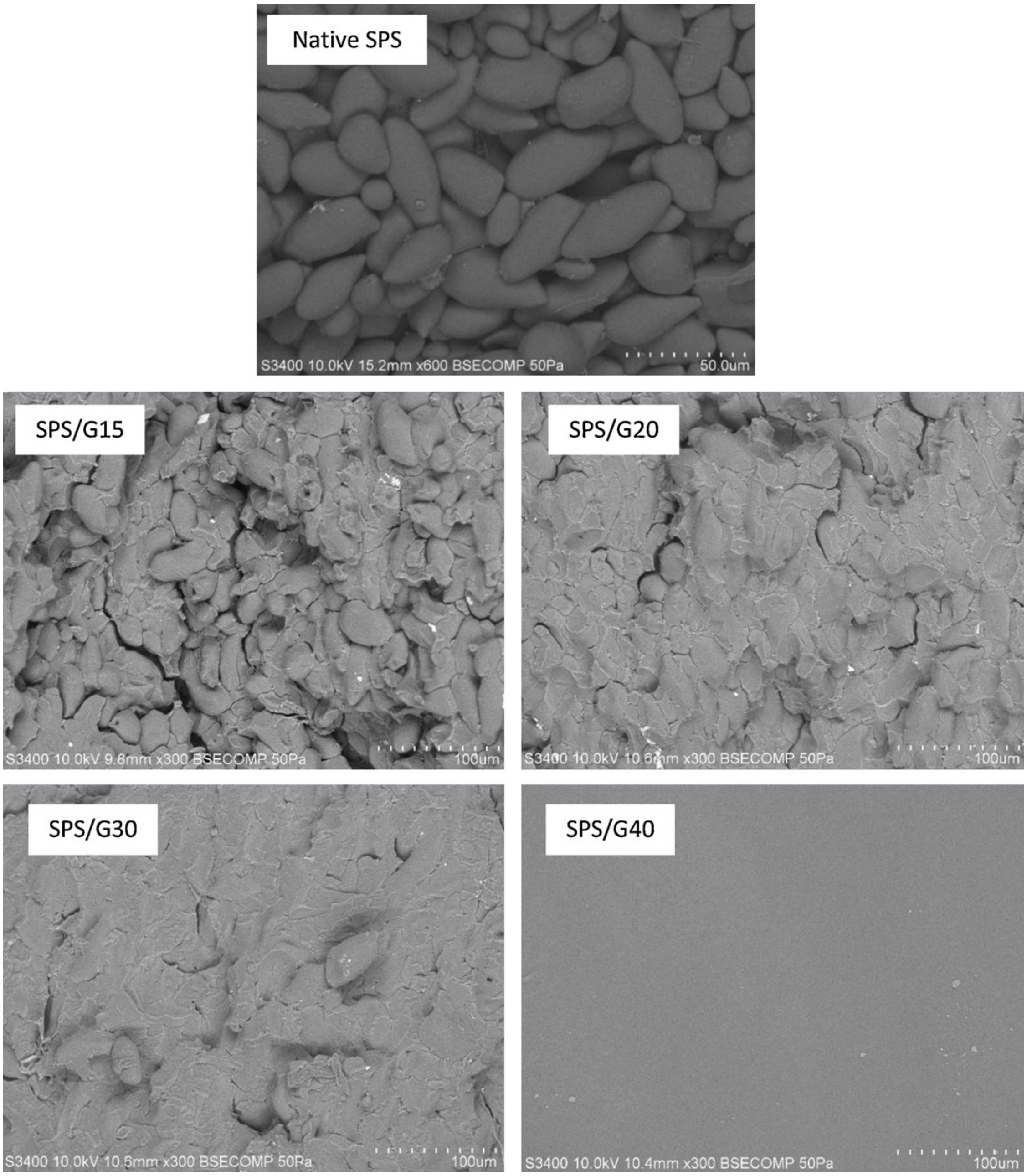

Fig. 7. SEM images of plasticized SPS.

intermolecular spacing, thereby decreasing brittleness (Lieberman & Gilbert, 1973). Similar results have been reported by Mathew and Dufresne (2002) for sorbitol and maltitol plasticized waxy maize.

4. Conclusions

Thermoplastic starch derived from sugar palm tree was successfully developed and effect of plasticizer to the thermal, mechanical and physical properties were investigated. The result showed that the addition of glycerol decrease the Tg of plasticized SPS. The mechanical properties of plasticized SPS increased with the increasing of glycerol until the optimum amount of 30 w/w%. Meanwhile, the water absorption of plasticized SPS decrease with increasing of glycerol.

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