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Barrier and Seal Properties of Reactive Blending of Poly(butylene succinate) Based Blends

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Abstract

Poly Butylene Succinate (PBS) is one of the biodegradable polymers with good physical properties but its barrier properties such as the oxygen barrier are poor. The aim of this study was to investigate the barrier and seal properties of PBS by blending with biodegradable poly hydroxybutyrate-co-hydroxyvalerate (PHBV). PBS and PHBV (80/20 and 70/30 %w/w) with and without reactive agents were prepared using an internal mixer. Film specimens of 100 micrometers in thickness were prepared using compression molding. The morphology, barrier properties and peel-seal strength were investigated. Morphological observations using scanning electron microscope (SEM) showed an improved dispersion of PHBV in the blends after adding the reactive agents. The oxygen barrier and water vapor barrier were determined using ASTM D3985 and ASTM E96, respectively. The results showed that the addition of PHBV into the blend films led to lower OTR and WVTR when compared to the neat PBS. The addition of reactive agents can further improve the OTR and WVTR of PBS/PHBV blends due to the compact and dense structure of the films. Peel-seal behavior of the films was examined by the different sealing temperatures, which determined the failure mechanism after peeling. The blend films with reactive agents after sealing temperature between 105 and 115°C were peeled from the substrate with adhesive and cohesive failures showing the easy peel mode.

Keywords: Poly(butylene succinate), Poly(hydroxybutyrate-co-hydroxyvalerate), Reactive blending film, Barrier properties, Peelable film

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1. Introduction

The growing environmental concern on plastic pollution has gained global attention. It was reported that more than 340 million tons of plastic waste were generated around the world, with around 46% of this waste from the packaging sector in 2018 (Wu, Misra, & Mohanty, 2021). For the food packaging applications, it is a single-use packaging alone that generates a large amount of plastic wastes. Amongst these, the plastic top seal for sealing trays is one of the interesting items, which is difficult to be replaced with the sustainable option. Top seal is mostly made from non-biodegradable polyolefins and poly(ethylene terephthalate) (PET) or even multilayer films. They are not easily recycled and usually sent to landfills after using. With the high material property requirements of these plastics including a light weight (micro-scale thickness), high toughness, complex structures (multi-layer) and contamination due to direct contact with foods (Wu et al., 2021), the collection, separation and recycle are very complicated. Urgent efforts are directed towards finding alternative solutions, including the use of renewable, biobased, and biodegradable plastics (Rodriguez-Uribe et al., 2021). One of the options can be the compostable plastics as they offer the composting option, especially for a food contact packaging, where recycling was complexed by the economics of scale and waste management. For food packaging uses, the poor water vapor and gas barrier properties of most biodegradable plastics are the major limitations for top seal applications. The main approach to improve this performance of biodegradable plastics includes polymer blending. The method is easily adopted in the industrial processes (Wu et al., 2021).

