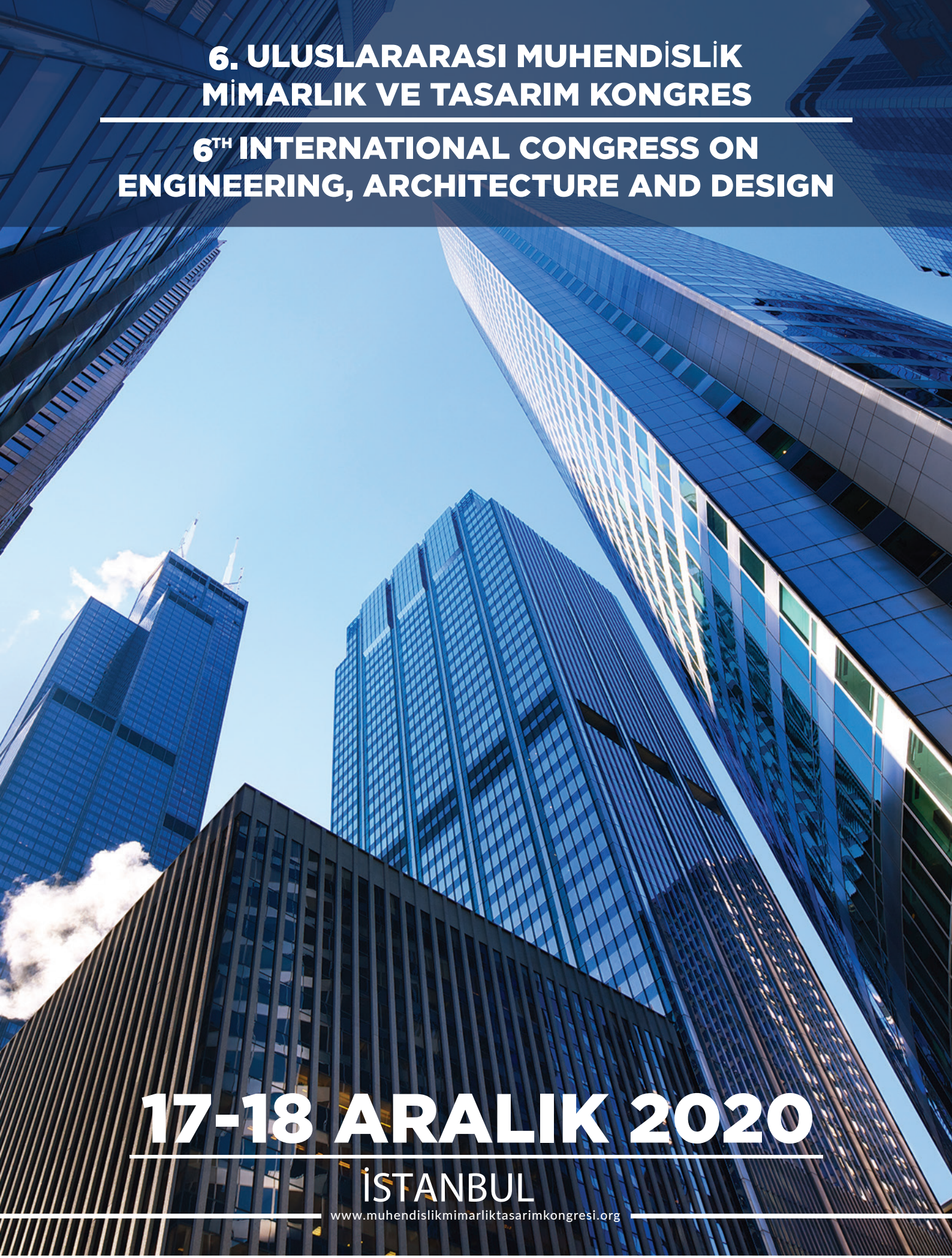
6. ULUSLARARASI MUHENDİSLİK MİMARLIK VE TASARIM KONGRES

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6. ULUSLARARASI MUHENDİSLİK MİMARLIK VE TASARIM KONGRESİ

KONGRE BAŞKANI
Prof. Dr. Hülya KALAYCIOĞLU

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6. ULUSLARARARASI MUHENDİSLİK MİMARLIK VE TASARIM KONGRESİ

TAM METİN SÖZEL SUNUMLAR

INVESTIGATION OF MECHANICAL AND THERMO-MECHANICAL BEHAVIOR OF FLAX FIBER-REINFORCED PLA BIO-COMPOSITES WITH A COUPLING AGENT AND INTERNAL LUBRICANT

