The Eurasia Proceedings of Science,
Engineering & Mathematics (EPSTEM)
ISSN: 2602-3199

The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM),2018

Volume 2, Pages 1-9

ICRES 2018: International Conference on Research in Education and Science

Preparation and Characterization of Biocomposite Polylactic Acid/Coconut Fibre

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Abstract: In this research, biocomposite was prepared by using coconut fiber as filler and polylactic acid (PLA) as matrix. Coconut fiber undergo three different treatments which are sodium hydroxide, bleaching and maleic anhydride. Biocomposite was produced by varied composition of coconut fiber into 2%, 4% and 6% to be added into PLA. Characterization of coconut fiber was carried out by using infrared spectroscopy analysis (FTIR) analysis in order to study the changes in functional groups and degree of crystallinity of coconut fiber after treatment. Characterization of the biocomposite produced was carried out by using mechanical test to study the mechanical properties of biocomposites. Based on the results from FTIR analysis, certain functional group in original coconut fibre structure disappeared after chemical treatment. XRD analysis also showed that bleached coconut fibre has the highest crystallinity. Overall, the result from tensile test showed that maximum load and the modulus Young of biocomposite increase with increase composition of coconut fiber until an optimum point which is at 2% coconut fiber. While elongation at break decreased with increasing composition of coconut fiber.

Keywords: Biocomposite, Tensile

Introduction

The petrochemicals derived from synthetic polymers cause environmental pollution because of its biological disintegration. It is not only cost-effective, but it also poses a waste problem. This has prompted researchers to find solutions to address this problem. Composite is a material that results from mixing two or more materials (fillers or reinforcing elements with a matrix compatible) to form a specific character or characteristic. In the composites, fibers and matrices will maintain their physical and chemical properties in order to produce a combination of unreachable properties if only one component exists (Alemdar & Sain 2008). Fiber-reinforced composite polymers are produced when the fibers are mixed into a polymer matrix. The matrix will act as a binder to hold fibers, transfer the burden imposed on fibers, and protect against damage (Agarwal Bhagawan 1980). Fiber is generally a load bearing part, where the matrix around it maintains the fiber in a particular location and orientation. Biocomposites are defined as composites that incorporate the original fibers so like sisal, jut and kenaf with the same polymer where there is biodegradable or non-biodegradable. The biocomposite is composed of a matrix which may undergo the process of biodegradation and the original fibers act as a filler or a reinforcing agent. The development of biocomposites rather than the original biodegradable and fiber polymers has been of interest in the field of composite science because biocomposites justify the perfect decomposition in the soil by not releasing any harmful or toxic components (Lee & Wang 2006). Biocomposites that use biodegradable polymers are reinforced with the original gentian which help to reduce the surrounding natural problems and solve the problem of non-fixed petroleum sources. Examples of biopolymers based on the original source are poly (lactic acid) (PLA), plastic starch, cellulose plastic and plastic soya.

Synthetic polymers undergo slow decomposition processes due to the high molecular mass and have hydrophobic properties. In recent years biodegradable polymers with appropriate mechanical and physical properties have been given attention to replace petrochemical-based synthetic polymers (Iovino et al. 2008).
Hence renewable and cheaper natural fibers have been introduced to be a substitute for synthetic fibers (Lee & Wang 2006). Synthetic polymers are widely used in daily life because they are easy to process, have constant

stability, low cost and antibacterial properties (Satyanarayana et al. 2009). In this research, both raw materials namely PLA and coconut fiber were used to produce biocomposite materials. PLA provides high mechanical strength and PLA processing method is simple in most devices but its cost is high and fragile properties have limited its application. The best method to handle this problem is the addition of natural fibers that are bioserrated. The advantages of using natural fibers in composites are low cost, good thermal properties and low density. Therefore, the addition of natural fibers into the PLA polymer can reduce the overall cost by not destroying the biodegradation performance of the polymer matrix (Yussuf et al. 2010).

Research Methodology

In the study, all the chemicals and raw materials used for the preparation of the samples are as follows: Coconut fiber is supplied by MTS Fibromat (M) Sdn. Bhd while the PLA (lactic acid) PLA is from E Sun. Acetic acid and solid sodium hydroxide (NaOH) are supplied by Systerm Chem AR. Solid sodium chloride ($NaClO_2$) used as a bleach is derived from Agros Organics while malic anhydride (MA) is from Merck Schuchardt. In addition, silicon oils supplied by Systerm ChemAR are also used throughout the investigation.

