Fabrication of Thermoplastic Elastomers (TPE) by Using Emulsion Method as an Alternative Material For Vehicle Bumper Protector

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Abstract
Research on Fabrication of Thermoplastic Elastomers (TPE) by Using Emulsion Method as an Alternative Material For Vehicle Bumper Protector aims to produce of the thermoplastic elastomers by emulsion method with variation of composition ratio of polypropylene grafting and Maleic Anhydride (PP-g-MA)(mL) : latex (mL) : glycerin, to have strong tensile strength results according to British Plastic Federation standard 0.5 – 2.4 (N/mm²) standard for bumper material elongation maximum of 22.62%. Emulsion method was used as sample preparation which is grafting polypropylene (PP) with Maleate Anhydride (MA) then continued with PP-g-MA Emulsion Making and Natural Rubber Latex Density. The observation technique of the test is done by FTIR PP-g-MA analysis, stability test, TPE visual analysis, TPE surface morphology using Scanning Electron Microscopy (SEM) tool and TPE tensile strength test. The results of FTIR analysis is that the samples closest to the carbonyl value of C = O with the highest absorption were without glycerine samples 1703.48 cm⁻¹ with the absorption of 94.84% and carbonyl C-O 1219 cm⁻¹ with the absorption of 95.19%. The stability testing of density values reaches the standard of the Plastic Federation of 0.91 - 1.30 g/mL, for samples having the highest and stable density values up to the seventh day of observation is a sample of PP-g-MA: Latex (75:25) which is 1.059 g/mL. In the SEM test on the PP-g-MA sample: Latex (75:25) with a average diameter pores size of 1.408 μm and the smallest diameter pore size of 0.728 μm. The highest value of tensile strength occurred in the sample with the comparison of PP-g-MA: Latex (75:25) 1.175 N/mm² and a maximum elongation of 22.62%.

Keywords
Polypropylene, Latex, Emulsion Method, Thermoplastic Elastomers

Received: 13 August 2018, Accepted: 13 October 2018
https://doi.org/10.26554/sti.2018.3.4.151-156

1. INTRODUCTION

Indonesia is a developing country which has increasing population that causes increasing population that causes increasing of transportation vehicles as it can ease citizens’ mobility. According to BPS (Centre of National Statistics Department) data in 2016, Indonesia has 14,580,666 units of car and this number increases every year of 1 million units. The increase of cars makes unavoidable traffic and accident problems, as 98.9 thousand accident cases has happened. In car accident, the body of car is usually bumped, crushed or broken. It happened because the body of car is usually bumped, crushed, or broken. It happens because the body of car is made of plastic material from petrochemical industry.

The petrochemical industry is a chemical industry that processes raw material of petroleum, natural gas or coal through the chemical process of physics, which produces a various chemical products of basic/upstream petrochemical product, intermediate petrochemical product or downstream petrochemical. The downstream petrochemical industry is an industry that produces petrochemical products in the form of final products or finished products.

Petrochemical basic materials—natural gas and their derivatives, i.e. ethane, propane, and butane, as well as crude petroleum derivatives, i.e. naphtha will provide petrochemical products such as olefins (alkenes), i.e. ethylene, propylene, and butadiene. The demand for petrochemical materials such as ethylene, propylene is commonly used as raw materials in the manufacture of plastics (thermoplastics). Thermoplastic materials combined with natural rubber will produce thermoplastic elastomers type materials.

The thermoplastic elastomers is a polymer block having elastic properties at room temperature up to 70 °C. The elasticity is caused by the nature of the physical crosslinking resulting from the intermolecular conductivity. The bond will be interrupted when the thermoplastic elastomers is heated over a
certain temperature and re-formed when cooled. The preparation of thermoplastic elastomers can be carried out in both liquid and emulsion phases.

According to Ismail and Suryadiansyah (2002), thermoplastic elastomers (TPE) provides better material utilization than thermosetting materials. The advantages of using TPE are it has simple compounds and rapid preparation; moreover, the byproducts are easily reprocessed and recycled (Pongdhorn sae-oui, dkk. 2010).

Polypolypropylene (PP) is an additional thermoplastic polymer with a large molecular weight distribution (Meyer and Keurentjes, 2005). According to Brydson (1999), there are three types of PP that are atatic (PPi), isotactic (IPP) and syndiotactic (SPP). PP has unique properties such as high melt temperature, low density, high chemical resistance, and heat resistance.

