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To cite this article: Mardawani Mohamad *et al* 2020 *IOP Conf. Ser.: Earth Environ. Sci.* **596** 012002

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The Effect of Alkaline Treatment on Mechanical Properties of Polylactic Acid Reinforced with Kenaf Fiber Mat Biocomposite

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Abstract. Kenaf fiber mat (KFM) reinforced polylactic acid (PLA) biocomposite have been developed by using film stacking and compression molding. In order to achieve the composite with good mechanical properties, the effect of processing parameters such as processing temperature and heating time on mechanical properties of PLA was studied. The optimum values of processing temperature and heating time on mechanical properties of PLA that were obtained were then being used to produce PLA/KFM biocomposite. PLA and kenaf fiber has distinct properties as natural fiber is hydrophilic whereas the polymer matrix is hydrophobic. In order to improve the fiber matrix adhesion, the KFM was chemically treated at different concentrations of NaOH for 3 hours at room temperature. Mechanical properties such as tensile and flexural strength, tensile and flexural modulus and elongation at break of untreated and alkali-treated PLA/KFM biocomposite were studied and compared. The obtained results showed that the alkali treatment have impact on the mechanical properties of PLA/KFM biocomposite. The study has demonstrated that the optimum alkali concentration was 4% NaOH concentration.

1. Introduction

The current awareness on global environmental issue such as natural resource depletion, emission from waste incineration and shortage of landfill space has exerted to preserve the natural resources. Natural resource such as fossil fuel is depleting every year with an increasing oil prices that has urged the public to minimize the dependence on the non-renewable resource. Research works have been carried out to reinforce the renewable and biodegradable plastic with natural fibers [1].

To reduce environmental burden from petroleum-based polymer material, development of bio-based composite has gained interest in polymer community. Biodegradable polymer such as polylactic acid (PLA), starch plastics, cellulose esters and aliphatic polyesters/co-polyesters has slowly replacing the non-biodegradable conventional plastics in various applications [2]. Among biodegradable plastics, PLA has been received significance attention because of its attractiveness being used in the production of biocomposite. PLA can be break down into carbon dioxide and water. PLA only requires low production energy as it can be obtained from renewable resources. Hence, it releases less



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greenhouse gases during production compared to non-renewable thermoplastic polymers because it does not contribute to environmental pollution and it has good mechanical properties that are comparable non-renewable thermoplastic such as polystyrene [3,4]. However, PLA has some deficiencies that can affect its applications, such as high production cost, brittleness, thermally unstable, low water vapour and gas barrier properties [5]. The production cost of PLA is lower when it reinforced with natural fiber.

Natural fiber is chosen as reinforcement agent because of its low cost, low density, environmental friendly, biodegradable, abundant and require less energy in producing composite compared to synthetic fiber. Among different types of natural fibers, kenaf have been chosen in reinforcing the composite. The fact that kenaf fiber is used as an ideal raw material because of its low cost, superior toughness and it grow very fast in 3 to 4 months [6]. Kenaf plant can be differentiated into two parts which are kenaf bast and kenaf core. Kenaf bast is the outer fiber while kenaf core is the inner fiber. The kenaf core fiber (KCF) has high content of hemicellulose and low content of cellulose which is major concern to be used in biocomposite fabrication. Studies have shown that kenaf bast fiber (KBF) composite possess higher tensile, flexural and impact properties than KCF composite due to the abundant cellulose content in KBF [7]. Chemical modification with alkali was employed to treat the surface of natural fiber to improve the interaction and mechanical properties between natural fiber and polymer matrix [8]. In this study, sodium hydroxide (NaOH) treatment was used to increase the interaction compatibility between PLA and kenaf bast fiber.

2. Materials and Methods

2.1 Materials

The materials that were used to develop the biocomposite is KBF in the form of non-woven mat, known as kenaf fiber mat (KFM). The kenaf fibers that used in this study were obtained from Lembaga Kenaf Tembakau Negara (LTKN), Kelantan.

