

The Use of Polyethylene-grafted-Maleic Anhydride as a Compatibiliser for Composites from a Mixture of Poly-Aluminum and Fibers

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Abstract

Used beverage carton (UBC), is one type of used paper/cardboard that has potential as a recycled fiber source. UBC is composed of 3 components, namely paper, polyethylene and aluminum foil with a composition of 74-80%, 20-21%, and 5% respectively. UBC recycling initially aimed to recover fiber component which is the largest component that can be reused as paper/paperboard raw material. However, the UBC recycling also obtained by-products in the form of polyethylene (PE) and aluminum that can be used as composite raw materials. In general, a composite is expected to have high strength but light weight. The PE-aluminum composite has high strength but is rather heavy. So that, the experiments of making PE-aluminum composites with the addition of fiber have been done. The usage of PE-g-MA (polyethylene-grafted-maleic anhydride) as a compatibiliser was also studied. Composites from a mixture of PE-aluminum and fiber (ratio 4:1) have been made with the variations of 0-5% PE-g-MA. The temperature of hot press were also varied 140°C and 165°C. As a control, composite of 100% PE-aluminum without PE-g-MA was also made. The resulting composites were tested for density (SNI ISO 534:2011), moisture content (SNI ISO 287:2009), water absorption (SNI 01-4449-2006 Item 8.2.5), and dimensional stability. The presence of fiber in the PE-aluminum composite was able to reduce the density, moisture content and water absorption properties of the resulting composite, but the dimensional changes after being immersed in water for 24 hours increased. The addition of PE-g-MA as an additive to a mixture of PE-aluminum and fiber composites, at a hot press temperature of 165°C, further reduced the water content and water absorption of the composites with a density close to the density of PE. The role of PE-g-MA as an interface component between the fiber phase and the PE-aluminum phase were shown in the 50x magnification of the composite cross-section using SEM (Scanning Electron Microscope) although its distribution in the composite is still uneven.

Keywords: PE-g-MA, composite, compatibiliser, polyethylene-aluminum, fiber, UBC

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1. Introduction

The issue of deforestation and increasing environmental pressure on the use of wood virgin fiber as paper raw material has made increasing demand on the use of secondary fiber from waste paper recycling. Almost all places in the world show a positive growth trend in consumption of waste paper as fiber source for paper production.

Based on data from the EPA (Environmental Protection Agency), in the United States there was an extraordinary growth of waste paper recycling from 1960 to 2017. Recycling in 1960 which was only 5 tons grew 65.9% to 44 tons in 2017. In 2018, the growth also increased to 68.1% (<https://www.thebalancesmb.com/an-introduction-to-paper-recycling-4036123>).

In Indonesia, the need for waste paper as paper raw material is also estimated to increase by 5% per year (<https://www.ojk.go.id/sustainable-finance/id/Lists/Agenda%20Nasional/Attachments/49/>).

Used beverage carton (UBC), is one type of waste paper/paperboard that has potential as a secondary fiber source. In general, beverage packaging cartons are composed of 3 components, namely paper, polyethylene and aluminum with a composition of 74-80% paper, 20-21% polyethylene, and the rest, 5%, is aluminum foil (Santosa, 2013; Zawadiak dkk., 2017; Bláhová dan Myrta, 2017; <https://www.tetrapak.com/id/sustainability/daur-ulang-kemasan>, Rizaluddin dkk., 2020-1 dan 2020-2).

Based on data from a beverage packaging carton producer, the recycling rate of UBC has increased from 2015 to 2018 as presented in Table 1 (<https://www.suara.com/bisnis/2019/09/10/093109/tetra-pak-daur-ulang-10388-ton-kemasan-karton-bekas-minuman>).

Table 1. UBC recycling rate

Year	Volume (ton)	% recycled
2015	2,885	7.5
2016	4,382	8.7
2017	6,637	14.7
2018	10,338	21.2
2019		22.5
2020	>13,000	24

Source: <https://www.suara.com/bisnis/2019/09/10/093109/tetra-pak-daur-ulang-10388-ton-kemasan-karton-bekas-minuman>

UBC recycling, in addition to recovering the fiber components in UBC that can be used as paper or paperboard raw material, it will also produce by-products in the form of polyethylene (PE) and aluminum (Figure 1).