Mitutoyo Measuring Tool

These measures are used to determine the thickness of the cut samples in the form of dumbbell. Measurement of sample thickness was taken on three different parts and the average readings obtained were recorded with ± 0.1

mm error.

Hollow Die Punch

This cutting tool is used to make a dumbbell shape on a 0.3mm thick sheet according to the 6051/000 standard.
Mixer Machine

The mixing machines used to provide matrices and composites are the PL 2 / Brabender Plasti-Coder model internal mixer. Measurement parameters such as temperature, rotor speed and time are set beforehand before blending takes

Strength Machine

The strain machine used is a mechanical testing machine (UTM) type Instron Universal model 5567 based on ASTM D 882/91.

Hydraulic Heat Press

The hydraulic heat suppressor used in the preparation of sample sheets is Carver Model 2697. The temperature and time are set before hand before the sample suppression begins.

Results and Discussion

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis is used to perform analysis on coconut fiber without treatment, alkaline treated coconut fiber, coconut fiber treated with bleach and coconut fiber treated MA. FTIR spectroscopy is used to examine any changes in the structure of the cellulose fibers that arise after chemical treatment is performed on the fibers. Identified types of bonds make sure to produce one infrared spectrum absorption. FTIR spectrum of fiber is shown in Figure 1.

There is a peak at about 3200 cm^{-1} to 3500 cm^{-1} on all the four absorption spectra shown. Based on Rout et al. (2001) the peak appearance of 3400 cm^{-1} in the four samples was due to the strain of O-H from hydrogen bonding of cellulose in the fibers. Additionally, from the spectrum shown peak at about 2906 cm^{-1} up to 2920

cm^{-1} are found in all samples. The peak is related to strain and vibrations by group-bonding (-CH2-) on cellulose and hemicellulose (Lopattananon et al. 2006). In addition there are two peaks around 898 cm^{-1} and 1059 cm^{-1} on all four absorption spectra. Based on Chen et al. (2009) the peak appearance of about 898 cm^{-1} and 1054 cm^{-1} is related to strain on C-O bonds and C-H vibrations within the cellulose structure.

A peak located at about 1734 cm^{-1} which distinguishes coconut fiber without treatment and with treated coconut fiber. Based on Alemdar et al. (2008), the peak at about 1737 cm^{-1} was due to the carbonyl bond. The appearance of this peak on untreated coconut fiber is due to the group of acetyl and ironic ester groups in the hemicellulose or ester chain for the carboxyl group in the lignin or hemicellulose. This peak is lost in treated coconut fiber because most lignin and hemicellulose have been removed. In addition, there is a loss of a peak around 1510-1513 cm^{-1} for the spectrum of coconut fiber treated with bleach, where this peak can distinguish coconut fiber treated with bleach and with other treatments or without treatment. Based on Alemdar et al. (2008) states that the summit around 1507 cm^{-1} is related to aromatic strain C = C for aromatic rings in the lignin. The loss of peak intensity is considered to be due to removal of lignin after coconut fiber is treated with bleach.

From the FTIR spectrum obtained, the extracted malachite extracts of anhydride, there is a unique peak that is absent in the other fiber FTIR spectrum of approximately 1706 cm^{-1}. Based on Chen et al. (2009), the peak of the wavelength range around 1700cm^{-1} is related to the carbonyl bond (C = O). The peak appearance in the maleic anhydride (MA) treated sample is due to the formation of an ester chain between the MA and the hydroxide group from the fibers and this peak is completely lost in alkali-treated fibers and bleach. In addition, there is a pathway change at the peak of approximately 3700-3000 cm^{-1} on the spectrum that distinguishes the maleic anhydride with the other. The top change in this coconut fiber sample is due to the change in the OH bond ratio between molecules and intramolecules (Cantero et al. 2003).