Indonesia is one of the world’s natural rubber producing countries with the production of about 2.7 million tons per year, the world’s synthetic rubber production about 12.941 million tons. The utilization of this natural rubber for non-tire materials such as latex products for TPE material making is only about 30% (Sondari et al., 2010) so to get the high demand for TPE, Indonesia annually imports TPE materials from other countries. TPE imports cause TPE prices to be very expensive. According to BPS data, Indonesia has imported TPE materials of 68.62 tons in 1994, while the opportunity to make TPE from natural rubber is available (Deswita et al., 2006). In the year 2000-2004, there was an average increase of 7.87 kilotons.

Based on the literature study, this research used latex as elastomers and polypolypropylene (PP) material because these materials are commercial polymer material, have high chemical resistance and heat resistance, which is expected that the mixture of propylene and natural rubber latex made by emulsion blending method will produce in a homogeneous TPE and strong tensile strength.

2. EXPERIMENTAL SECTION

2.1 Materials and Tools
The materials used in this research are Granules Isotactic Polypropylene (PP) for analysis 99.6%, Maleic anhydride (MA), Xylene for analysis 60%, Benzene for analysis 60%, Hydrogen peroxide for analysis, Glycerin pure, Carboxymethylcellulose (CMC), Latex 60% natural rubber.

Equipment used in this research includes: Mixer Philips 60 rpm, Beaker Glass 500 ml, Erlenmeyer 250 ml, Magnetic Heated Stirrer HMS-79, 300 °C thermometer, Spatula with Spoon & 30 cm stirrer, Analytical Balance OHAUS 200g, Scanning Electron Microscope JEOL 6510LA, Oven Memmert 220 °C, Hydraulic Universal Material Tester 50 kN, IR spectrophotometer.

2.2 Procedure
2.2.1 Grafting Polypropylene (PP) with Maleate Anhydride (MA)
Make grafting Polypropylene with Maleate Anhydride (PP-g-MA) by adding 1 gram Maleate Anhydride added 90 mL xylene, then add 20 gr polypropylene. Heat in an Erlemeyer for 20 minutes at 170 °C, in order to blend all the ingredients perfectly, so that grafting can occur subsequently dissolved in 1 mL of benzene, and 1 mL of hydrogen peroxide with 10 mL of xylene and added to the mixture first, for 10 minutes, benzoyl peroxide serves as an oxidizer.

2.2.2 Making PP-g-MA Emulsion
Conducted within the beaker glass, the PP-g-MA emulsion was obtained by mixing the PP-g-MA solution in xylene at a concentration of 30% (v/v) stirring rate ± 200-400 rpm followed by addition of 10% glycerine emulsifier, 200-400 rpm.

2.2.3 Making PP-g-MA Emulsion with Natural Rubber Sensitive Latex
Conducted in beaker glass equipped with high-speed stirrer. Furthermore, the second mixture of latex is stored for stable emulsion stability. The thermoplastic elastomer emulsion is made by mixing the PP-g-MA emulsion with natural rubber latex (LPKA). The result of mixing of PP-g-MA emulsion with LPKA in various ratios using 10% glycerine emulsifier is called a thermoplastic elastomer emulsion.

2.2.4 Testing
The test was performed by analyzing the physical, chemical, and mechanical properties, on the analysis of physical properties measured density value and pore diameter size on SEM equipment, chemical analysis was done by observing the absorption of wavelength of C = O and CO yield from grafting PP-g-MA.

3. RESULTS AND DISCUSSION

3.1 Fourier Transform Infrared Test Result (FTIR)
The FTIR test conducted at the Islamic University of Indonesia, the specification of the tool used the FTIR UATR Spectrum Two Perkin Elmer, the FTIR is used to place the snapshot on the diamond censor (to reflect the infrared ray) and then copy data on the computer screen to identify the wave crests from the group C = O and CO yield from grafting PP-g-MA.

There were 4 test pieces consisting of non-emulsion PP-g-MA without glycerine, PP-g-MA by emulsion method on PP-latex comparison sample 25:75, 50:50, and 75:25.

3.2 Density Test Result
The density test is started by measuring the mass the thermoplastic elastomers. The volume of the thermoplastic elastomers is then measured by immersing it to the measuring cylinder filled with water. The stability of density can be determined by
Figure 1. The peak graph of FTIR for sample PP-g-MA: Latex (75 : 25) without glycerine for 1703,48 cm\(^{-1}\) adsorption functional group C=O and for 1219,95 cm\(^{-1}\) adsorption functional group carbonil C=O.