Poly butylene succinate or PBS is one of the biodegradable aliphatic polyester and is synthesized from 1,4-butanediol and succinic acid monomers by the polycondensation reaction. PBS has mechanical properties comparable to polyolefins like polyethylene (PE) and polypropylene (PP) (Muthuraj, Misra, & Mohanty, 2017). On the other hand, the disadvantage of PBS with packaging application is its poor barrier to moisture and oxygen (Rodriguez-Uribe et al., 2021). A study conducted by V. Siracusa et al. (Siracusa, Lotti, Munari, & Dalla Rosa, 2015) reported the oxygen transmission rate (OTR) of PBS films prepared by compression molding and with a thickness of 144.7 µm was ~281 $cm^3.m^{-2}.day^{-1}.bar^{-1}$. J. Xu (Xu, 2015) reported the water vapor transmission rate (WVTR) of PBS films made by using hot-press and with a thickness of 51 µm was 83.8 $g.m^{-2}.day^{-1}$. To obtain high oxygen/water vapor barrier blends, one component with a high barrier should be incorporated. Accordingly, the polymer blending by high barrier properties materials can result in products with better barrier properties. Among biopolymers, Polyhydroxybutyrate-co-hydroxyvalerate (PHBV) as a family of Poly(hydroxyalkanoates) (PHAs) has relatively high barrier properties and proven biodegradability has attracted the attention of the packaging application. This group of polymer is a polyester derived from microorganism, including bacterial fermentation of starting raw sugars or lipids (Nuchanong, Seadan, Khankrua, & Suttiruengwong, 2021; Rodriguez-Uribe et al., 2021). Since the oxygen barrier of PHBV is much better than most biopolymers such as PBS, Polylactic acid (PLA), Polycaprolactone (PCL), and some commodity polymers such as PP and PE, it is a good candidate for blending. This polymer is however still very expensive. Although the water vapor barrier of PHBV was inferior to that of PP and PE, it is clearly better than that of biodegradable polyesters such as PBS, PLA, PBAT and PCL (Rodriguez-Uribe et al., 2021). Thus, PHBV can help to enhance the barrier properties of biopolymer blends. The poor barrier of PLA films has been found to improve with the addition of PHBV by I. Zembouai et al. (Zembouai et al., 2013). The OTR of PLA was decreased by about 35.3, 43.2 and 81.5% with the addition of 25, 50 and 75 wt% of PHBV for PHBV/PLA blend, respectively. On the other hand, the value of WVTR of PLA was decreased by about 22.7, 36.6 and 58.9% by addition of 25, 50 and 75 wt% of PHBV, respectively. M. Cunha et al. (Cunha, Fernandes, Covas, Vicente, & Hilliou, 2016) studied the melt blended PHBV/PBAT (70/30). It was reported that WVP of the film was close to PHBV, but the elongation at break of the film showed the low flexibility. The compatibility between PBS and PHBV was poor, resulting in relatively large particle size and a weak interfacial adhesion in their blends by Y.J. Phua et al. (Phua, Pegoretti, Medeiros Araujo, & Mohd Ishak, 2015) and P. Ma. et al. (Ma, Hristova-Bogaerds, Lemstra, Zhang, & Wang, 2012). Certain compatibility needs to be introduced into the blend system in order to acquire better performance. To achieve oxygen/water vapor barrier and mechanical balance in biodegradable polymer blends, effective compatibilizers were normally applied (Wu et al., 2021).

Therefore, the objective of this work was to study the addition of reactive compatibilizer in 80/20 and 70/30 wt% of PBS and PHBV blend. The polymer blend with and without reactive compatibilizer was evaluated by oxygen transmission rate (OTR), water vapor transmission rate (WVTR) and seal strength test for study the possibility to use with top seal application.

2. Materials and Methods

A commercial grade polybutylene succinate (PBS) FZ91PM with MFR 5 g/10 min, density 1.26 g/cm^3, melting temperature around 115°C was purchased from PTT MCC Biochem Co. Ltd. Thailand). Polyhydroxybutyrate-co-hydroxyvalerate) (PHBV) Enmat Y1000P with 3 mol% hydroxyvalerate (HV) content was purchased from Tianan Biologic Material Co. (Ningbo, P. R. China). It has the density of 1.25 g/cm^3, melting temperature around 175-180°C. Multi-functional epoxy chain extender (ECE) Joncryl ADR 4468 in solid flake form with epoxy equivalent weight 310 g/mol was purchased from BASF Co., Ltd.

2.1 Blend preparation

PBS and PHBV pellets were dried at 60ºC overnight before further use. The ratios of PBS and PHBV were 80:20 and 70:30 by weight. For reactive blending, PBS/PHBV with various contents of ECE were studied at 0.1, 0.2 and 0.3 phr. PBS/PHBV blends with and without reactive agents were melt-blended using an internal mixer at 180°C with rotor speed of 100 rpm for 10 min. Films were prepared using compression molding at 190ºC for 3 min to obtain films of about 100 µm thickness.