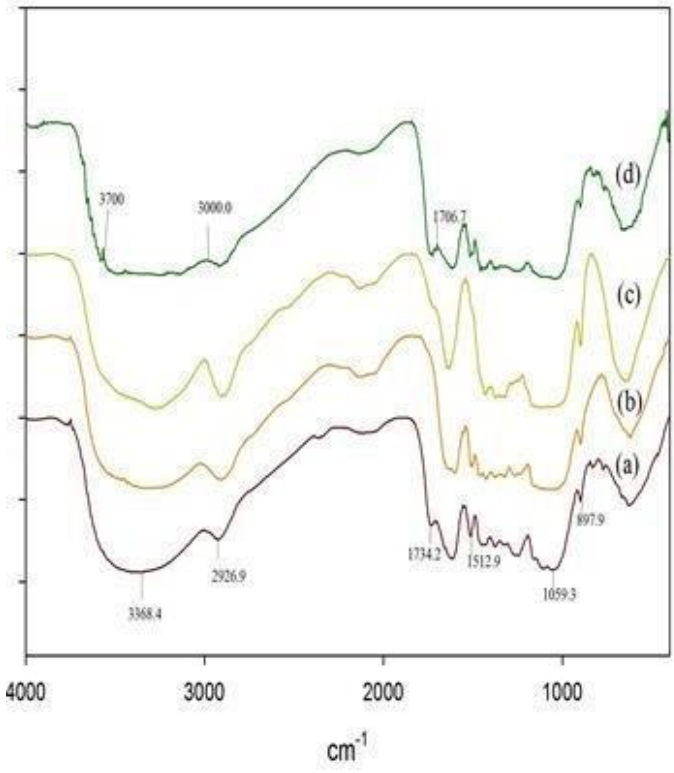

Figure 1 FTIR spectrum for (a) coconut fiber without treatment, (b) alkaline treated coconut fiber, (c) coconut fiber treated with bleach, (c) coconut fiber treated MA

Maximum Stress Test

Maximum stresses for PLA biocomposites enhanced by 0%, 2%, 4%, 6% non-treated coconut fiber, NaOH treated, bleached and treated with MA are presented in Figure 2 and the maximum stress value of each biocomposite is shown in Table 1 . From the results obtained, the maximum stress value of biocomposite reinforced by coconut fiber without treatment, treatment with NaOH, treatment with bleach and treatment with

MA is increasing until the optimum value of the 2% composition of coconut fiber and decreased steadily. The good distribution and the interface between the matrix and the fibers are critical factors for determining the mechanical properties of the resulting composites (Arrakhiz et al. 2012). Therefore, the treated coconut fiber has a higher maximum stress than the non-treatment of coconut fiber.

The maximum stress value decreases after the 2% content of the coconut fiber may be due to the addition of the filler which may interfere with the continuity of the polymer matrix. In addition, the decline in stress strength after an optimum level of achievement may also be due to the presence of large filler fibers that result in molding. Thus, the load transfer is not uniform because agglomeration occurs in the matrix (Liu et al. 2009). In addition, the increased composition of coconut fiber will increase the exposure of the interface area and weaken the interaction between the matrix and the filler. This resulted in a decrease in stress strength (Haque et al. 2009).

Based on the maximum stress value of the coconut fiber without treatment is 28.1 MPa while the maximum stress value of coconut fiber with NaOH treatment is 32.1 MPa. The increase in coconut fiber without treatment with NaOH treated coconut fiber was 4.0 MPa at 14%. This explanation is supported by Gu (2009) that compressive stress strength reinforced by alkaline treated coconut fiber increased after alkaline treatment was performed. This is because most fats, lignins and pectin covering the surface of the fibers will be removed and enhanced the interface bonding between the matrix and the filler. Alkaline treatment also reduces the diameter of the fibers and increases the aspect ratio and thereby enhances the mechanical properties of the composite (Mohanty et al. 2000).

In addition, from the results, the maximum stress value for treated fibers treated with bleach is higher than the maximum stress value of the untreated coconut fiber. The maximum stress value for the unmanaged coconut fiber is 28.1 MPa while the maximum stress value for the coconut fiber treated with bleach is 34.2 MPa. The increase in coconut fiber without treatment and with coconut fiber with bleach treatment was 6.1 MPa of 22%. Rosa et al. (2009) states that coconut fiber treated with bleach will produce a smooth surface by removing the wax coating and increasing the interaction between the matrix and filler based on the mechanic test. In addition, the removal of hemicellulose and lignin in natural fibers during treatment with bleach will result in good interaction between the matrix and the enhanced fiber (Mustata et al. 2012).