2.1.1 Chemicals

The polymer matrix selected in this study is PLA that was obtained from the Nature Works LLC Company from USA (2003D). PLA has a density of 1.25g/cm^3 and a melting temperature of 150-180°C. The chemicals that were used in this study are sodium hydroxide in pellet form supplied by R&M Chemicals with molecular weight of 40 and acetic acid glacial supplied by Bendosen. Different concentration of sodium hydroxide such that 0, 2, 4, 6 and 8 wt% was used in the chemical treatment of KFM.

2.1.2 Preparation of Kenaf Fiber Mat

The kenaf bast fiber in the form of non-woven mat was used. The KFM was cut using a scissor and dimension of 150 mm long, 150 mm width and 3 mm thickness was taken. The KFM was dried in an oven at 60°C for 24 h to minimize the moisture content. The dried KFM were store in polyethylene bag to prevent penetration of water vapor prior to composite fabrication.

2.2 Preparation of PLA Sheets

The PLA pellets were dried in the oven at 60°C for 24 h to minimize the moisture content. The PLA in dumbbell shape were prepared under three different temperatures which were 170°C, 180°C and 190°C. The PLA was pre-heated for 10 minutes while the processing time varied from 10 minutes to 30 minutes and the cooling time was 6 minutes. The sample ID and corresponding composition was listed in Table 1. The optimized condition was used to prepare PLA samples and PLA/KFM composite.

Table 1. Processing parameters involved in the preparation of PLA sheets.

Sample ID	Process Temperature (°C)	Pre-heat time (minute)	Heating time (minute)	Cooling Time (minute)
S1	170	10	10	6
S2	170	10	20	6
S3	170	10	30	6
S4	180	10	10	6
S5	180	10	20	6
S6	180	10	30	6
S7	190	10	10	6
S8	190	10	20	6
S9	190	10	30	6

2.3 Chemical Treatment for Kenaf Fiber Mat (KFM)

NaOH in pellet form was selected for the fibers treatment. The alkaline solution concentration was used with a weight volume percentage (w/v%). The NaOH pellet was dissolved in distilled water at room temperature in this study. The weight of the NaOH pellet was obtained by using weight per volume formula.

Different concentration of NaOH and 1% acetic acid was used in chemical treatment method. The samples preparation was divided into two different groups, namely, untreated KFM and treated KFM. For preparation of treated KFM, KFM was treated by 2, 4, 6 and 8 wt% concentration of NaOH for 3 hours at room temperature. After treatment, the KFM was washed with distilled water containing 1 wt% acetic acid followed by rinsing with enough distilled water to remove any remaining NaOH. The purpose of washing by distilled water containing 1 wt% acetic acid is to neutralize the excess NaOH that remaining on the fiber surface. The sample ID and corresponding compositions are listed in Table 2.

Table 2. Treatment of KFM with different concentrations of NaOH in 3 hours of soaking duration at room temperature.

Sample ID	Concentration of NaOH (w/v %)	Soaking Duration (Hour)
Untreated KFM (UK)	0	0
2% Alkali-treated KFM (2% ALK)	2	3
4% Alkali-treated KFM (4% ALK)	4	3
6% Alkali-treated KFM (6% ALK)	6	3
8% Alkali-treated KFM (8% ALK)	8	3

2.4 Composite Preparation

The KFM with two PLA solid sheets, forming a three material layers were placed in a steel mold covered by stainless steel plates a both sides. Wax was used as releasing agent to avoid sticking of sheets with steel plates. The stacked materials were placed into compression moulding machine and pre-heated at 190°C without applying any pressure for 10 minutes to allow complete melting. The melted composite was then hot pressed at 190°C for 10 minutes under pressure of 5 ton. The steel mold was put in separate press of the machine and cooling was done for 6 minutes under 5 ton pressures at room temperature. Similar methods were employed for the biocomposite laminates with

different treated NaOH concentration of 2, 4, 6, 8 wt %. The PLA/KFM biocomposite was cut into required dimensions for mechanical tests.

2.5 Physical Characterization

2.5.1 Tensile Test

The tensile test was performed according to ASTM D3039 procedure using Instron Universal Testing machine (UTM) with a load cell of 50 kN at room temperature. The specimen was tested at cross head speed of 5 mm/min. The test specimens were prepared by cutting the KFM composite in rectangular form (150 mm x 25 mm x 3 mm) by using a jigsaw. The width and thickness of specimen was measured with a Vernier calliper before the tensile testing was conducted where the accuracy of the reading was up to 0.1 mm.