2.2 Characterization

Scanning electron microscopy (SEM). To investigate the morphology of PBS/PHBV blends with and without reactive agents, all blended samples were cryo-fractured in liquid nitrogen and sputter coated with Au/Pd. The fracture surface was investigated by Field emission scanning electron microscopy (FESEM) (MIRA3, TESCAN) with a chamber pressure <0.009 Pa under high vacuum mode. A 2500×magnification was used for all samples.

Oxygen transmission rate (OTR). The oxygen transmission rate (OTR) was carried out according to ASTM D3985 using Gas Permeability Tester (type GDP-C, Brugger Feinmechanik GmbH). OTR measurements were performed at 23°C at 50% of RH, and the results were expressed in cm^3.m^{-2}.day^{-1}.bar^{-1}.

Water vapor transmission rate (WVTR). The water vapor transmission rate (WVTR) of the film samples was studied using the "Cup method" referring to ASTM E96. Silica gel was used as a desiccating agent, and the cups had a specific exchange surface of S = 4.59 cm^2. The experimental setting consists of a cylindrical vessel filled with a desiccating agent and sealed with the investigated film. The mass increase in the cups, due to the water absorption of silica gel, was plotted against time. The WVTR was calculated from the slope of the mass uptake of desiccating agent profile versus time at 23°C at 75% of RH, and the results were expressed in g.m^{-2}.day^{-1}.

Seal-Peel test. Heat sealing process involves sealing temperature between 95 and 110°C and sealing time 8 s (18 rpm.) were directly set on a continuous sealing machine (BSV-5I, Tupack Co., Ltd.). All of the films were sealed on a PBS blend sheet. The sheet prepared from PBS/PLA/PBAT was about 800 µm thickness to imitate the container rim or the tray's lip. Peel strength of films was measured by a supported 180° test (Technique C) according to ASTM F88. The grip distance and peeling rate was 25.4 mm and 200 mm/min, respectively. The testing machine was a universal testing machine (NRI-TS500-5B, Narin instrument Co., Ltd.) with a load cell of 5 kN. Peel strength values were the average values of five repeated measurements.

3. Results and Discussion

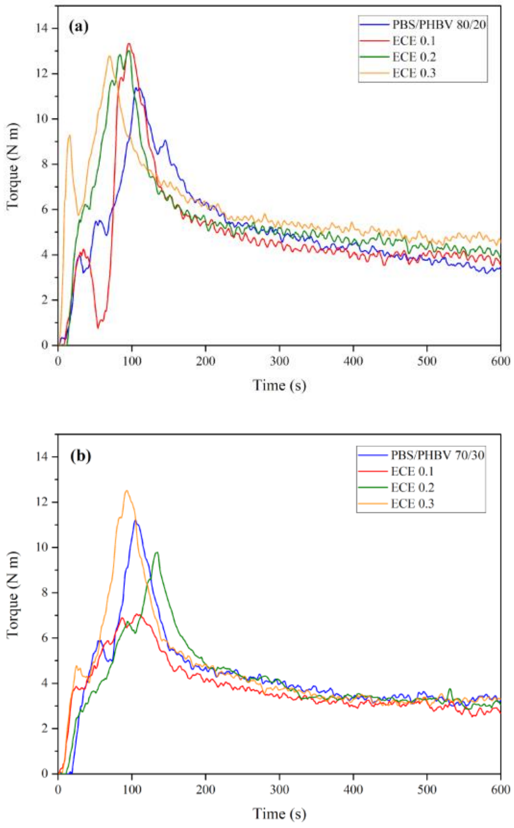

Figure 1. Mixing torques of PBS/PHBV (a) 80/20 (b) 70/30 blend with and without reactive agents at 180ºC and rate 100 rpm.

