Characterization of ethylene–vinyl acetate (EVA)/modified starch expanded compounds for outsole material

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Submitted: 24 March 2021

Revised: 12 April 2021

Accepted: 17 May 2021

ABSTRACT

The use of non-biodegradable material in shoe components has negative impacts on environmental sustainability when disposed into landfills due to poor biodegradability. This study prepared ethylene–vinyl acetate (EVA)/modified starch expanded compounds as outsole material to overcome environmental pollution. This research aimed to investigate the effects of EVA/modified starch ratio on the properties of the compound. Ethylene–vinyl acetate (EVA) copolymer was blended with a modified cassava starch in various amounts (10–50 phr) using a two-roll mill. Effect of modified starch content was characterized its tensile strength, elongation at break, tearing strength, 50% permanent set, density, abrasion resistance, flex resistance, biodegradability, and morphology. The optimum value of tensile strength (61.33 kg/cm²), elongation at break (895%), and tear strength (16.62 N/mm) were obtained for samples containing 20 phr modified starch. The EVA compound containing 30 phr of starch showed the optimum 50% permanent set (4.85%) and the highest abrasion resistance with the smallest volume loss of 439.99 mm³. The addition of modified starch up to 50 phr provided good flexural resistance to 150000 number of cycles. The morphology image showed that distribution of modified starch particles was not homogeneously dispersed in the EVA/modified starch expanded compound. Incorporation of modified starch in EVA compound was improving its biodegradability.

Keywords: biodegradability, EVA, modified starch, morphology, outsole, physical properties.

INTRODUCTION

The use of non-biodegradable material in shoe components has negative impacts on environmental sustainability when disposed into landfills because of its poor biodegradability. Therefore, the researchers strive to produce shoe components materials that environmental friendly. The outsole is one of the important shoe components that directly contact with the ground. In the footwear industry, ethylene–vinyl acetate copolymer (EVA) is widely used in the manufacture of shoe components including outsoles and insoles (Lopes *et al.*, 2015), which brings several benefits such as cost reduction, comfort, lightness, and softness (Ferreira, 2018).

Ethylene Vinyl Acetate (EVA) is a copolymer consisting of ethylene and vinyl acetate (Sisanth, 2017, Brito e Dias *et al.*, 2018). The end property depends on vinyl acetate (VA) content; VA content usually varies from 10 to 40% (Nautiyal, 2012),

the elastomeric grade of EVA consists of VA content varying from 40–60 wt % (Sisanth, 2017). The application of EVA for footwear has been reported by many researchers. Liu (2017) reported the application of EVA as the support layer shock absorption material for basketball shoes. A study related to EVA as a material for shoe midsoles has been reported by Wang (2012) and Onodera (2017). Lopes *et al.* (2015) reported natural and synthetic rubber/waste–EVA (Ethylene–vinyl acetate) composites for sustainable application in the footwear industry. The production of many petroleum-based products, such as EVA, used to manufacture footwear results in serious environmental pollution when disposed of into landfills because of its less biodegradability. To increase the biodegradability of EVA, natural polymers were added to make a mixture. Starch is popular for its ability to be fully biodegradable in land and water, as well as elevates the

biodegradability of non-biodegradable plastics (Gautam & Kaur, 2013; Tanase *et al.*, 2016; Sessini *et al.*, 2019).

The biodegradability of starch and its low cost are some points that make increasement of starch application in the polymer industry. It also fits in with the eco-friendly trends supported by law regulations. Starch can be modified into thermoplastic starch (TPS) to enhance its processing and potential applications. The most common plasticizers which added to make TPS are the mixture of water and glycerol (Obashi and Igwe, 2014; Maran *et al.*, 2014; Mendes *et al.*, 2016). According to Valle *et al.* (2018), TPS has been blended with many polymers such as polyvinyl alcohol (PVA), polylactic acid (PLA), Poly(ϵ -caprolactone) (PCL), polyhydroxybutyrate (PHB), and other polymers. Blend of TPS with EVA in food packaging applications have been reported by Da Roz *et al.* (2012), Rodriguez-Perez *et al.* (2012), and Sessini *et al.* (2019). To our knowledge, the blends of EVA/TPS for outsole materials have not been reported by researchers.