Figure 2. The peak graph of FTIR for sample PP-g-MA emulsion with glycerine sample PP-g-MA : Latex (25 : 75) for 1660,80 cm\(^{-1}\) adsorption functional group C=O and for 1219,95 cm\(^{-1}\) adsorption functional group carbonil C=O.

Figure 3. The peak graph of FTIR for sample PP-g-MA emulsion with glycerine sample PP-g-MA : Latex (50 : 50) for 1685,96 cm\(^{-1}\) adsorption functional group C=O and for 1218,56 cm\(^{-1}\) adsorption functional group carbonil C=O.

Figure 4. The peak graph of FTIR for sample PP-g-MA emulsion with glycerine sample PP-g-MA : Latex (50 : 50) for 1663,60 cm\(^{-1}\) adsorption functional group C=O and for 1213,96 cm\(^{-1}\) adsorption functional group carbonil C=O.
Figure 5. Diagram of thermoplastic elastomer density measurement on sample PP-g-MA : Latex (75 : 25), the value of density did not change which is stable at 1.059 g/mL.

Figure 6. The average pore diameter of Sample thermoplastic elastomers PP-g-MA : Latex (75 : 25) without glycerine as emulsifier is 11.175 µm and the smallest pore on the surface area thermoplastic elastomer 1.275 µm.

one-week observation. We observed its density in the first day, third day, and seventh day. The density data results are shown on figure 5.

3.3 Thermoplastic Elastomers Surface Morphology
The morphological analysis was conducted at Jakarta State University Department of Materials Engineering. The observation was done by using JEOL 6510LA Scanning Microscopy Electronic (SEM) with 2000 X magnification. Figures 6 until 9 are the microstructure analysis by showing the pore size of the thermoplastic elastomer surface.

3.4 The Result of Mechanical Properties of the Thermoplastic Elastomers
Testing of mechanical properties with a tensile strength test is conducted at Polytechnic of Sriwijaya by using Hydraulic Universal Tester 50 kN tensile strength test apparatus. The sample was clamped on the apparatus and then several forces are applied to the sample until it reaches its maximum load limit. Data of tensile strength test results can be seen in Table 1.

Figure 7. The average pore diameter of Sample thermoplastic elastomers PP-g-MA : Latex (75 : 25) with glycerine as emulsifier is 1.408 µm and the smallest pore on the surface area thermoplastic elastomer 0.728 µm.

Figure 8. The average pore diameter of Sample thermoplastic elastomers PP-g-MA : Latex (50 : 50) with glycerine as emulsifier is 3.596 µm and the smallest pore on the surface area thermoplastic elastomer 0.922 µm.

Figure 9. The average pore diameter of Sample thermoplastic elastomers PP-g-MA : Latex (25 : 75) with glycerine as emulsifier is 1.614 µm and the smallest pore on the surface area thermoplastic elastomer 0.971 µm.
In this research, fabrication of thermoplastic elastomers with polypropylene raw material was processed by grafting Maleate Anhydride and Emulsion on Natural rubber concentrated Latex by using glycerin and Carboxymethylcellulose (CMC) with comparison of sample of PP-g-MA: Latex of 75:25 (3:1), 50:50 (1:1) and 25:75 (8:1). Tests conducted on this research include physical, chemical and mechanical analysis. The test follows British Standard of Plastic Federation and standard tensile strength for bumper material. In the physical analysis, the density test was conducted by observing the density for 7 days with first, third and seventh day observations, aimed to analyze the emulsion stability of the thermoplastic. The test is carried out manually by measuring the mass of thermoplastic elastomers samples and immersing it in water then the increasing volume is recorded as the volume of the sample. The density data recorded in all three samples has a fixed density of the sample with the PP-g-MA ratio: Latex (75:25) of 1.059 g/mL. PP-g-MA particle fills the matrix space of the flexible latex so that the sample density is stable. This value has also met the density standard value of the thermoplastic elastomers of the British Plastic Federation of 0.91-1.30 g/mL.

Physical analysis is also done by analysing the diameter of pores of thermoplastic elastomers. When the diameter of pores is greater, its tensile strength and density are smaller and vice versa. The smaller pores diameter indicates that PP-g-MA particles make the sample more dense and has better tensile strength. The size of the pores can be affected by the technique of blending and moulding manually when preparing the thermoplastic elastomers product, the measured pore diameter size is high value with the micrometer (μm) unit where the result data on the average pore size is the smallest in the ratio sample PP-g-MA: Latex 75:25 (3:1) ie 1.408 μm with the smallest pore diameter 0.728 μm.