Mixing torque values. The mixing torque of the internal mixer can be related to the molten polymer viscosity. The evolution of PBS/PHBV 80/20, 70/30 blend with and without reactive agents mixing torque curves is shown in Figure 1. The mixing torque curve of the non-reactive PBS/PHBV 80/20 blend is lower than that of the reactive PBS/PHBV 80/20 blend as a result of the reaction between ECE epoxide group and carboxylic end group of polyester through condensation reaction, indicating some degree of chemical reaction. This finding is consistent with other researches that also modified polyester through condensation reaction (Nanthananon, Seadan, Pivsa-Art, Hiroyuki, & Suttiruengwong, 2017; Nuchanong et al., 2021; Phuangmali, Seadan, Khankrua, & Suttiruengwong, 2021; Yang, Xin, Mughal, Li, & He, 2017). The reactive epoxide groups can react with carbonyl groups in both polyesters to form copolymer, leading to better compatibility between two polymers. For PBS/PHBV 70/30 blend (Figure 1 b), the mixing torque was not different when adding and increasing ECE contents. The reason for this might be the expense of two or more competing reactions of PHBV, such as the competition between the degradation reaction and condensation reaction (Nuchanong et al., 2021). Therefore, the change in the viscosity of the melt is not pronounced.

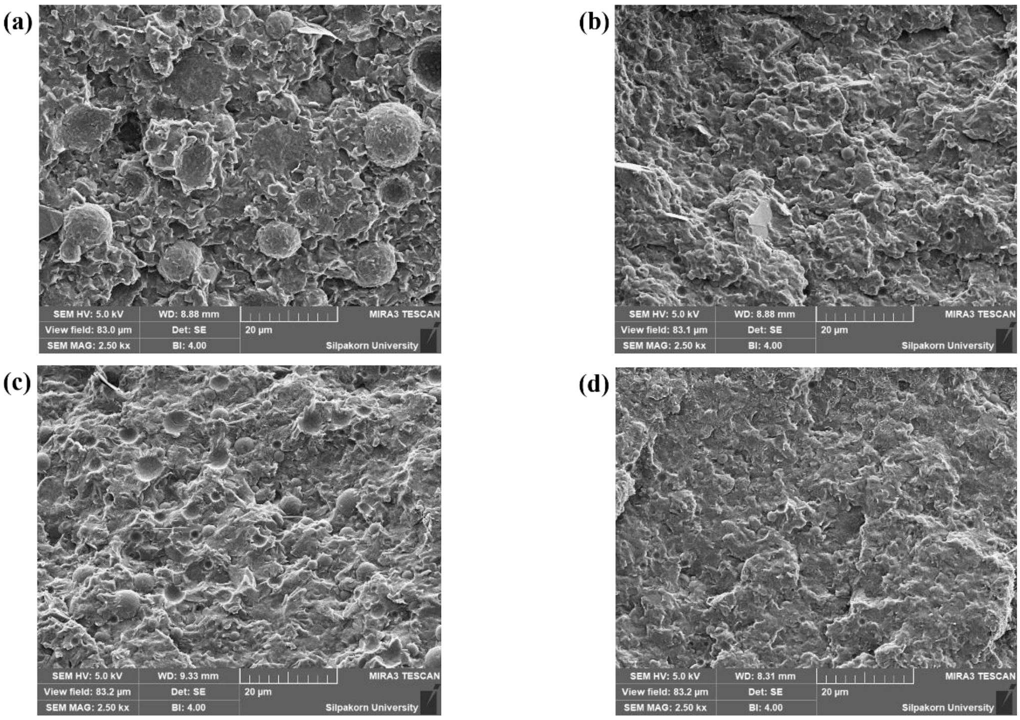

Figure 2. SEM micrographs of cryo-fracture surface of (a) non-reactive PBS/PHBV 80/20 and reactive PBS/PHBV 80/20 with ECE (b) 0.1, (c) 0.2 and (d) 0.3 phr.