In addition, the maximum stress value of MA treatment treated with MA is higher than the maximum stress value of the untreated coconut fiber. From the maximum stress value of the unmanaged coconut fiber is 28.1 MPa while the maximum stress value for MA treated coconut fiber is 34.8 MPa. The increase in coconut fiber without treatment with coconut fiber with MA treatment was 6.7 MPa at 24%. Natural fiber treatment with MA will show the better mechanical properties is due to the reduction of wetting process by the formation of covalent bonds between MA molecules and fibers (Avella et al. 1995).

Table 1. Maximum stress value against the composition of coconut fiber

Composition of coconut fibre (%)	Maximum stress (MPa)			
	Without treatment	Treatment alkaline	Treatment bleach	Treatment MA
0	26.1	26.1	26.1	26.1
2	28.1	32.1	34.2	34.8
4	26.8	28.4	30.0	34.4
6	24.5	25.6	26.5	30.7

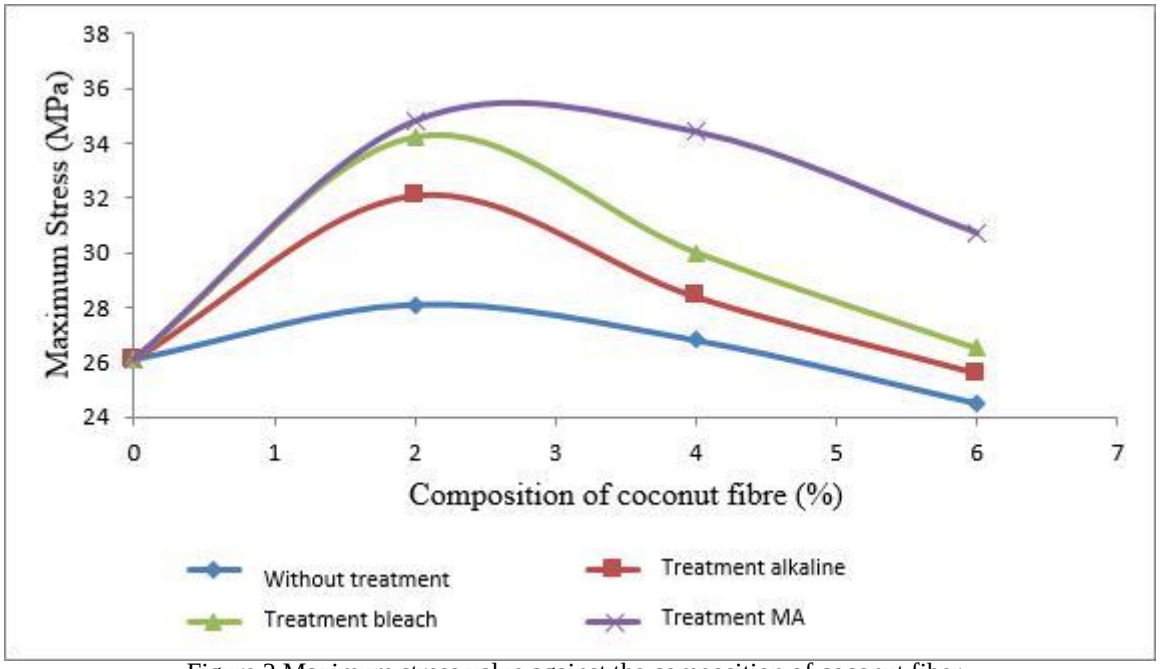

Figure 2 Maximum stress value against the composition of coconut fiber

Young Modulus

Young Modulus of PLA biocomposites enhanced by 0%, 2%, 4%, 6% non-treatment of treated, preserved NaOH coconut fiber, treated bleach and treated with MA is shown in Figure 3 and the results of Young modulus values for each biocomposite are shown in Table 2. From the results obtained, the value of Young's modulus of biocomposite reinforced by coconut fiber without treatment, treatment with NaOH, treatment with bleach and treatment with MA is increasing until the optimum value of the 2% composition of coconut fiber and decreased steadily. It is believed that the enhancement of filler content is aimed at enhancing the properties of rigidity and material stiffness (Ishak Ahmad & Abdullah 2009) and the high filler content is the cause of decreased composite mechanical properties caused by agglomeration process (Pickering et al. 2003)