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Abstract: A natural-fiber-reinforced bio-based polymer matrix can be considered a sustainable and environmentally harmless source. Natural fibers such as flax, sisal, jute, harakeke, and hemp are bio-based, which makes them fully biodegradable, simply recyclable for detoxification of materials, and appropriate for carbon dioxide footprint purposes. In the current study, short flax fiber-reinforced PLA matrix bio-composite was manufactured using extrusion and injection methods. To enhance the interfacial adhesion between the flax fiber and the PLA matrix, alkali treatment was applied to the flax fiber before the extrusion process. The effect of coupling agent and internal lubricant on the composite materials was investigated concerning the mechanical, and thermomechanical properties of the injected composites. The flexural and tensile moduli of the composites were significantly enhanced compared with those of pure PLA samples. The results show that the compatibilizer has a significant effect on the thermal, thermo-mechanical, and mechanical properties of the composite. Dynamic mechanical thermal analysis and thermogravimetric analysis (TGA) results show that the storage modulus of PLA/flax fiber composites is improved compared with those of both pure PLA and composites with the coupling agent. Compared with PLA, the impact strength of composite increased by more than 2 times; while, the elongation at the break did not suffer an obvious reduction.

Keywords: PLA, Bio-Composite, Mechanical Properties

INTRODUCTION and THEORETICAL FRAMEWORK

Plastic pollution is the accumulation and permeation of petrochemical and non-degradable plastics into the environment. Plastic pollution is widespread throughout the planet ranging from the top of the highest peak to the deep of the oceans[95]. It is assumed that there will be more plastic in the ocean than sea creatures if the plastic pollution rate continues at this speed by 2050. For this reason, the use of eco-friendly polymers that are bio-degradable, lower carbon emission, is increasing day by day[96].

Furthermore, green composites consist of a natural matrix (bio-based polymers) and natural reinforcement. Green composites or bio-composites come from sustainable and biodegradable resources. Synthetic fibers have left their place to natural fibers such as flax, hemp, sisal jute etc. due to natural fiber has decreased greenhouse gas (GHG) emissions, oil dependence, and total cost. Polylactide (PLA) is commonly used as matrix materials. PLA is a biodegradable polymer from renewable resources is an alternative thermoplastic aliphatic polyester polymer to petroleum-based polymers. Moreover, PLA has been widely utilized in academic and industrial applications because of its lower toxicity and high tensile strength, and Young's modulus. However, it has still limitations for more commercial applications due

[95] https://oceanconservancy.org/trash-free-seas/plastics-in-the-ocean/
[96] https://seasave.org/plastic-pollution/

to its brittle structure. Compounding PLA with natural fiber is a very common method to decrease its density. To develop interfacially interactions between matrix-natural fiber, maleic anhydride (MAH) is one of the most preferred compatibilizers. The term lubricant or lubricating agent is often described as certain additives incorporated into plastics materials (Al-Malaika, 2018:46). Materials that reduce the friction of moldings and other finished goods when these are rubbed against the next materials.

PURPOSE

This study aims to investigate the thermal, mechanical, and thermomechanical influence of short flax fiber/ PLA bio-composites manufactured by injection-molding. These composites are manufactured from sustainable sources that use natural flax fiber as reinforcement materials and bio-based PLA as a matrix. Thermal and thermomechanical investigations were carried out using differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and dynamic mechanical thermal analysis (DMA). The mechanical properties were examined through tensile, flexural, and impact testing. This study also aims to investigate the effect of the coupling agent MAH and internal lubricant on the mechanical and thermal properties of the bio-composites. These bio-based composite developed an alternative for automotive components that are manufactured with petroleum-based polymers and synthetic fibers.

SCOPE and METHOD

The selected grade Purapol LX175 Commercial grade Polylactic acid (PLA) was supplied by Total Corbion Company. The PLA possesses a density of 1.24 g/cm^3 and a residual monomer content of <0.3% in pellet form. Coupling agent maleic anhydride (MAH) has a 0.93 g/cm which was supplied by DuPont Company (Fusabond E226, Turkey) in 99% purity. The MAH has about 1.75 g/10 min melt flow rate (MFR). It is melting and boiling temperatures are about 102 °C. The internal lubricant was supplied from the domestic market and it has 0. 98 g cm^{-3} density, 400 °C boiling temperature, and around 390% elongation at breaks. The reinforcement materials were flax fiber. It was supplied from a local market in Turkey with a density is 1.5 g/cm^3.