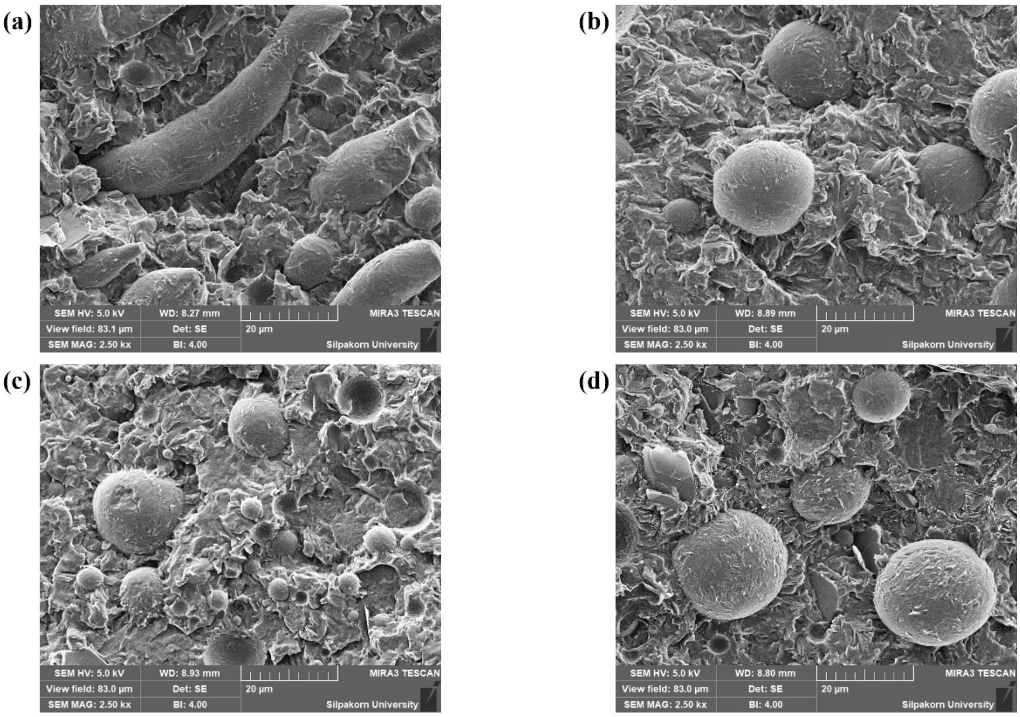

Figure 3. SEM micrographs of cryo-fracture surface of (a) non-reactive PBS/PHBV 70/30 and reactive PBS/PHBV 70/30 with ECE (b) 0.1, (c) 0.2 and (d) 0.3 phr.

Morphological observation. The micrographs of the PBS/PHBV blends are studied by SEM. Figure 2 shows the SEM micrographs of the cryo-fractured surface of the non-reactive and reactive PBS/PHBV (80:20) blends. The physical PBS/PHBV blends (Figure 2 a) shows that PHBV droplets are dispersed in the PBS matrix with an average droplet size of about 10 μm. After addition of ECE to the PBS/PHBV blends (Figure 2 b - d), the sizes of PHBV droplets are smaller than non-reactive one. The particle sizes are about 2 μm. It is evident that there was a reaction through the condensation reaction when adding ECE into the blends. Similar observation was made by I. Zembouai et al. (Zembouai et al., 2013). They reported that PHBV and PLA form mainly a two phase-system. In the 25/75 blend, the phase was formed by PHBV and the inclusions (bead-shaped) were PLA domains. P. Ma and coworker (Ma et al., 2012) also reported after adding of the reactive agent to the PHBV/PBS melts, which acted as compatibilizer between PHBV and PBS and a network consisting of both PHBV and PBS were formed.