Unprocessed coconut fiber has a lower Young modulus than the Young modulus value of NaOH treated coconut fiber. From the results of Young's modulus values of non-treatment of coconut fiber is 2278.7 MPa while Young's modulus value of coconut fiber with NaOH treatment is 2320.9 MPa. The increase in coconut fiber without treatment with coconut fiber with NaOH treatment was 42.2 MPa which was 1.85%. The increase in Young modulus of alkaline treated coconut fiber is due to the removal of non-cellulose components on the surface of the fibers. Removal of non-cellulose components will reduce the density of the fibers and the rigidity of the interfibrillar area and then make the microbibrill better able to rearrange the structure. Therefore, when a strain test is carried out a better load transfer is available (Arrakhiz et al. 2012). In addition, Mohanty et al. (2000) states that alkaline treatment will increase the interaction between the matrix and the filler due to the removal of natural impurities, thus affecting the natural fibers mechanical treatment and increasing the value of the modulus of the resulting composite sample.

Unprocessed coconut fiber has a lower Young modulus than the Young modulus value of treated bleached fibers. From the results of Young's modulus values of non-treatment of coconut fiber is 2278.7 MPa while the Young modulus value of treated bleached coconut fiber is 2346.6MPa. The increase in coconut fiber without treatment with coconut fiber with bleach treatment was 47.9 MPa, which was 3.0%. The increase in Young modulus values after bleaching is due to the removal of hemicellulose, lignin and pectin will enhance the mechanical properties of the composites and reinforce the properties of composite voltages. MA treatment of coconut fiber has a higher Young modulus than the Young Modulus value without treatment. From the results of Young's modulus value of non-treatment of coconut fiber is 2278.7 MPa while the Young modulus value of MA treated coconut fiber is 2390.0 MPa. The increase in coconut fiber without treatment with coconut fiber with MA treatment was 111.3 MPa which was 4.9%. Natural fiber treatment with MA has increased the wetness with the matrix and enhances the mechanical properties of the composite (Ma 2009).

Table 2. Young modulus values against the composition of coconut fiber

Composition of coconut fibre (%)	Young Modulus (MPa)			
	Without treatment	Treatment NaOH	Treatment bleach	Treatment MA
0	2107.5	2107.5	2107.5	2107.5
2	2278.7	2320.9	2346.6	2390.0
4	2250.3	2312.0	2326.4	2330.2
6	2164.9	2216.6	2231.0	2220.3