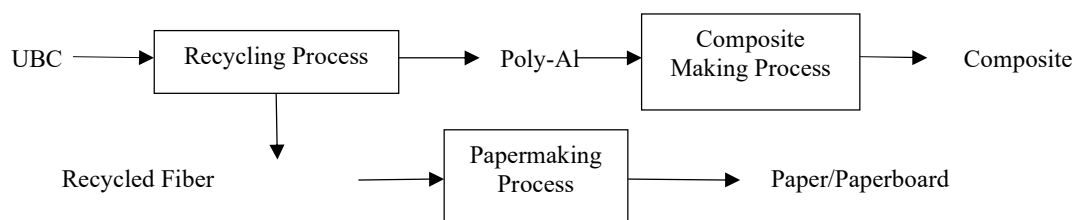


Figure 1. UBC recycling process

According to Zawadiak et al. (2017), UBC recycling can be done with or without using of hydropulper. The last method is generally applied in order to energy recovery or obtaining low grade products. Lamination board products can be obtained by this way where UBC pieces of 1-5 mm of size are hot pressed at a temperature of 170°C. The recycling process using hydropulper is carried out as usual the waste paper recycling in general, however, with a slight modification in the separating process between pulp slurry and polyethylene-aluminum.

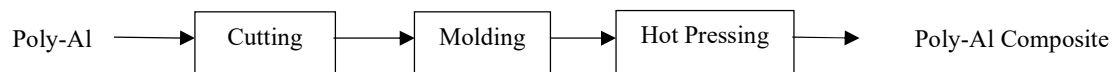


Figure 2. Poly-Al composite making process

By-products of UBC recycling, i.e. polyethylene and aluminum, can be used then as raw materials of composites namely poly-Al composite. The poly-Al composite making process can be seen in Figure 2 (<https://www.tetrapak.com/id/sustainability/daur-ulang-kemasan/proses-daur-ulang-polietilen-dan-aluminium>).



Figure 3. Products made poly-Al composites

(Source: <https://www.tetrapak.com/id/sustainability/daur-ulang-kemasan/proses-daur-ulang-poliethilen-dan-aluminium>)

Currently, various products have been made from poly-Al composite, including corrugated roofs, stationaries, furniture and others. Some of them can be seen in Figure 3.

In general, the important characteristics expected from a composite product are high and massive strength. This can be obtained from poly-Al composites, however, its density is rather high so that its use requires special handling.

In its usage, composites are expected to have a high strength with light in weight. The addition of fiber can be done to reduce the composites weight. The experiment of adding bagasse fiber into polymer composites with a dose variation of 0-15% reduced the composite density from 1.464 g/cm³ without bagasse to 1.1178 g/cm³ at the addition of 15% bagasse (Clareyna and Mawarni 2013). In addition, the usage of cellulose fibers in polymer composites is also intended to produce more environmentally friendly composites (Kahar et al., 2012).

An experiment to decrease the density of the poly-Al composite from recycled UBC has been carried out. According to Rizaluddin et al. (2020), the addition of 5-30% fiber in the manufacture of poly-Al composites increased thickness, water absorption, and moisture content, it decreased its density. The addition of fiber up to 15% increased the composite bending strength.

In this article, an experiment on using of compatibiliser to further improve the quality composite made from a mixture of poly-Al and fiber is reported.

2. Compatibiliser PE-g-MA

Polyethylene-Graft-Maleic Anhydride or PE-g-MA is one of the most widely compatibilisers used in polymer blends (Fatimah et al., 2015; Kahar et al., 2012; Mengual et al., 2017; Rzayev, 2011). Other names for this material are maleated copolymer, maleated polymer, or maleic copolymer (<https://www.sigmaldrich.com>).