In the footwear industry, EVA is one of the materials which popularly known as expanded rubber or foam rubber. It is necessary to make a mixture to obtain an expanded compound that will determine the required material characteristics so EVA can be molded on the outsole process. The expanded compound processing is usually associated with the incorporation of blowing agents during compound processing. Blowing agents commonly used for the formation of cellular structures in polymeric matrices can be physical, such as liquids with low boiling points, or solid chemical compounds that decompose under heat by releasing gases such as nitrogen and carbon dioxide (Zimmermann *et al.*, 2014). Azodicarbonamide is the most popular blowing agent to produce EVA. The solid form of azodicarbonamide deteriorate into gas when its heated. The gas then extends to form tiny bubbles when its confined inside the semi-liquid plastic EVA compound. This study aimed to investigate the effects of EVA/modified starch ratio on the properties of EVA/modified starch expanded compound.

MATERIALS AND METHODS

Materials

Rubber grade EVA (Levapren 500) with vinyl acetate (VAc) content of 50 wt% and Indonesia

tapioca flour (trade name Orang Tani) with 17.87 wt% amyloses and 82.13 wt% amylopectin content were purchased from a local supplier. The commercial-grade of chemicals such as dicumyl peroxide (DCP) as a crosslinking initiator, glycerol, and water as a starch plasticizer, CaCO₃ (filler), azodicarbonamide (ADCM) as a foaming agent, ZnO as an activator, stearic acid as a co-activator, Zn stearic as a lubricant were purchased from a local supplier. The compost and effective microorganism 4 (EM4) were purchased from a local supplier

Methods

Preparation of modified starch

Firstly, tapioca starch was modified by mixing the starch with water and glycerol at 60:25:15 of starch-glycerol-water ratio. These materials were mixed thoroughly by using a high-speed mixer at 350-500 rpm until attaining a homogeneous powder for 45 min. The mixture then kept for 2 days in a plastic bucket to plasticize and swell the granular starch molecule.

Preparation of EVA/modified starch expanded compound

EVA/modified starch expanded compounds were prepared by mixing EVA, modified starch, and additives using a two-roll mill laboratory scale. The blends were prepared according to the formulation in Table 1. The ratios of EVA/modified starch were varied from 100/0; 90/10; 80/20; 70/30; 60/40 and 50/50. After completion of the mixing process, the compounds were allowed to rest 24 h at 25 °C before subsequent processes. Subsequently, these EVA/modified starch expanded compounds were molded into a sheet using an electrically heated hydraulic press at 165 °C, 150 kg/cm² for 15 minutes (for 2+0.1 mm thickness) and 20 minutes (for 6+0.1 mm thickness).

Characterization

Testing of tensile strength, elongation at break, and tear strength were performed on a Universal Testing Machine (UTM, Tinius Olsen-H25K). Tensile strength and elongation at break test specimens were prepared according to ISO 37:2017(E) with a dumbbell-type 2 specimen. The tests were done at laboratory temperature (23±2 °C) with a crosshead speed of 500 mm/min. The tear strength of samples was measured

Table 1. The formulation of EVA/modified starch expanded compounds.

Sample code	Ingredients (phr) ^a							
	EVA,	Tapioca starch,	CaCO ₃	ZnO	Zn stearate	Stearic acid	DCP	ADCM
MS0	100	0	1	3	0,5	2	0,5	5
MS10	90	10	1	3	0,5	2	0,5	5
MS20	80	20	1	3	0,5	2	0,5	5
MS30	70	30	1	3	0,5	2	0,5	5
MS40	60	40	1	3	0,5	2	0,5	5
MS50	50	50	1	3	0,5	2	0,5	5

^aphr: parts per hundred parts of resins by weight

in accordance with ISO 34-1:2015(E) with an angle-type specimen using. The values presented were the mean values of three measurements in all cases.

Density was tested according to ISO 2781:2018(E) Method A by comparative measurement of samples mass in air and water using an Electron Densimeter (EW-200SG) from Mirage. Abrasion resistance test was done with a rotary drum abrasion tester (Bareiss) according to ISO 4649:2017 using the cylindrical shape specimens with a diameter of 16 ± 0.2 mm and a height of 6 mm. Abrasion resistance test result was expressed as a relative volume loss compared to an abrasive sheet calibrated using a standard reference compound.

The test of 50% permanent set (permanent extension) was characterized according to the relevant Indonesia National Standard test methods SNI 0778:2009 Sol Karet Cetak using a Permanent Set Tester. The flex resistance test was measured by using a Ross flexing machine and the flex continued up to 150 kcs according to the relevant Indonesia National Standard test methods SNI 0778:2009 Sol Karet Cetak point 6.2.7. The morphology of the EVA/modified starch expanded compounds was characterized by a Video measuring system (ARCS-SI 901).