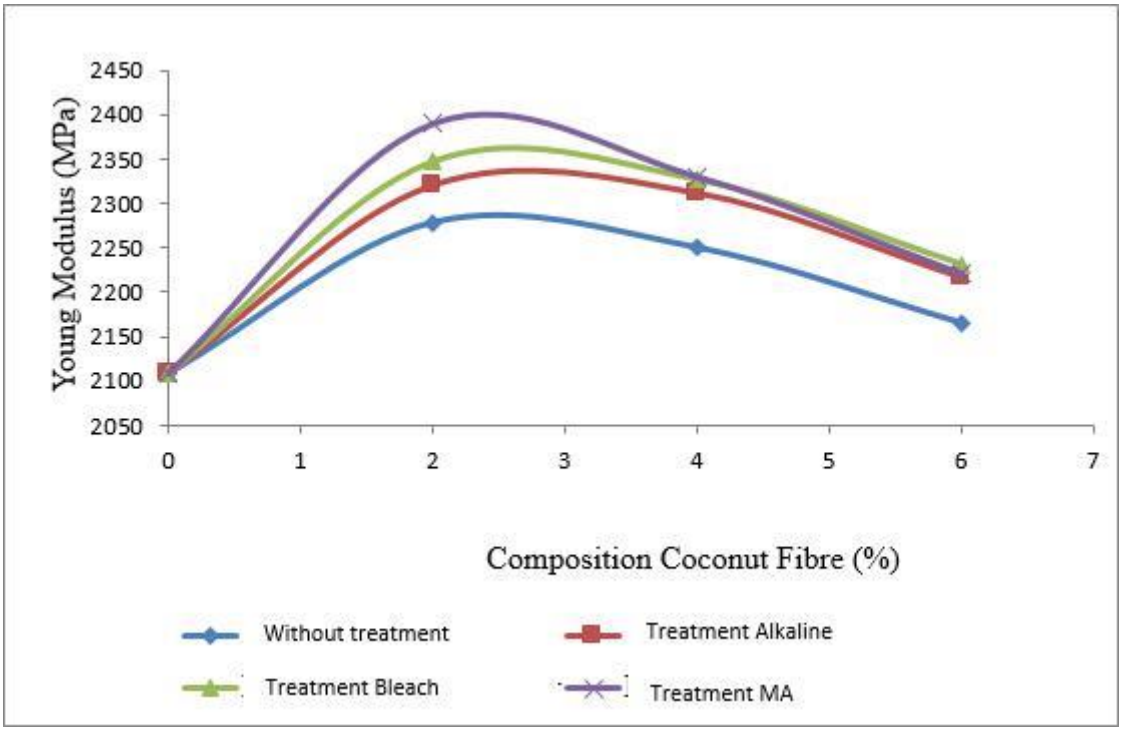

Figure 3. Young modulus values against the composition of coconut fiber

Strain on Backpoint

Strong strain of PLA biocomposites strengthened by 0%, 2%, 4%, 6% on non-treatment of treated, treated NaOH, treated with bleaching and MA treatment are shown in Figure 4 and the result of strain value at break point for each biocomposites are shown in Table 3. From the results obtained, the strain value at break point for biocomposite reinforced by coconut fiber without treatment, treatment with NaOH, bleach treatment and treatment with MA was reduced by the addition of the percentage of coconut fiber. Increased composition of filler will cause composite to become more rigid and hard. This has indirectly reduced the composite resistance and caused resistance to break down to become lower (Ismail et al. 2001). Based on Liu et al. (2009), The addition of fibers will result in a weak bond between the fibers and the biodegradation polymer and causes the formation of microcorrelation between the phases, resulting in a faster crack than the pure biodegradation polymer film.

The strain value at the breaking point of coconut fiber treated by chemical treatments has decreased. Decreased strain value is also due to uneven fiber agglomeration. Uneven fiber spread can cause some areas to have fibers only. Thus, the only fiber having the simpler micro-fracture and the only matrix will be weak (Din 2007).

K.Mohanty et al. (2005) states that alkaline treatment will reduce the diameter of the fibers and increase the aspect ratio thus improving the mechanical properties of the composites. The stiffness increase for natural fibers is due to crystallization (cellulose fibers).Overall, from Figure 4, the strain value at the breaking point of the non-treatable PLA-enhanced PLA biocomposites was the lowest and the treated MA with strain values at the highest breaking point. This is a chemical treatment solution that will increase the strain at the breaking point of the fiber. The strain on the breaking point of the unharmed fibers is low due to the three-dimensional cross-linking of cellulose and lignin. Chemical treatment will break the network structure and increase the strain at the breaking point (Kalia et al. 2009).

Coconut fiber treated with bleach has a strain value at dropping point which decreases overall. The decline in strain values at the breaking point is probably due to the agglomeration and the nonheterogenic size of the fiber in biocomposites (Averous & Boquillon 2004)

Coconut fiber treated with MA indicates inconsistent decline. From the graphs obtained, the strain value at the breaking point is increased to an optimum point of 2% of the coconut fiber treated with MA. However, the strain value at the breaking point is high, this can be explained by the treatment of the bonding of fibers that will cause the fibers to be more hydrophobic because the MA will react with the hydroxide group in the cellulose molecule and cause the hydrogen bond to break down and produce a strong bond between the cellulose molecule and the MA. Therefore, good attachment between matrix and filler can be produced (Mohanty et al. 2000).

