Eco-friendly Fibre from Recycled Polypropylene of Bottle Cap Waste and Lignin

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Abstract. Ecofriendly fibre is one of potential alternatives to fulfill the rising demand in textile material supply which is limited due to the decreasing reserve of oil. Large amount of polypropylene waste from bottle cap and lignin as a byproduct from pulp industry are potential solutions. Grinded polypropylene bottle cap was blended with lignin powder in concentration of 5 wt. % processed by melt spinning at 170°C temperature. The fibres produced have an average diameter 170 and 250 micrometres. In view of the mechanical properties, the tensile strength is 11.9 MPa for fibre with 170 micrometres diameter and 14.7 MPa for fibre with 250 micrometres diameter. Fibre surface morphology was further studied using micron microscope, and the result shows black flocks spread in the fibre, indicating that the lignin does not blend evenly.

1. Introduction
Indonesia stands as the second biggest mismanage plastic waste country in the world after China [1]. This condition is potentially getting worse due to increment of plastic products demand as the population growth. On the other hand, world oil production as main material for plastic product including synthetic fibre has been decreasing. One largely applied solution for this issue is by reusing, reducing and recycling all plastic products including plastic bottle cap. However, recycling the bottle cap waste as fibre material has not been studied

Currently, many apparel producers are making eco-friendly fibre from recycled polyethylene terephthalate of plastic bottle waste. Study about synthetic fibre from plastic bottle cap waste is very interesting due to the abundant supply of this waste. According to Container Recycling Institute, at least there were 42.6 billions unit plastic bottle sold at 2010 in US [2]. The estimated waste weight is around 76,680 tons (average bottle cap weight is 1.8 grams). Moreover, Polypropylene also has interesting properties such as low melt processing temperature, low thermal conductivity (lower than wool) which is very useful for apparel in cold weather, good mechanical properties stability in wet condition, excellent chlorine resistance, etc. In addition, there is another material which is also abundant in the nature, the material is called lignin.
Lignin is the part of wood beside cellulose and other components. Lignin is the second most abundant biopolymer in the world after cellulose [3] readily available in the nature and also relatively inexpensive [4]. Lignin is also byproduct of pulp and paper industries which are using kraft process. Through this process, cellulose is isolated from wood and the other wood components including lignin are extracted into black liquor. Lignin can be gained by precipitating black liquor. This process well known as bio-refinery and currently available commercially.

In the past, there were some experiments about virgin polypropylene and lignin fibre. This paper focuses on producing and characterizing fibre from recycled polypropylene of bottle cap waste and lignin.

2. Experimental

2.1. Materials

Lignin for this research is obtained from Domtar Biochoice lignin. This lignin is byproduct of kraft process which only uses southern pine woods. The presentation of the solids is 66.31 %, presentation of the ash is 0.46% & pH is 3.45. Recycled polypropylene is taken from single plastic bottle cap waste. The bottle cap waste is cleaned prior blending process. The blending composition of polypropylene/lignin is 95/5 by weight.

2.1.1. Lignin analysis

Thermal analysis of the lignin was performed with differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) using a LINSEIS STA Platinum Series (simultaneous thermal analysis). DSC analyses were conducted with heating rate of 10 °C /min until the temperature of 310 °C. Sample weights were 5.3 ± 0.5 mg. Fourier Transform Infra Red (FTIR) analysis was run on Shimadzu.

Figure 1. Lignin FTIR spectra
The FTIR spectra is in line with the information from supplier information that lignin material was obtained from softwood (southern pine). The softwood FTIR spectra has more intensity absorption band at 1269 cm\(^{-1}\) than at 1230 cm\(^{-1}\) and the intensive absorption existed on 1033 cm\(^{-1}\) (around 1030 cm\(^{-1}\)) is very typical for softwood lignin spectra [5]. The melting (softening) temperature of lignin was found around 187 °C.

**Figure 2.** Lignin DSC analysis

**Figure 3.** Lignin TGA analysis
2.1.2. Polypropylene waste analysis
Thermal analysis of the polypropylene was performed with differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) using same equipment that used for analysing lignin with same heating rate until the temperature of 310 °C. Sample weights were 1.8 ± 0.5 mg. Fourier Transform Infra Red (FTIR) analysis was run on Shimadzu.

The FTIR spectra relatively is similar with the spectra in the handbook. FTIR spectra shows the intense absorption at 1087, 1465, 1381, 2850 & 2918 cm\(^{-1}\) [6]. The melting temperature of polypropylene was found around 125 °C.

![Polypropylene waste FTIR spectra](image)
2.2. Melt spinning machine
Melt spinning machine is built using plunger and dead weight to push molten polymer out through spinneret. The spinneret has single hole in 5 mm diameter. Autonics TZ4M is used as Temperature controller in this spinning machine. Its Digital temperature controllers offer quick setup and provide precise temperature management. This melt spinning machine uses Fotek Solid state relay with current load 40 Ampere. The maximum temperature can be reached with this melt spinning machine is 310 °C with heating rate is 7 °C / min. Fibre winder is using AC motor with capacity 480 – 600 RPM.