The biodegradation test was carried out by the burial method in the compost soil. Biodegradability of the EVA/modified starch expanded compounds was evaluated by weight loss percentage (WLP) according to ASTM D-5988. The blends were cut into dumbbell shapes (a dumbbell-type 2 specimen). These dumbbell specimens were buried in a plastic pot containing compost enriched with effective microorganism 4 (EM4). The compost containing manure, charcoal husk, sand, soil, and cocopeat. The specimens were buried in the

compost at a depth of 10 cm from the surface to be the subject of attacking microorganisms in compost. The tested period was 14 days and lasted 48 days. The formula of WLP was as follows,

$$\text{WLP (\%)} = [(W_0 - W_1)/W_0] \times 100\% \quad (1)$$

Where WLP (%) refers to the weight loss percentage of the specimen; W_1 refers to the weight of specimen after degradation; W_0 refers to the weight of the specimen before degradation.

RESULTS AND DISCUSSION

Tensile Strength

The tensile strength of a material is a parameter of how much stress in the form of tension a material can accept before it finally breaks. Tensile strength of the EVA/modified starch expanded compounds containing various modified starch content were shown in Figure 1.

The modified starch loading gave a different response to the tensile strength properties of the blends. The addition of modified starch up from 10 to 30 phr increased the tensile strength as compared to the blends without modified starch (MS0). The tensile strength of the EVA compound without modified starch was 26.87 kg/cm² while with the addition of 10 phr modified starch expanded to 39.2 kg/cm², which means that there was an increase of 45.89%. The optimum tensile strength (61.33 kg/cm²) was obtained at 20 phr modified starch loading due to good dispersion of starch into the EVA matrix.

The good dispersion of starch into the EVA matrix was validated by morphology image (see Figure 8b). However, with a further increase in starch loading, the tensile strength gradually decreased. The decrease in tensile strength at the higher starch concentration (40-50 phr) was due

to starch-starch interaction more dominance than the starch-EVA interaction, which resulted in agglomeration of the starch particles. It could be seen in the morphology images (Figure 8). The agglomerates arose due to the inhomogeneous distribution of the filler in the matrix, thereby reducing the interfacial tensile force and weakening the tensile strength (Fang *et al.*, 2014). According to Ayu *et al.* (2018) and Hamadache *et al.* (2019) increasement of starch content produces more voids, resulting a weakening structure of the blends due to poor interfacial bonding between polymer matrix and starch.

The Indonesian National Standard (SNI) for the EVA/starch base expanded compound for outsole did not exist yet, and international standards have not been found either. For this reason, the quality of the resulting expanded compound was compared with the quality requirements of SNI 0778:2009 Sol Karet Cetak for quality classification 3 with a minimum tensile strength value of 50 kg/cm². The tensile strength values of the expanded compound containing 20 and 30 phr of modified starch were 61.33 kg/cm² and 55.63 kg/cm² respectively, so these values met the requirements of SNI 0778:2009 Sol Karet Cetak.

Elongation at Break

Elongation at break of the EVA/modified starch expanded compounds at different modified starch content was presented in Figure 2.

Figure 2 showed the elongation at break value of the EVA/modified starch expanded compounds was increasing along with the rising amount of tapioca starch up to 30 phr and afterward, it decreasing with higher modified starch loading. Elongation at break of EVA compound without modified starch was 705.33%. The addition of

modified starch of 10 phr, 20 phr, and 30 phr gave an enhancement in elongation at break of 16.64%, 26.89%, and 9.78% respectively, compared to the EVA compound without modified starch. The highest value of elongation at break value (895%) was observed for samples containing 20 phr modified starch.

According to Ali *et al.* (2013), an rise in the amount of starch increases the ductility of the sample. The addition of 40 phr modified starch decreased elongation at break of 50.52% compared to the elongation at break of the EVA compound without modified starch. The decrease in elongation at break of EVA/starch composites suggested that the introduction of starch particles in the EVA matrix caused poor interactions between the EVA matrix and the starch particles (Hamadache *et al.*, 2019).

Based on SNI 0778:2009 Sol Karet Cetak, the minimum elongation at break value was 150%. From Figure 2, it can be seen that the elongation at break of all the expanded compounds met the requirements of SNI 0778:2009 Sol Karet Cetak.

Tear Strength

Tear strength used to measure the tear resistance is the force required to pull apart a material until it rips (Tang *et al.*, 2019). The tear strength of the EVA/modified starch expanded compounds containing various modified starch were shown in Figure 3.

Figure 3 showed that an addition in the starch content (10 to 50 phr) led to an increasement in the tear strength of the blends. The tear strength also followed the same trends as tensile strength. The tear strength value of EVA compound without modified starch was 8.98 N/mm. The addition of 10 phr of modified starch increased the tear

Figure 1. Tensile strength of the EVA/modified starch expanded compounds.