Table 3. Strain value at the breaking point against the composition of the coconut fiber

Composition of coconut fibre (%)	Strain at break point(MPa)			
	Without treatment	Treatment alkaline	Treatment bleach	Treatment MA
0	2.6	2.6	2.6	2.6
2	2.3	2.4	2.5	3.1
4	2.2	2.3	2.3	3.7
6	2.1	2.0	2.1	2.5

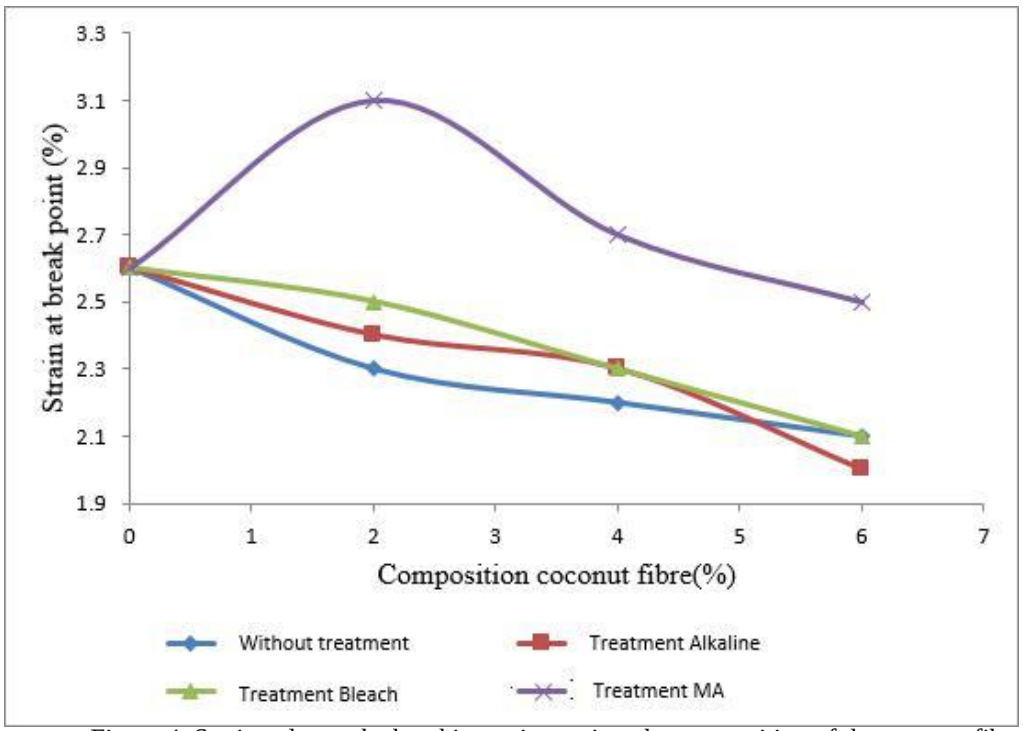

Figure 4. Strain value at the breaking point against the composition of the coconut fiber

Conclusion

This study was carried out for the preparation and characterization of poly-based biocomposites (lactic acid) which is reinforced by coconut fiber. Coconut fiber is treated with three types of treatments NaOH alkaline treatment, bleaching and anhydride treatment. The treatment composition of coconut fiber without treatment with certain treatment was varied to 2%, 4% and 6% to be added to the poly (lactic acid) matrix to produce biocomposites. FTIR infrared spectroscopy analysis was performed to characterize coconut fiber without treatment with certain treatments. Whereas the biocomposites produced will perform mechanical properties tests.

Overall, tensile tests, FTIR and morphological tests give a good result that the addition of coconut fiber fillers can improve the mechanical properties of biocomposites. In addition, chemical treatments carried out by coconut fiber will also increase the interaction between matrix with filler and improve mechanical properties of biocomposite.

FTIR analysis has been used to carry out analysis on coconut fiber without treatment, coconut fiber treated by NaOH, bleaching agents and MA. FTIR analysis is used to detect functional groups from Fourier Transform infrared spectrum records based on visible wave value. Through the analysis, increased cellulose content and removal of hemicellulose and lignin contents can be observed after alkaline treatment and bleaching process is carried out. Certain treatments will provide the removal of certain substances and provide changes in the structure of coconut fiber after chemical treatment.

The strain test results are to show the effectiveness of load transfer from matrix to fiber. The tensile test has three parameters discussed in the stress test, Young modulus and strain at break point. Overall, coconut fiber which treats with chemistry has the highest stress and modulus value as compared to the treatment without coconut fiber. This is because chemical treatments have removed lignin, hemicellulose and other foreign substances, increasing the coconut fiber bonding properties with the PLA matrix. Whereas the strain at the breaking point of the PLA-enhanced, non-treatment of biodegradable PLA showed a decline by the addition of coconut fiber composition.

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