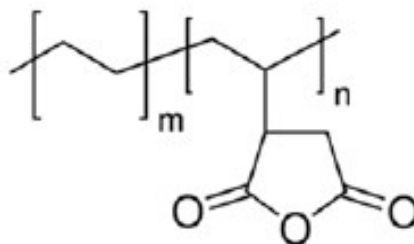


Figure 4. Chemical structure of PE-g-MA

(Source: Mengual et al, 2017; <https://www.sigmaldrich.com>; Rzayev, 2011)

Based on the experiments conducted by Zhai et al. (2004), PE-g-MA was prepared by mixing HDPE (high-

density polyethylene) with MA (maleic anhydride), DCP (dicumyl peroxide) and DMSO (dimethyl sulfoxide) in a high-speed mixer for 5 minutes with a stirring speed of 1100 rpm, then extruded in a single screw extruder with a rotating speed of 30 rpm. Rzayev (2011) also reported the use of dicumyl peroxide in the process of grafting maleic anhydride into low density polyethylene which was melted in a reactor bath. In addition, it was also reported the use of polyamide 66 in maleic anhydride reactive mixture grafted onto PE in a twin-screw extruder.

Table 2. Properties of PE-g-MA

Parameter	Maleic-anhydride, ~0,5 wt.% ¹⁾	Specification ²⁾
Color	NA	White – off white
Shape	NA	Beads/pellets
Viscosity at 140 °C	500 cP	≤ 600 cP
Saponification	6 mg KOH/g	3-6 mg KOH/g
IR Spectrum	NA	according to structure
Transition temp., Tm (DSC)	107 °C at peak	NA
Solubility:		
- H ₂ O	Insooluble	NA
- Toluene	Soluble	NA
- Xylene	Soluble	NA
Density at 25°C	0.92 g/mL	NA

Note : NA : Not available

Source: ¹⁾ Mengual et al., (2017)

²⁾ <https://www.sigmaaldrich.com>

The chemical structure and properties of PE-g-MA are presented in Figure 4 and Table 2 respectively. PE-g-MA has a melting point of 80oC, insoluble in water, and a density of 0.92 g/mL at 25oC (https://www.chemicalbook.com/ProductChemicalPropertiesCB7406517_EN.htm).

Generally, there are three purposes of using PE-g-MA, i.e. to improve the properties of the base material, as a compatibiliser in a mixture consisting of two ingredients, and as an additive in a mixture of various materials (Mengual et al., 2017).

As a compatibiliser in polymer mixtures, PE-g-MA will act as a support for polar and non-polar parts (Mengual et al., 2017).

Basically, composites consist of 2 phases, ie matrix phase and reinforcement, filler, or fiber phase. The first phase is the largest volume in a composite, while the second one is the material that function as a composites' main load-bearing. Figure 5 shows the parts of composite based on that two phases (<http://nurun.lecturer.uin-malang.ac.id/wp-content/uploads/sites/7/2013/03/Material-Komposit.pdf>).

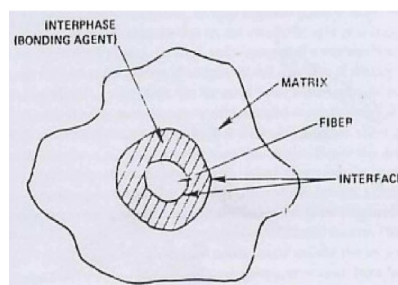


Figure 5. Parts of composite

(Source: <http://nurun.lecturer.uin-malang.ac.id/wp-content/uploads/sites/7/2013/03/Material-Komposit.pdf>)

Figure 5 shows the presence of an interphase section which is a binder between the two phases/materials, as well as an interfaces section which are a phase surfaces that borders other phases.

Based on the above description, in this experiment, the addition of PE-g-MA into the composite mixture is to be function as an interphase that connects the polyethylene-aluminum phase with the fiber phase.

3. Experiment

In this experiment, the fiber and poly-Al obtained from used beverage carton (UBC) recycling in the Laboratory

of Center for Pulp and Paper were used. PE-g-MAH (polyethylene-grafted maleic anhydride) from the Polymer Technology Center was used as a compatibiliser.