Preparation of composites

In this study, alkali-treated and untreated flax fibers were added to the PLA matrix with and without MAH. Flax fibers were cleaned with 5% sodium hydroxide (alkali treatment) for 1 h at room temperature to uproot their surface impurities. Alkali treatment also improves adhesion between flax fiber and matrix due to alkali treatment grew up to surface roughness of the fiber as a consequence rough surface provided mechanical interlocking. After alkali cleaning the fibers were washed with distilled water a couple of times to reduce the pH values of the fibers to under 7.



Figure 1. (a) PLA granules; (b) raw flax fiber; (c) maleic anhyride

After the washing process, the flax fibers dried in the sunshine and an oven respectively to remove residual moisture. Composite was mixed using the extrusion process and produced the injection-molding process. Figure 1(a) shows the PLA, Figure 1(b) shows the flax fiber, and Figure 1(c) shows the maleic anhydride granules. Before the all processing step, the components were dehydrated in an oven at 75°C for 3 h again and again. PLA, maleic anhydride, and the internal lubricant were mixed under mechanical stirring for an hour. The components were prepared using a digital balance scale with 1 g precision according to a mass fraction.

Table 1. Compositions of the produced composites

Sample	PLA/MAH/flax/L (wt./wt. %/%)	PLA/MAH/flax/L (vol./vol. %/%)
PLA	100/0/0/0	100/0/0/0
PFL15	85/0/15/0	87.2/0/12.8/0
PFL15MAH/L	79/1/15/5	76.1/1.1/16.6/5.5

Table 1 indicated the composition formulations of the manufactured composites. Then, pre-mixed compounds were fed into a co-rotating twin-screw extruder with an L/D ratio of 30 (Microsan Instrument Inc. Kocaeli - Turkey) 25 mm screw diameter at temperatures of 165–170–180–190°C at 75 revolutions per min (rpm) screw speed. After the extrusion process, the mixed blends were pelletized by using a crusher before being fed for injection-molding. The injection-molding process was run at the temperature of 165–170–175–180°C using an injection molding machine (Hastek Orche BT120, Turkey). Die temperature was selected at 190°C, which was lower than the thermal degradation temperature of the flax fiber. The injection machine ran at a 150 rpm screw speed for melting and at 50 mm s^{-1} injection speed under 100 bar molding pressure. The specimens for, mechanical and thermal tests were used as follows ASTM D 638 type 4 and the ASTM D 790-17.

Mechanical Test

Tensile, flexural, and impact tests were applied to the injected samples to investigate the mechanical properties of the composites. The tensile test was performed to find out the significant mechanical properties of the samples such as tensile strength (TS), Young's modulus YM and elongation at break according to the ASTM D 638 standard. The tensile tests were performed using the Instron testing machine with a 10 KN load cell at a crosshead speed of 1 mm/min. Three-point bending tests were conducted on the samples using a Shimadzu equipped with a 50 KN load cell following the ASTM D790-17 standard. A crosshead speed was calculated from the standard as to be 1.75 mm/ min for a 67 mm gap. Unnotched impact tests were carried out according to BS EN ISO 179-1:2010 standard using a Zwick Izod impact machine with a maximum pendulum ability of 5 J. The sample notch was prepared as 3 mm and the samples were 7 cm long. For all mechanical tests, over five specimens were tested for each formulation to increase the reliability of the test, and the mean values are given in this study with standard deviations. The impact strength (IS) of specimens was obtained from the following equation;

$$A_{cu} = \frac{E_c}{bh} \times 10^3 \qquad \text{Eq. 1}$$

Where, E_c (joules) is the absorbed energy by breaking, b (mm) is the width, and h (mm) is the thickness of the sample.