2.5.2 Flexural Test

The flexural test is to measure the force required to bend a beam under three-point loading condition. The flexural test was performed according to ASTM D790 procedure using Instron Universal Testing machine (UTM) with load cell of 50 kN at room temperature. The span-to-depth ratio was 1:16 and tested at cross head speed of 5 mm/min. The test specimens were prepared by cutting the KFM composite in rectangular form (150 mm x 25 mm x 3 mm) by using jigsaw.

3. Results and Discussion

3.1 Mechanical Properties of Polylactic Acid (PLA)

3.1.1 Tensile Strength of Polylactic Acid (PLA)

Figure 1 presents the measured average tensile strength of three specimens involved the pure PLA processed at 170°C, 180°C, 190°C and three different heating time which were 10 minutes, 20 minutes and 30 minutes. The tensile strength decreased from 43.60 MPa to 26.60 MPa when temperature increased from 170°C to 190°C. These findings presented that the tensile strength of PLA reduced when the processing temperature increased from 170°C to 190°C at 20 and 30 minutes of heating time. At high temperature and long heating time, the PLA will undergo thermal degradation which leads to reduction in tensile strength and becoming more brittle. The thermal degradation in PLA was due to the chain splitting. The PLA tensile strength that processed at 10 minutes heating has decreased from 45.53 MPa to 42.12 MPa when the temperature increased from 170°C to 180°C. However, it was noticed that the tensile strength of PLA increased from 42.12 MPa to 50.39 MPa when the temperature increased from 180°C to 190°C. This could be due to the PLA pellets has completely melted at high temperature and low heating time which resulted in high tensile strength. The tensile strength for 170°C and 180°C at 10 minutes heating were low could be due to incomplete melting of PLA which resulted brittle. The specimen processed at 190°C, 10 minutes heating recorded the highest tensile strength value of 50.39 MPa followed by PLA processed at 170°C with the value of 45.53 MPa. Therefore, these two conditions were used to produce PLA/KFM biocomposite.

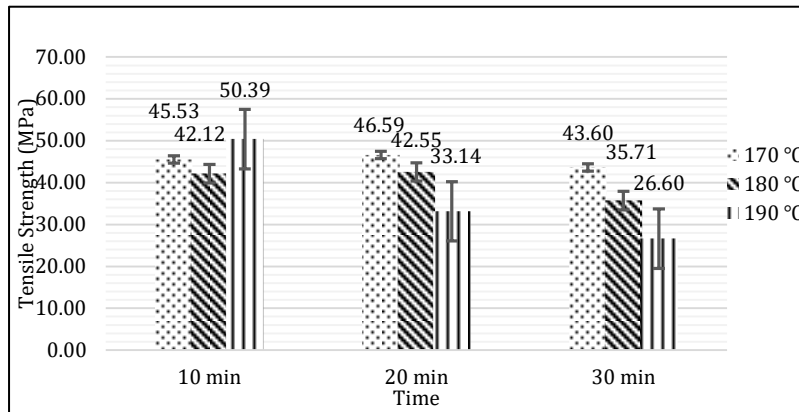


Figure 1. Tensile strength of PLA at different temperature and heating time.

3.1.2 Tensile Strength of PLA/Kenaf Biocomposite

Figure 2 shows the measured average tensile strength of three specimen involved pure PLA, untreated and 2, 4, 6 and 8% alkali-treated PLA/KFM composite at different temperature.