The equipment used include a chopper, analytical balance, composite mold made of metal equipped with a teflon sheet and lubricant, hot press that can reach a temperature of 165°C, as well as equipment for testing the physical properties of the composite.

Table 3. Composition variations

Code	Poly-Al (%)	Fiber (%)	PE-g-MA (%)
B	100	0	0
M-0	80	20	0
M-2.5	80	20	2.5
M-5	80	20	5

Poly-Al and fiber were chopped separately in a crusher to a size of about 0.5x0.5 cm. Furthermore, the chopped poly-Al and fiber were mixed with PE-g-MA in various compositions as shown in Table 3. The mixture was stirred until evenly distributed and then a certain amount of it was taken to obtain a composite thickness of + 3mm. The mixture then was hot pressed in a composite mold which had been based with a Teflon sheet and lubricated. The pressing was carried out for 10 minutes at a pressure of 25-30 kg/cm² and temperature variations of 140°C and 165°C.

The composites resulted were tested for density (SNI ISO 534:2011), moisture content (SNI ISO 287:2009), water absorption (SNI 01-4449-2006 Item 8.2.5) and change in dimensions. The measurement of dimension change in principally was carried out by measuring the changes of composites' length and width after being immersed in water for 24 hours. The dimension changes reported were the largest dimension change of composites' length or width. In addition, the surface structure of the composite was also observed using a Scanning Electron Microscope (SEM) at a magnification of 50x.

4. Discussion

4.1 Density

The addition of 20% fiber into the poly-Al composite without PE-g-MA (code M-0) decreased the density. As previously stated, the density of composites generally decreases with the addition of more fiber into the composite (Clareyna and Mawarani, 2013; Rizaluddin et al., 2020-1). The cellulose fibers density varies greatly depending on the source of fiber raw material. For example, bagasse fiber has a density of 0.341 g/cm³, wheat straw 0.909 g/cm³, rice straw 1.36 g/cm³ up to 1.5-1.6 g/cm³ cotton fiber (Suryanto et al., 2014). Meanwhile, the LDPE or Low Density Polyethylene and aluminum foil has a density of 0.910-0.940 g/cm³ and 2.70 g/cm³ respectively

[\(https://id.wikipedia.org/wiki/Polietilena#:~:text=LLDPE%20dicirikan%20dengan%20densitas%20antara,dengan%20jumlah%20yang%20cukup%20signifikan.;](https://id.wikipedia.org/wiki/Polietilena#:~:text=LLDPE%20dicirikan%20dengan%20densitas%20antara,dengan%20jumlah%20yang%20cukup%20signifikan;)

[https://www.google.com/search?safe=strict&sxsrf=ALeKk029X7UKYWUjT18sx8GdmaZFndDq_A%3A1612746433126&ei=wY4gYL6eB5LGrQG15nAAQ&q=densitas+aluminium+foil&oq=densitas+aluminium+foil&gs_lcp=CgZwc3ktYWlQAZlFCCEQoAE6BwgAEEcQsAM6BAgiECc6BAgAEA06AggAOgUIABCxAzoECAAQCjoHCAAQRhD7AToICAAQFhAKEB46BggAEBYQHjoHCCEQChCgAVD15hpYgJobYJaiG2gBcAJ4AYA BrAaIAewxkgEOMy4xNy4yLjAuMS4yLjkYAQCgAQGqAQdnd3Mtd2l6yAEIwAEB&sclient=psy-ab&ved=0ahUKewj-gonSjNnuAhUSYysKHaJrBhgQ4dUDCAw&uact=5\).](https://www.google.com/search?safe=strict&sxsrf=ALeKk029X7UKYWUjT18sx8GdmaZFndDq_A%3A1612746433126&ei=wY4gYL6eB5LGrQG15nAAQ&q=densitas+aluminium+foil&oq=densitas+aluminium+foil&gs_lcp=CgZwc3ktYWlQAZlFCCEQoAE6BwgAEEcQsAM6BAgiECc6BAgAEA06AggAOgUIABCxAzoECAAQCjoHCAAQRhD7AToICAAQFhAKEB46BggAEBYQHjoHCCEQChCgAVD15hpYgJobYJaiG2gBcAJ4AYA BrAaIAewxkgEOMy4xNy4yLjAuMS4yLjkYAQCgAQGqAQdnd3Mtd2l6yAEIwAEB&sclient=psy-ab&ved=0ahUKewj-gonSjNnuAhUSYysKHaJrBhgQ4dUDCAw&uact=5).)