Thermal Analyses

The thermal properties of the bio-composites were studied by the following analyses which are differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and dynamic mechanical thermal analysis (DMA). DSC analyses were performed with a heating profile of−50 to 220°C at a rate of 10 min^{-1} under a nitrogen atmosphere using (Perkin Elmer, DSC). The DSC analyses were applied to determine significant transition temperatures such as melting temperature (Tm), glass transition temperature (T_g), and to measure the degree of crystallinity (X_c) of the bio-composites. The DSC device was run for about 56 min for each sample. Prior to DSC analysis, the injected samples were cut about 6 to 9 mg in weight and kept in a climatic chamber. The samples were heated at 50 °C then cooled down to -50°C and held for 1 minute. After then, samples were reheated to 250 °C before cool down to 30 °C they were held for 1 min at 220°C. The crystallinities rate (X_c) of PLA and bio-composites were calculated by the first heating cycle using equation 2.

$$X_{(\%\ crystallinity)} = \frac{(\Delta_{Hm} - \Delta_{Hc})}{(W_{PLA} \times \Delta_{Hm}^{\circ})} 10^2 \qquad \textbf{Eq. 2}$$

where Δ_{Hc} is the crystallization enthalpy, W_{PLA} is the weight fraction of pure PLA, Δ_{Hm} is the heat of fusion of each sample, for 100 % crystalline PLA heat of fusion of PLA (ΔHm) is 93.6 j/g (Gamon, 2013:26), (Tuominen , 2002:22).

Thermal gravimetric analyses were conducted by using SII Nanotechnology Exstar TG/DTA 6300 device to investigate thermal stability and thermal degradation temperatures of PLA and the bio-composites. A test took about 52 min at a heating rate of 10°C min^{-1} under a nitrogen atmosphere. TGA analyses were applied on the samples at a temperature between 25°C to 550 °C.

DMA analyses were performed on the specimens to investigate the storage modulus (E'), loss modulus (E"), and damping peak (tan δ) using a tension mode at a frequency of 1 Hz using a DMA Perkin Elmer – DMA 8000 tension device. The temperature range was conducted between −50 and 150 °C at a heating rate of 5°C min^{-1}. The DMA specimens were cut using a precision cutting machine with dimensions of 10 mm x 10 mm x 4.2 mm. Melt flow index (MFI) measurements were applied using (Cometectch Company Taiwan) an MFI device at 190 °C under 2.16 kg load according to ASTM D1238 for pure PLA and the bio-composites.

FINDINGS

Melt flow index (MFI) value is a significant feature of polymer and polymer-based composite for processability during extrusion and injection molding.

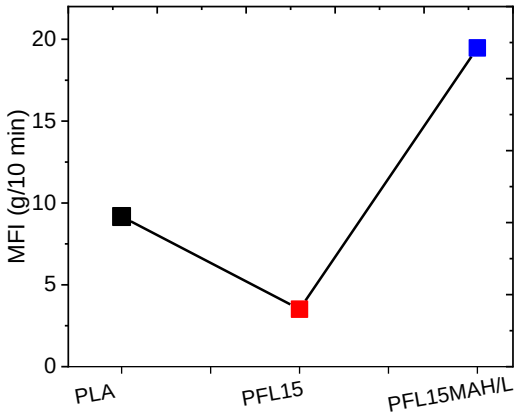


Figure 2. Melt flow index of pure PLA and bio-composites

Figure 2 shows the MFI values of the specimens. It is apparent from the results, the addition of the internal lubricant to the PFL bio-composite enhanced the MFI values from 3.5 to 20 g/10 min.During the extrusion and injection molding process the addition of flax fiber into the PLA led to a decrease in the fluidity of the mixture. In order to improve the fluidity and processability of the mixture, the process temperature increased up to 190 °C. However, an increase in process temperature led to burn/damage of the flax fiber. For this reason 5% wt. internal lubricant added into the mixture of the composite to improve the fluidity.

Effects of internal lubricant and MAH on the TS, YM, FS, ductility and, IS of the bio-composites and PLA were studied using tensile, three-point bending, and impact tests. Tensile, three-point bending, and impact test results are showed in Figure 3. Table 2 illustrated the mechanical properties of the prepared bio-composites and PLA. It is clear that YM values increase with the addition of flax fiber into pure PLA. For the PLA, PFL15, and PFL15MAH/L specimens, the YM values were calculated as 5.13, 5.81, and 5.33 GPa, respectively. FM values of the samples were calculated as 2.3, 2.9, and 2.3 GPa, respectively. The addition of flax fiber into the PLA matrix promoted an increase by about 26% when compared to pure PLA. However, the FM value dropped to 2.3 GPa with the addition of the MAH and internal lubricant.