In case of the cryo-fractured surfaces of PBS/PHBV (70:30), PHBV is typically dispersed in a form of the filament in the PBS matrix as shown in Figure 3 a. In Figure 3 b, PBS/PHBV (70:30) blend with the addition of the reactive agent showed the spherical particles instead of the filament shape. The filament changes to spherical droplets with a larger particle size of about 20 μm and the smaller about 4-5 μm. The interfacial adhesion between both polymers for both compositions of the blends as depicted in Figure 3 c and Figure 3 d after adding ECE.

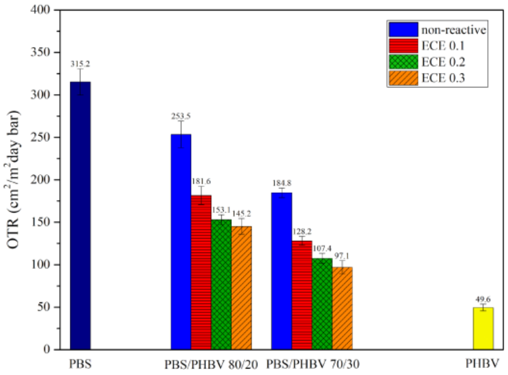

Figure 4. Oxygen transmission rate (OTR) of PBS/PHBV blend with and without reactive agents.

Barrier properties. Gas barrier properties of PBS/PHBV blends were investigated by means of oxygen and water vapor transmission rate tests. Evaluations were performed on compressed films with thickness about 100 µm. Similar trends are observed for both gas permeation evolutions as a function of compositions.

Oxygen transmission rate (OTR). The results are depicted in Figure 4 PHBV possesses a good oxygen barrier when compared to PBS. OTR values of PBS/PHBV blend are decreased with increasing PHBV contents in the blends. When adding ECE, the OTR value decreased furthermore when compared to non-reactive PBS/PHBV blends. OTRs of PBS/PHBV 80/20 and 70/30 blend are improved by 54 and 69.2% after the addition of ECE 0.3 phr, respectively compared with that of PBS. The reason for this is still unknown. This shows clearly the role of PHBV as an efficient barrier promoter for PBS, even at the low content. The lower OTR values obtained after adding ECE suggest an improved interfacial adhesion between both polymers helps create the compact and dense structure of the films (Chinga-Carrasco & Syverud, 2012).

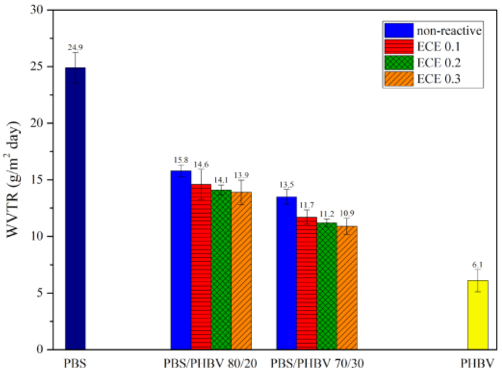

Figure 5. Water vapor transmission rate (WVTR) of PBS/PHBV blend with and without reactive agents.

Water vapor transmission rate (WVTR). WVTR results are presented in Figure 5. It is seen that by increasing PHBV contents the water vapor barrier was better when compared to PBS alone. PBS/PHBV blends show the decrease in WVTR values with increasing PHBV contents. Moreover, OTR of reactive PBS/PHBV blends exhibit lower than that of non-reactive blends. WVTR values of PBS/PHBV 80/20 and 70/30 blend compared with PBS are improved by 44 and 55.6% after the addition of ECE 0.3 phr, respectively. The incorporation of PHBV can improve the moisture barrier properties of PBS. This was also in good agreement with I. Zembouai (Zembouai et al., 2013), who reported the barrier property of PHBV and PLA blend. After improving the barrier properties of the blends, it was found that barrier properties lied between PLA and PHBV barrier properties. It was reported that the decrease in barrier properties as crystallinity increased was mainly explained by two factors. The first was the inclusion of impermeable crystallites which decreases the amount of amorphous phase through which the gas molecules can permeate. The second was that impermeable crystals increase the tortuosity of the transport path (Zembouai et al., 2013).