The analysis of chemical properties is conducted by using FTIR instrument to analyze carbonyl bonds C = O and C-O occurring in grafting method performed on polypropylene and maleic anhydride. From the result data, it is found that wavelength absorption of C = O has met the adsorption standard C = O group of 1600 -1750 cm⁻¹. Sample PP-g-MA: Latex (75 : 25) without glycerine of polypropylene grafting with pure maleate anhydride before the emulsion treatment successfully has wavelength data of 1703.48 cm⁻¹ with a percentage of 94.84%. The lowest absorption percentage of the wavelength of the C = O group occurs in the comparison of PP-g-MA: Latex of 75:25 (3:1) with the value of 86.48%. In the C-O group, the PP-g-MA : Latex (75 : 25) without glycerine sample has a wavelength of 1219.95 cm⁻¹ with an absorption percentage of 95.19%, then the sample which has the lowest absorption percentage in the 75: 25 (3: 1) sample is 85.46%. The difference in absorption percentage is due to the influence of an emulsion method which adds emulsifier glycerin and solid particle vibration to Polypropylene which causes the absorption intensity at C = O and C-O group to decrease to 8.36%.

In the analysis of mechanical properties measured by tensile strength test in units (N/ mm²), various forces are applied to the samples until it reaches its maximum limit. Based on the standard British plastic federation and tensile strength testing standards of the tensile strength bumper material in the range of 0.5 to 2.4 (N / mm²), it is found that three thermoplastic elastomers samples made by comparison of PP-g-MA: Latex (25:75), (50:50) and (75:25) have met the standard values of 0.926, 1.022, and 1.175 N / mm². The highest value of tensile strength of the sample in the comparison of PP-g-MA: Latex (75:25) at 1.175 N / mm², this corresponds to the small surface pores of 0.728 μm and the high density 1.059 g / mL so that the strength between the matrix and the filler on the thermoplastic elastomers have strong bond. It is proved by the largest tensile strength test value of 1.175 N / mm². The linear relationship between tensile strength and elongation meet the standard bumper tensile test with a maximum of 22.62% while the standard maximum elongation is 22.62%.

3.5 Discussion

Table 1. Tensile strength test results on thermoplastic elastomers PP-g-MA and latex by emulsion method

<table>
<thead>
<tr>
<th>Method</th>
<th>Specimen (PP-g-MA:Latex)</th>
<th>B (mm)</th>
<th>H (mm)</th>
<th>Area (mm²)</th>
<th>Max Load (N)</th>
<th>Tensile strength (N/mm²)</th>
<th>Maximum Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unemulsion (without glycerine)</td>
<td>75:25</td>
<td>25</td>
<td>6</td>
<td>150</td>
<td>70.7</td>
<td>0.471</td>
<td>17.46</td>
</tr>
<tr>
<td></td>
<td>50:50</td>
<td>25</td>
<td>6</td>
<td>150</td>
<td>138.9</td>
<td>0.926</td>
<td>19.71</td>
</tr>
<tr>
<td>Emulsion</td>
<td>75:25</td>
<td>25</td>
<td>6</td>
<td>150</td>
<td>138.9</td>
<td>0.926</td>
<td>19.71</td>
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<td>50:50</td>
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<td></td>
<td>25:75</td>
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<td>150</td>
<td>138.9</td>
<td>0.926</td>
<td>19.71</td>
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4. CONCLUSIONS

Based on FTIR analysis, the without glycerine sample reaches the keton value of C = O with the highest absorption of 1703.48 cm⁻¹ with absorption percentage of 94.84% and C-O carbonyl absorption of 1219 cm⁻¹ with absorption percentage of 95.19%. This is because the grafting samples are pure and have not been emulsified so that the intensity of the grafting is still high from the carbonic group CO and C = O male anhydride. The stability testing of density values reaches the standard of the Plastic Federation of 0.91 - 1.30 g / mL, for samples having the highest and stable density values up to the seventh day of observation is a sample of PP-g-MA: Latex (75:25) which is 1.059 g/mL. Meanwhile, based on SEM test on the PP-g-MA
sample: Latex (75:25) has a mean pore value of 1.408 µm and the smallest pore value at 0.728 µm. The highest value of tensile strength occurred in the sample with the comparison of PP-g-MA: Latex (75:25) 1,175 N / mm² and a breaking extension of 22.62%.

REFERENCES