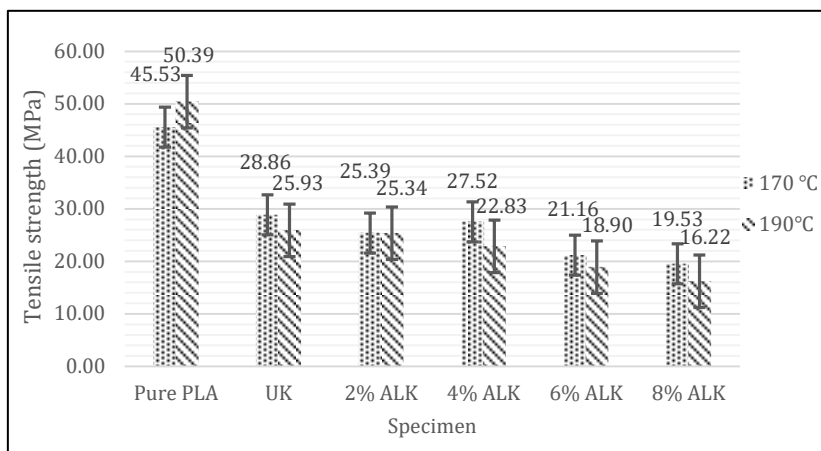


Figure 2. Tensile strength of PLA/Kenaf composite at different temperature and NaOH concentration.

As illustrated in Figure 2, PLA/KFM composite that was processed at 170°C increased in tensile strength from 25.39 MPa in 2% alkali-treated kenaf to 27.52 MPa in 4% alkali-treated PLA/KFM composite. However, the tensile strength has decreased to 21.16 MPa followed by 19.53 MPa in 6% and 8% alkali-treated PLA/KFM composite respectively. The results were similar with the findings of Ibrahim et al. (2010) [9] that the 4% NaOH concentration gave the highest tensile strength among the treated fiber. The fiber treated with 6% and 8% NaOH concentration showed decreased tensile strength were mainly due to lignocelluloses has degraded and the fiber surface has ruptured. A high concentration of NaOH would damage the fiber and as a result the tensile strength of the fiber will be decreased [10]. The 2% alkali-treated PLA/KFM composite has lower tensile strength compare to 4% PLA/KFM composite which could be due to low concentration of NaOH. The fiber that treated

at low concentration of NaOH was inefficient to remove improve the impurities from fiber surface, which resulted the poor bonding of fiber and matrix [9].

3.1.3 Flexural Strength and Flexural Modulus of PLA/Kenaf Biocomposite

Figure 3 shows the measured average flexural strength of three specimens involved pure PLA, untreated and 2, 4, 6 and 8% alkali-treated PLA/KFM composite at different temperature.

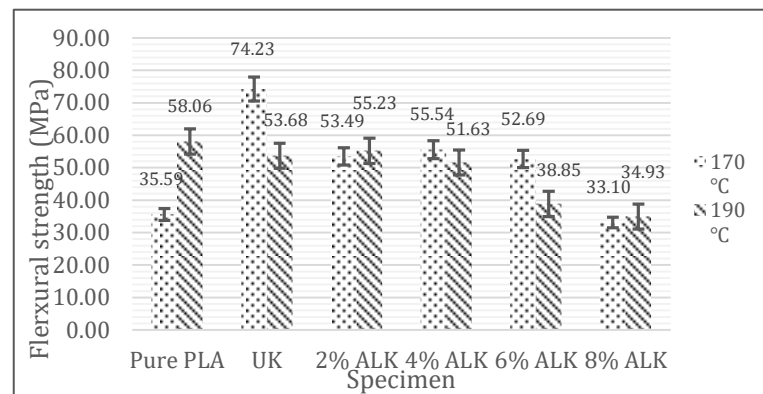


Figure 3. Flexural strength of PLA/KFM composite at different temperature and NaOH concentration.

As illustrated in Figure 3, the PLA/KFM composite processed at 170°C, the flexural strength for alkali-treated composite increased from 53.49 MPa in 2% alkali-treated PLA/KFM composite to 55.54 MPa in 4% alkali-treated PLA/KFM composite, about 3.83% increment was measured. However, the flexural strength has decreased to 52.69 MPa in 6% alkali-treated PLA/KFM composite and continues decreasing to 33.10 MPa in 8% alkali-treated PLA/KFM composite. This could be due to fiber degradation occurred at high concentration of NaOH. From this finding, the 4% alkali-treated PLA/KFM has highest flexural strength among all alkali-treated PLA/KFM composite. However, the flexural strength of alkali-treated PLA/KFM composite is lower than untreated PLA/KFM composite at 170°C. The result indicated that the alkali treatment condition should be further optimized in terms of the immersion temperature and immersion time.