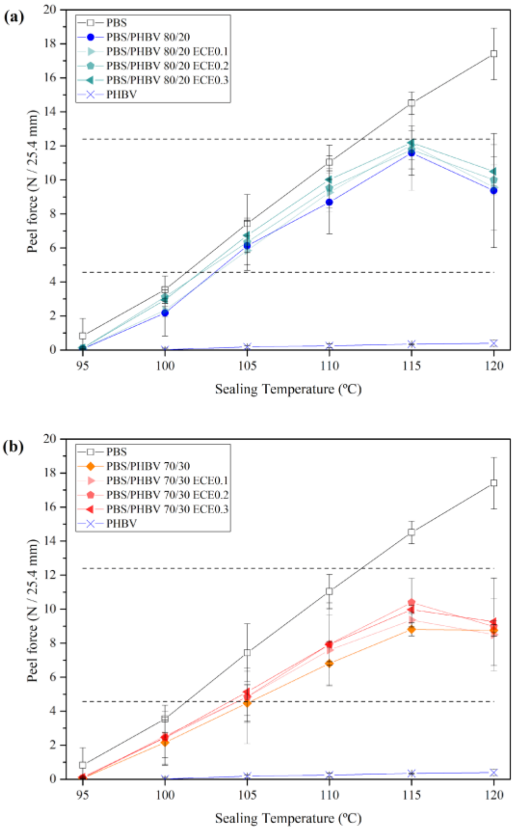

Figure 6. Peel force of (a) PBS/PHBV 80/20 blends and (b) PBS/PHBV 70/30 blends with and without

ECE in different sealing temperatures on PBS blends sheet.

Seal-Peel test. In this research, PBS/PHBV films were fabricated and used to investigate the peel properties as for an easy-peel range application. The peel force of the film sealed on the prepared PBS blend sheet was carried out to evaluate the interaction between PBS/PHBV films and PBS blend sheet. From the seal and Peel test shown in Figure 6, Peel force tends to decrease with increasing PHBV contents. The compact and dense structure of the films could be the reason for this. On the other hand, depending on the preparation of the films (in this case the compression molding was used), PHBV minor phases can reside at the surface because of its low interaction with PBS (Liewchirakorn, Aht-ong, & Chinsirikul, 2017). Since the heat seal was studied, optimum heat seal temperatures of some of the blends may fall out of this range. Therefore, the blends could be used in packaging applications with easy-open peelable features (in-dash lines), which have a peel force of 4.45-12.24 N/25.4 mm (1 to 2.75 lb/in as reported by reported by J. Zhang et al.) (Diaz, Pao, & Kim, 2016; Zhang et al., 2009). However, all blends film at sealing temperature 105 and 115ºC had a tendency to be used as peelable film for easy-peel films.

Peel–seal behaviors in easy-peel range with different sealing temperatures (105-115ºC) peeling film from PBS blend sheet substrate (800 µm). Three types of the film's failure (Liewchirakorn et al., 2017) observed in this study are schematically illustrated in Figure 7 a to c. Adhesive failure was found in sealed PBS/PHBV 80/20 blend at 105-110 ºC sealing temperatures and in 70/30 blend at 105 ºC sealing temperatures on PBS blend substrate. This failure is located at the interface between the film and substrate, resulting in the lowest peel force values. For the cohesive failure, the film tends to peel off from the substrate and peel advances during the test. There is some residue observed on both peeled surfaces of PBS blend substrate and PBS/PHBV blend films. This found in the case of the PBS/PHBV 80/20 and 70/30 blend films were sealed at 115ºC, 110ºC, respectively. The films demonstrate tearing failures at a high sealing temperature of 115°C. PBS/PHBV 70/30 blend films exhibit the complete tear. This tearing failure means no peel during the peeling process due to a high seal force between film and substrate (or seal strength is higher than the inherent tensile strength of the film).