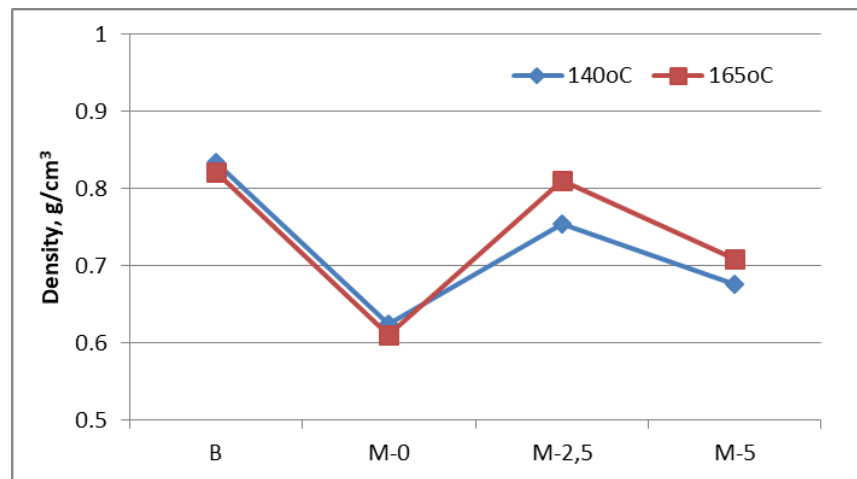


Figure 5. Composite density

The poly-Al obtained from UBC recycling process is only about 25% of total UBC recycled. The poly-Al obtained consists of 20% polyethylene and 5% aluminum, therefore the poly-Al recycled from UBC is assumed to consist of 80% polyethylene and 20% aluminum. This causes the density of the poly-Al composite to be almost the same as the density of polyethylene. The addition of PE-g-MA compatibiliser to the composite of mixed poly-Al and fiber increased the density closer to that of polyethylene. This is in line with Rizaluddin et al. (2020-2) which states that the composite of poly-Al from UBC recycling has an average density in the range of 0.81-0.93 g/cm³.

The temperature of hot press was not significantly influenced the density of composite resulted, but in general, the composite with slightly higher density was resulted by a higher hot press temperature. The higher the pressing temperature of composite of polyethylene-based increases the spreading of polyethylene melt in the composite (Kardiman et al., 2019; Widodo et al., 2020). This causes composite become denser at higher pressing temperatures.

Table 4. The requirement of density, moisture and water absorption of composite in SNI and JIS

Standard	Density (g/cm ³)	Moisture (%)	Water Absorption (%)
SNI 02-2105-2006 Particle Board	0.40-0.90	≤14	
SNI 01-4449-2006 Fiberboard	0.40-0.84	≤13	<17
SNI 8154:2015 Wood-Plastic Composite	≥0.60	<12	
JIS A 5908:2003 Particle Board	0.40-0.90	5-13	
JIS A 5905:2003 Fiberboard	≥0.35	5-13	

Source: Rizaluddin et al., 2020-2

Compared to the requirements for several composite products listed in Table 5, the density of all composites resulted in this experiment met the SNI 03-2105 and JIS A 5908:2003 for particle board which are in the range of 0.40-0.90 g/cm³, as well as SNI 8154: 2015 Wood-Plastic Composite and JIS A 5905:2003 Fiberboard, i.e. more than 0.60 g/cm³ and 0.3 g/cm³ respectively.

4.2 Moisture

Moisture content is an important property of composite since it influences the physical and mechanical properties of composites. In general, the moisture content of a material is affected by the relative humidity (RH) of the surrounding air. The higher the RH of the air around a material, the material will absorb water vapor from the air, so it increase the moisture content of the material. In contrary, the moisture content will decrease if the material is exposed to the air with a low RH where the moisture from the material will evaporate into the surrounding air.