The flexural strength for alkali-treated PLA/KFM composite decreased from 55.23 MPa to 34.93 MPa when the concentration of NaOH increased from 2% to 8% NaOH at 190°C. The 2% NaOH treated PLA/KFM composite showed the highest the highest flexural strength among all alkali-treated PLA/KFM composite at 190°C. The flexural strength of PLA/KFM composite has similar trend as tensile strength of PLA/KFM that processed at 190°C. The flexural strength of 2% NaOH treated PLA/KFM composite was the highest with the value 55.23 MPa compared to untreated PLA/KFM with the value 53.68 MPa. This result was consistent with findings by Ibrahim et al. (2010) [9] that the treated kenaf composite has higher flexural value compared to untreated kenaf composite. The alkali treatment results in fibrillation, breaking down the fiber bundle in finer and increasing the effective surface area for mechanical interlocking thereby promoting more resin interpenetration at the fiber surface [11, 12]. Therefore, the interfacial bonding between is matrix and fiber was improved and leads to increase in flexural strength.

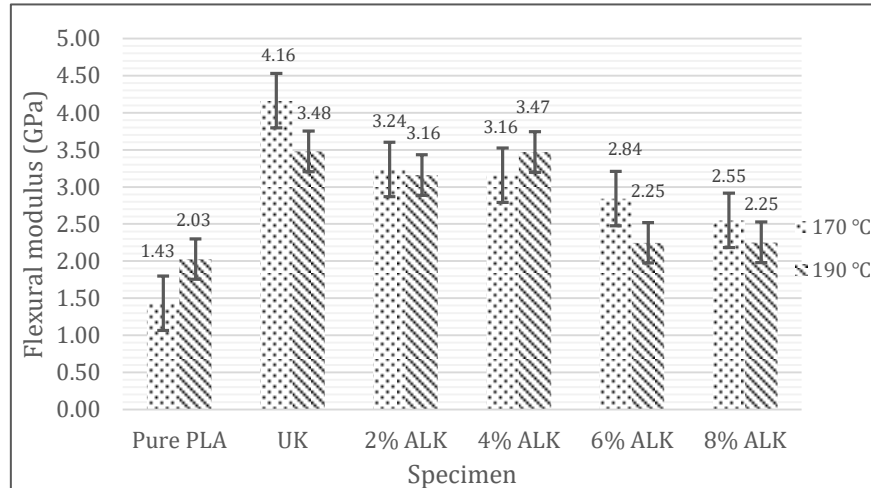


Figure 4. Flexural strength of PLA/KFM composite at different temperature and NaOH concentration.

Figure 4 presents the average flexural modulus of three specimen involved PLA/KFM composite with pure PLA, untreated and 2, 4, 6, 8% alkali-treated PLA/KFM composite at different temperature. For PLA/KFM composite that processed at 170°C, the flexural modulus decreased from untreated PLA/KFM composite (4.16 GPa), 2% alkali-treated PLA/KFM composite (3.24 GPa), 4% alkali-treated PLA/KFM composite (3.16 GPa), 6% alkali-treated PLA/KFM composite (2.84 GPa) and 8% alkali-treated PLA/KFM composite (2.55 GPa). It can be observed that the flexural modulus of PLA/KFM composite decreased linearly when increasing in the concentration of alkaline. In the other words, the stiffness of PLA/KFM composite decreased when increasing in the concentration of alkaline. This could be attributed to the fiber low interaction of the fiber in the matrix. Another possible reason could be due to the presence of void content in the sample has causing has low flexural modulus.

4 Conclusion

In conclusion, the tensile strength of PLA decreased when the processing temperature increased at constant heating time. High processing temperature with short processing time has beneficial effect on the tensile strength of PLA. The optimum temperature and processing time of PLA is 190°C and 10 minutes. It was found that the alkali treatment has the effect on the mechanical properties of PLA/KFM composite. The result showed that 4% of NaOH concentration gave the highest tensile strength among alkali-treated. As a result, the alkali treatment has improved the mechanical properties of PLA/KFM biocomposite.

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