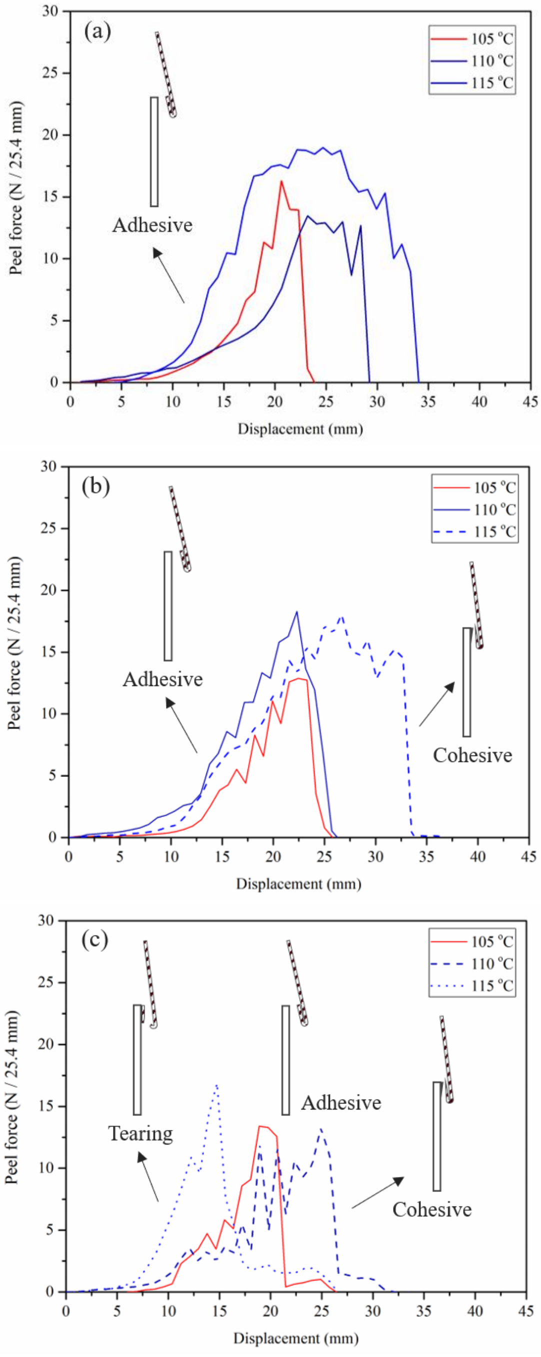

Figure 7. Typical peel failure curve of (a) PBS, PBS/PHBV blends (b) 80/20 and (c) 70/30 in different sealing temperatures on PBS blends sheet.

4. Conclusions

Biodegradable PBS and PHBV blends were prepared by melt mixing with and without ECE. Film specimens were prepared using compression molding. The SEM images indicated the decreased PHBV dispersed phase as well as improved interfacial adhesion after adding ECE in PBS/PHBV blends, indicating improved compatibility between these polymers. The addition of PHBV into the blend films without ECE altered the barrier properties, where the decrease in both OTR and WVTR when compared with the neat PBS was observed. Meanwhile, the addition of ECE can further improve the OTR and WVTR of PBS/PHBV blends due to the compact and dense structure of the films. PHBV could act as an efficient barrier promoter for PBS. For the seal-peel strength test, after sealing temperature between 105 and 115°C that exhibited easy-peel characteristics. Peel–seal behaviors in an easy-peel range were peeled from the PBS blend substrate, adhesive and cohesive failures were apparent during the peeling process. It can be concluded that the blend film with PHBV minor phase with ECE can be used for top seal application.

5. Acknowledgement

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