The moisture content of composite affects the swelling and variation in density of the composite (Fortineau et al., 2007). Figure 6 shows the moisture content of composite resulted from the experiment.

The presence of fiber in a poly-Al composite (code M-0) caused the increase of composites' moisture content

compared to the poly-Al composite without fiber (code B). Basically, cellulose fiber has hygroscopic properties, the ability to absorb moisture from the surroundings or release its moisture content into surrounding air until an equilibrium condition is reached (Carr et al., 2006). The addition of fiber into poly-Al composite caused the composite to be more hygroscopic so that its' moisture content (code M-0) was higher than that of without fiber (code B).

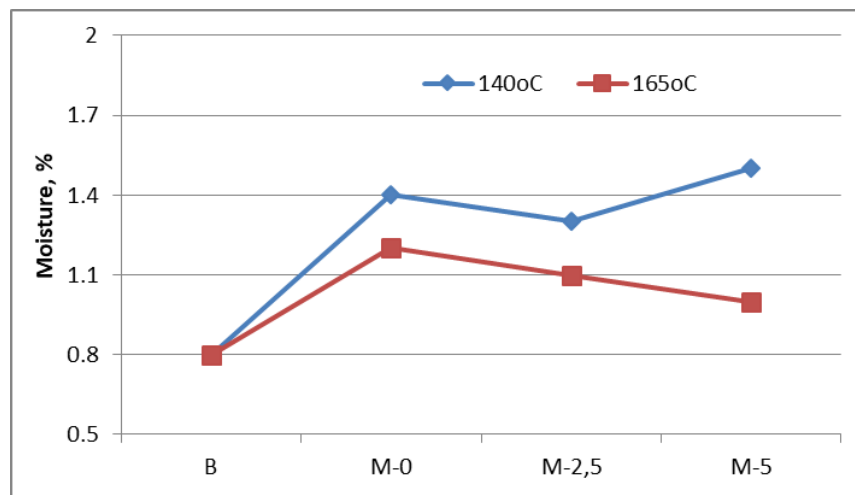


Figure 6. Moisture content

The use of PE-g-MA compatibiliser reduced the moisture content of composites, especially at the pressing temperature of 165°C. As previously stated, the addition of PE-g-MA into poly-Al and fiber mixture serves as an interphase binder. Cellulose fiber has a hydroxyl group that causes the fiber to be hygroscopic. The moisture content of the fiber is strongly influenced by the surrounding RH conditions. The pressing process with high temperature makes PE-g-MA melts and binds the fiber phase to the poly-Al phase, thereby reducing the amount of hydroxyl groups on the fiber and making the composite more resistant to moist conditions. This allows the composite to have a lower moisture content. Increasing the PE-g-MA dosage and pressing temperature decreased further the moisture content of composite. This was caused by the increasing number and distribution of PE-g-MA in the composite, so that it becomes more resistant to the moist environment.

Compared to SNI requirement in Tabel 4, the composite resulted in this experiment have met the requirement. Meanwhile, compared to JIS, the moisture content of the composite resulted were below the required range of moisture content values.

4.3 Water Absorptivity

The water absorptivity is related to the ability of a material to absorb water in the form of liquid phase. In composites, this property is very important since it affects the mechanical properties and causes major changes in the behavior of the composite (Fortineau et al., 2007; Majid et al., 2010).