Figure 2. Elongation at break of the EVA/modified starch expanded compounds.

strength value by 11.80%. The optimum tearing strength (16.62 N/mm) was observed for the EVA/modified starch blends containing 20 phr modified starch. The tear resistance can be affected by the number and size of micropores formed during the heat forming process. The large and uneven pore size resulted in low tear resistance.

According to ISO/TR 20880- Footwear — Performance requirements for components for footwear — Outsoles, the minimum tear strength is 8,0 N/mm (for density ≤ 0.9 g/cm³) or 6.0 N/mm (for density > 0.9 g/cm³). Therefore the tear strength of all the resulting expanded compounds met the requirements of ISO/TR 20880- Footwear — Performance requirements for components for footwear — Outsoles.

Permanent Set

The permanent set refers to the residual extension remaining after a material sample is stretched and released (Diani *et al.*, 2009). The 50% permanent set was used in this research. The permanent set of the EVA/modified starch expanded compounds containing various modified starch were shown in Figure 4.

The lower the permanent set, the higher the ability of the material to retain its elasticity. EVA compound without modified starch showed a permanent set value of 8.21%. The incorporation of 10; 20 and 50 phr of modified starch to EVA compound had little influence on the permanent set of EVA compound. The optimum permanent set (4.85%) was observed for the EVA/modified starch blends containing 30 phr modified starch which meant a decrease of 40.93% compared to EVA compound without modified starch. The value of 50% permanent set required for the SNI rubber sole was 6% maximum. Therefore,

Figure 3. Tear strength of the EVA/modified starch blends.

the EVA/modified starch expanded compound containing 30 phr modified starch (MS30) met the Indonesia National Standard SNI 0778:2009 requirements.

Density

The density was defined by the difference of weight in air and water divided to weight of samples in air. As it could be seen, with the addition of 10-50 phr modified starch to EVA compounds, the sample densities increased (Figure 6). Similar results have been observed by Rodriguez-Perez *et al.* (2012) which an increase in the starch content leads to an increase in the density. The addition of 10 phr modified starch to the EVA compound (MS0) increased the density by 3.67%.

A higher density (0.913 g/cm³) was observed for the EVA/modified starch expanded compound containing 20 phr of modified starch. The density of this formulation may be influenced by the voids of a blend material and by the interface bond between the EVA matrix and starch. EVA matrix and modified starch that was not bonded properly caused low density due to the space around the starch which did not adhere to the matrix and vice versa. Interfacial characteristics between modified starch particles and EVA matrix as well as the microstructural difference gave a strong relationship between morphological. According to SNI 0778: 2009 Sol Karet Cetak, the maximum density value is 1.4 g/cm³, and Figure 5 showed that the density of all expanded compounds met the requirements of the Indonesia National Standard SNI 0778: 2009 Sol Karet Cetak.

Abrasion Resistance

Abrasion resistance is the ability of a material to resist mechanical action such as rubbing, scraping

Figure 4. The permanent set of the EVA/modified starch expanded compounds.

or erosion that tends progressively to remove material from its surface (Arayapranee, 2012). The smaller volume loss indicated better abrasion resistance. Abrasion resistance of the EVA/modified starch expanded compounds containing various modified starch was shown in Figure 6. The enhancement of volume loss during the abrasion test compared to EVA compound without starch content was shown by an increasement in the amount of modified starch up to 20 phr. These indicated inhomogeneous mixing of the sample.

The addition of modified starch from 40 to 50 phr increased abrasion resistance. This abrasion enhancement with the presence of starch might be due to good interaction between modified starch and the EVA matrix of expanded compounds. Figure 6 showed that the smallest volume loss was 439.99 mm³ represented by the compound containing 30 phr of starch. The volume loss required by SNI 0778:2009 Sol Karet Cetak is a maximum of 350 mm³, this meant that all the compounds produced did not meet these requirements. Figure 8 showed the inhomogeneous dispersion and distribution of the starch particles in the EVA matrix.

Flex Resistance

Flex resistance is the ability to sustain numerous flexing cycles without the occurrence of crack or damage to the surface. The flexing resistance of the microcellular samples was determined using a Ross flexing machine. Flexing cycles required according to SNI 0778: 2009 Sol Karet Cetak is 150000 cycles. According to Mohamad Aini *et al.* (2019), the reduction of flexibility increases the stiffness of composites, resulting in the reduction of flexing resistance. Table 2 showed that the

Figure 5. The density of the EVA/modified starch expanded compounds.

addition of modified starch material up to 50 phr still provided good flexing resistance.