2.3. Fibre spinning
Thermal extrusion of the lignin fibers was performed at 170 ± 2 °C. After reaching the desired temperature, the heating temperature was maintained for 20 minutes to ensure the uniformity of molten polymer temperature. The molten polymer then quenched in open air at 24 °C temperature and drafted by winder. The fibres diameter were arranged by drafting setting. In this research the fibre diameter target was 170 micrometres (Fibre A) & 250 micrometres (Fibre B) concerning the broken fibre factor when the fibre spinning was conducted.

2.4. Mechanical strength of fibre
The mechanical strength of the fibers was measured in accordance with the ASTM standard (D 3379-75) with Mesdan Tensolab using a 1000 N load cell and a gauge length of 2.5 cm. Fibre diameters were examined using a calibrated Nikon optical microscope with Dinolite software assistance.

3. Result and discussion

3.1. Fibre diameter
The fibre diameter of these fibres varies with slubs (thick places which has bigger diameter in yarn/fibre) which are distributed unevenly. The slub places were distributed unevenly. This condition may cause broken fibre which will lead to production problem. For woven textile application, this condition will lead to production problem because it may cause broken fibre/yarn when the loom is running. The varying fibre diameter will also cause problem in machine setting such as the yarn tensioner at the knitting machine or gun hole at the loom. However, these slubs can create unique appearance in textile product, which are used by jeans maker for their product.

![Figure 7. Fibre A with diameter target 170 micrometres](image)

![Figure 8. Fibre B with diameter target 250 micrometres](image)

### 3.2. Fibre surface

The images from micron microscope shows black flocks within the fibre. The flocks are distributed unevenly and vary in size. These flocks are actually a group of lignin which is not well melted and
blended with the polypropylene. Although the dimension of both materials have been grinded (to achieve minimum dimension), pounded and mixed, the molten polymer seems to be not well blended. The processing temperature and compatibility of both polymers need to be studied further to achieve better blends.

**Figure 9.** Black flock within fibre A surface (170 micrometres diameter)

**Figure 10.** Black flock within fibre B surface (250 micrometres diameter)

### 3.3. Ultimate tensile strength of fibre

Ultimate tensile strength of fibre B with 0.25 mm diameter has higher ultimate tensile strength than fibre A. Both fibres were produced using same spinneret diameter and same plunger weight to push molten polymer out of spinneret, so the volume of polypropylene and lignin which passed through the spinneret were the same in both condition. The lignin is flocking and is not well blended with polypropylene and the dimensions range of flocking lignin for both condition are same. The difference of RPM winder results on the difference of fibre diameter due to draft resulted. The RPM winder is proportional to the draft resulted. The higher draft resulted, the smaller fibre diameter resulted. This condition will also affect to increment of polymer molecular orientation degree [7] as we can see in Figure 5. The increment of polymer molecular orientation may improve fibre strength.
According to tensile testing result in Figure 12, fibre B has higher ultimate tensile strength than fibre A. This condition is not in line with relation of polymer molecular orientation with tensile strength but the result from fibre surface analysis shows that the fibre surface of fibre A is rougher than fibre B as we can see in Figure 13 and 14. This condition may cause increment of stress concentration and broken fibre in tensile strength test.

**Figure 11.** Polymer crystalinity and molecular orientation

**Figure 12.** Maximum force and ultimate tensile strength of fibres

**Figure 13.** Surface of fibre A (170 micrometres diameter)

**Figure 14.** Surface fibre B (250 micrometres diameter)
4. Conclusions
Recycled polypropylene and lignin are potential materials for synthetic fibre, because both material has enough spinability to produce fibre by using melt spinning method. Further study is needed to produce high mechanical properties of recycled polypropylene lignin fibre, particularly to create good polymer blending. A good polymer blending will lead to even distribution of polypropylene and lignin in the fibre. The surface of fibre is also important to support mechanical properties of fibre resulted. All these conditions are very important especially for technical textile application. Poor polymer blending potentially leads to problem such as broken fibre.

5. References
[1] Jenna R. Jambeck, Miriam Perryman, Roland Geyer, Anthony Andrady, Chris Wilcox, Theodore R. Siegler, Ramani Narayan, Kara Lavender Law, 2015, Plastic waste inputs from land into the ocean
[2] Susan V. Collins, Jason Medbury, Fatemeh Bagheri, Chris Saprnicht, February 12, 2015, Container Recycling Institute, sciencemag.org
[3] Brett G. Diehl, Nicole R. Brown, Curtis W. Frantz, Matthew R. Lumadue Fred, Accepted 28 April 2013, Effects of pyrolysis temperature on the chemical composition of refined softwood and hardwood lignins, Elsevier, Cannon CARBON 60 (2013) 531 – 537
[4] J.F. Kadla, S. Kubo, R.A. Venditti, R.D. Gilbert, A.L. Compere, W. Griffith C, accepted 8 July 2002, Lignin-based carbon fibers for composite fiber applications, Pergamon, Carbon 40 (2002) 2913–2920, 200
[5] Ivan Bykov, Characterization of natural and technical lignins using FTIR spectroscopy, 2008, Lulea University of Technology, Master Thesis, Department of Chemical Engineering dan Geosciences
[6] A.H. Kupstov & G.N. Zhinzhin, 1998, Handbook of Fourier transform raman and infrared spectra, Elsevier
[7] Raghavendra R. Hegde, M. G. Kamath, Atul Dahiya, Polymer crystallinity.

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