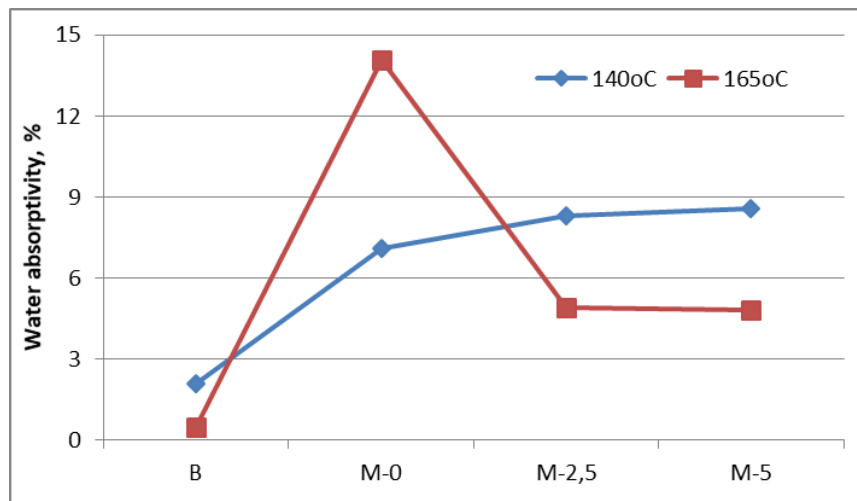


Figure 7. Water absorptivity

The experiment response to the composites' water absorptivity were as same as moisture content. The addition of fiber (code M-0) increased water absorptivity of composite. The hydroxyl groups of cellulose fiber, in addition of giving the hygroscopic properties, it also provides hydrophilic properties which is the ability of material to absorb water in liquid form.

The use of PE-g-MA compatibiliser, both at pressing temperatures of 140°C and 165°C, did not affect significantly to the decreasing of water absorptivity of composites. However, the pressing temperature of 165°C provides lower water absorptivity.

Compared to the requirements of SNI 01-4449-2006 for Fiberboard, the composite resulted in this experiment met the requirements, which is lower than the maximum limit of 17%.

4.4 Dimensional Change

Dimensional change of composite is also an important properties, especially for composites used for construction or furniture. Even a small change in composite dimensions can be fatal to a construction products. Dimensional change of composite is also an important properties, especially for composites used for construction or furniture. Even a small change in composite dimensions can be fatal to a construction products.

Evaluation of composite dimensional changes was carried out by measuring the changes in composite dimensions after being immersed in water for 24 hours. The test results are influenced by the water absorptivity of composite. Dimensional change of the composite resulted in this experiment are presented in Figure 8.

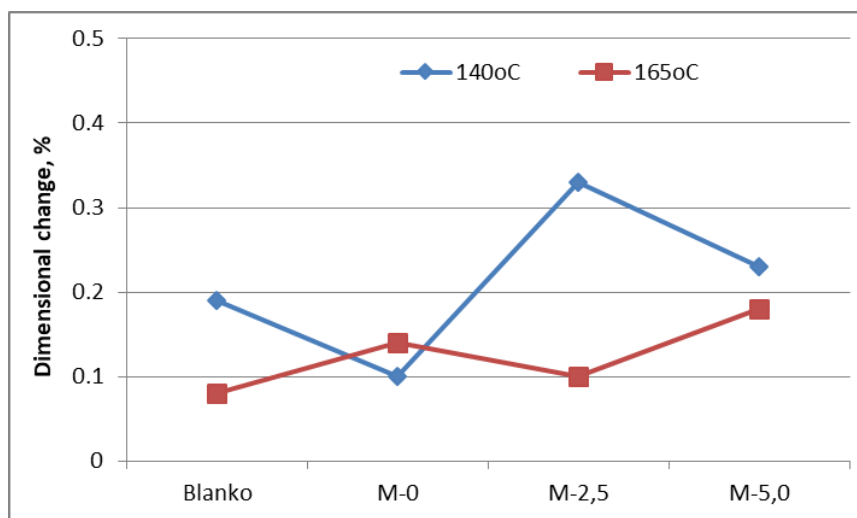


Figure 8. Dimensional change

The addition of fiber to the composite (M-0) increased the dimensional changes of the composite, but after adding PE-g-MA (M-2.5) there was a slight decrease in the dimensional changes. The effect of pressing temperature does not give a clear pattern but at higher temperatures tends to give better dimensional stability to the composite.

In addition to pressure and pressing time, the temperature is also important parameter in the hot pressing process (Kumar and Balachandar, 2014). The temperature of 140°C may be insufficient to produce a composite of mixed poly-Al and fiber with good physical properties.