Based on the flex resistance requirements of SNI 0778:2009 Sol Karet Cetak, all EVA/modified starch expanded compounds met the requirements of SNI 0778:2009 Sol Karet Cetak.

Biodegradability

The weight loss of EVA/modified starch expanded compound at different modified starch content after compost burial test were presented in Figure 6. It showed the weight loss of EVA/modified starch generally increased with a rise in modified starch content and burial time. This weight loss might be due to the invasion of microorganisms into the blends sample. From the data in Figure 6, it was obvious that the EVA compound without modified starch (MS0) remained unchanged, even after 42 days, and no weight loss was noticed. This was due to the very small accessibility of starch to microorganism that affected in slow biodegradability rate. The EVA/TPS blend with 10% starch content showed fast weight loss up to 14 days, while for the next 28 days, the blend remained nearly unchanged. It indicated that the starch had been biodegraded in the first 14 days. The EVA/TPS blend with 50% modified starch content showed very fast in weight loss for the first 14 days.

The increasing content of starch in the EVA/starch blend accelerated the rate of weight loss. It was due to the dispersed part of starch began to bond and became more interlinked. The higher weight loss (13.61%) was measured for samples containing 50% modified starch in a period of 42 days. Thermoplastic starches have been reported

Figure 6. Abrasion resistance of the EVA/modified starch expanded compounds.

Table 2. Flexing resistance of the EVA/modified starch expanded compounds.

Testing parameter	Formulation					
	MS0	MS10	MS20	MS30	MS40	MS50
Flexing resistance for 150kcs	crack	No crack	No crack	No crack	No crack	No crack

to provide better biodegradability (Rodriguez-Perez *et al.*, 2012; Macedo & Rosa 2015; Sessini *et al.*, 2019).

Morphology of the EVA/modified Starch Expanded Compounds

Figure 8 showed the morphology image of the EVA/modified starch expanded compounds with different modified starch content; a (10 phr), b (20 phr), c (30 phr), d (40 phr), e (50 phr). It can be seen that the good dispersion of starch into the EVA

matrix was observed by an expanded compound containing 20 phr modified starch (Figure 8b). Figure 8 also showed that along with the increase of modified starch content the dispersion of modified starch became poor, and according to Guo *et al.* (2021) it caused agglomeration and resulted in the decrease of cell size uniformity. Figure 8e showed an image of the EVA/modified starch expanded compounds containing 40 phr starch content, there were many large open cells due to starch agglomeration.

Figure 7. Weight loss of the EVA/modified starch as a function of burial time. **Figure 8.** Morphology image of the EVA/modified starch expanded compounds with different modified starch content: a (10 phr), b (20 phr), c (30 phr), d (40 phr), e (50 phr).

CONCLUSIONS

EVA/modified starch expanded compound with different modified starch contents (0-50 phr) were prepared by mixing EVA, modified starch, and additives using a two-roll mill laboratory scale. The starch was tapioca starch that modified by mixing the starch with water and glycerol at 60:25:15 (starch:glycerol:water). Effect of modified starch content was characterized its tensile strength, elongation at break, tearing strength, 50% permanent set, density, abrasion resistance, flex resistance, biodegradability, and morphology. The optimum tensile strength (61.33 kg/cm²) was obtained at 20 phr modified starch loading. The highest value of elongation at break value (895%) was observed for samples containing 20 phr modified starch. The optimum tearing strength (16.62 N/mm) was observed for the EVA/modified starch blends containing 20 phr modified starch. The addition of 10-50 phr modified starch to EVA compounds increased the density. A higher density (0.913 g/cm³) was observed for the EVA/modified starch expanded compound containing 20 phr of modified starch. The EVA compound containing 30 phr of starch showed the optimum 50% permanent set (4.85%) and the highest abrasion resistance with the smallest volume loss of 439.99 mm³. The addition of modified starch up to 50 phr still provided good flexural resistance to 150000 number of cycles. The biodegradation test was carried out by the burial method in the compost soil for up to 42 days, and the biodegradability was evaluated by weight loss percent. The higher weight loss (13.61%) was measured for samples containing 50% modified starch in a period of 42 days. The increasement of modified starch content in the EVA matrix gave a poorer dispersion of modified starch. In general, the results of the EVA/modified starch expanded compound test have met the requirements of SNI 0778: 2009 Sol Karet Cetak, but the abrasion resistance was still below the quality requirements.

ACKNOWLEDGEMENTS

This research was funded by the Indonesian government under DIPA 2020. The authors are grateful to the Head of Center for Leather, Rubber and Plastics, and to Research Team for the support during the research that has been conducted.

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