According to the test method which measuring the dimensional change of composite after being immersed in water for 24 hours, the test results obtained are closely related to the composite water absorptivity. Figure 7 shows that the addition of PE-g-MA at pressing temperature of 165°C resulted composite with low water absorptivity. This is in line with the low change in composite dimensions at the same mixture composition and pressing temperatures.

4.5 Surface Photograph

Figure 9 shows photographs of composite surface using Scanning Electron Microscope (SEM) with a magnification of 50 times. The following are the notation of photographs in Figure 9.

Table 5. Notation of photographs

Notation	Composition of Mixture	Temperature, °C
A	B	140
B	M-0	140
C	M-5	140
D	M-0	165
E	M-5	165

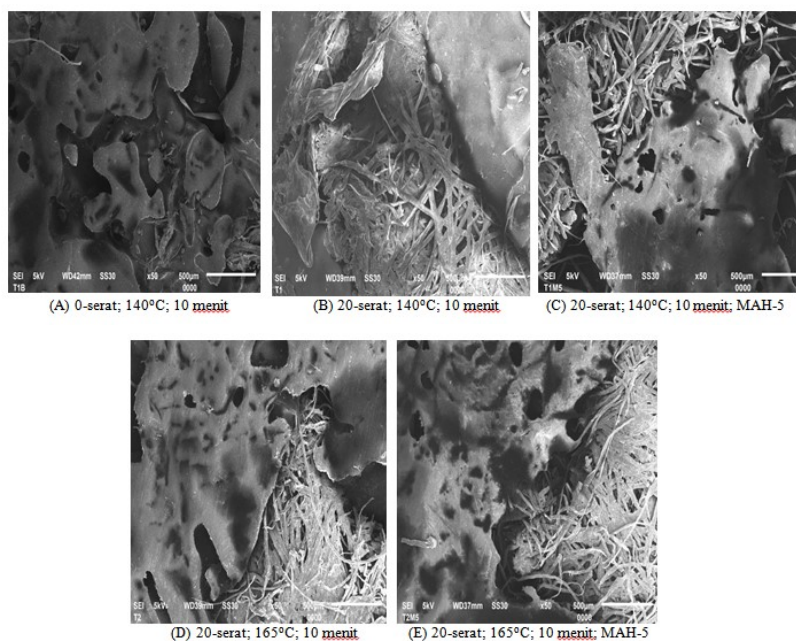


Figure 9. SEM Photographs of Composite Surface

The image A shows poly-Al melt at the composite surface , while image B shows the poly-Al melt and fiber on the composite surface.

As a compatibiliser, PE-g-MA added to the composite poly-Al and fiber seems not function well at the pressing temperature of 140°C, since it was insufficient to melt and distribute PE-g-MA evenly into the mixture of poly-Al and fiber (image C). However, at a higher pressing temperature of 165°C, both without and with the addition of 5% PE-g-MA, the poly-AL and PE-g-MA were melted and distributed better among the fiber in the composite (image D and E).

The experiment of composite making from a mixture of LDPE (low density polyethylene) and kenaf fiber with the addition of TSS (thermoplastic sago starch) at a pressing temperature of 150oC has been reported. The results showed that the SEM image of the composite surface was better than without the addition of TSS. TSS is functions as a compatibiliser between LDPE and kenaf fiber (Majid et al., 2014).

Higher pressing temperature increased the effectiveness of PE-g-MA as a compatibilizer between poly-Al and fiber, and produced a composite with a better and even surface.

5. Conclusion

PE-g-MA used in the manufacture of composites made from a mixture of poly-Al and fiber, showed a fairly good function as a compatibiliser, i.e. interface link between poly-Al and fiber. This is indicated by the improvement of composite properties including the decrease the moisture content, water absorption and dimensional change of composite, especially at pressing temperature of 165oC. The presence of fiber in the poly-Al composite reduced the density of the composite that made the composite lighter. Furthermore, the addition of PE-g-MA, which is a polyethylene-based compatibiliser, increased the density of composite that tends to approach the density of polyethylene.

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