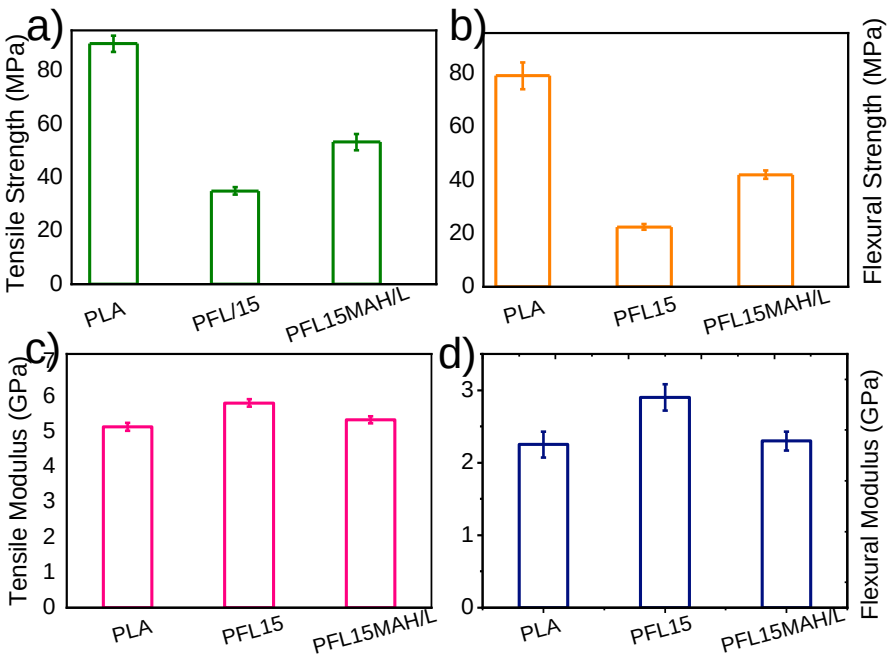


Figure 3. Tensile and flexural properties of PLA and bio-composite; (a) Tensile strength, (b) flexural strength, (c) tensile modulus, (d) flexural modulus

Compared to the PFL15 bio-composite, the strength of PFL15MAH/L is increased from 35 MPa to 53 MPa. This improvement is associated with the good interface of the PLA matrix and flax fiber, being a significant feature for the load to be transferred. A similar characteristic was observed in the FS results, which means that the incorporation of the flax fiber into PLA, the use of the internal lubricant, and MAH cause a notable increment by about 50%. It is important to increase the interfacial adhesion quality because it directly affects to load transfer capacity of the composite from matrix to fiber (Georgiopoulos, 2018: 02).The addition of internal lubricant provided a good kneading of the composite mixture during the extrusion and injection process as a consequence, it promotes well interfacial adhesion between the PLA matrix and flax fiber. The coupling agent also improves interfacial adhesion between fiber and matrix since the MAH causes to increase in the number of chemical bonds in the fiber-matrix interface result in, fibers are well trapped within the matrix (Avci, 2020:62). Alkali treated bio-composite shows good interfacial adherence between the matrix and alkali-treated fibers since alkali-treated fiber has a rougher surface than untreated fiber. The rough surface of the former forms more contacting points to occur mechanical interlocking adhesion between the matrix and the fibers.

Table 2. Mechanical properties of PLA and bio-composite

Samples	Tensile Modulus (GPa)	Flexural Modulus (GPa)	$\sigma_{tensile}$	$\sigma_{flexural}$	$\%\varepsilon_t$	$\%\varepsilon_f$	Impact strength (kJ/m^2)
PLA	5.13±0.11	2.3 ± 0.18	90.1±3	79.03±5	10.2±1.9	5.43±1	15.9±1
PFL15	5.81±0.10	2.9±0.18	34.9±1.4	22.43±1	2.21±0.1	0.87±0.07	7.79±0.14
PFL15MAH/L	5.33±0.1	2.3±0.13	53.2±3	41.9±1.6	5.08±1.7	2.3±0.7	10.75±0.1

Figure 4 shows the results of the Izod impact test. It is apparent that that the impact strength of the bio-composite without additives increases with the addition of the MAH and the internal lubricant. It means that the addition of internal lubricant and MAH in the composite is critical for the realization of high IS value. These additives also vitally affect the elongation at breaks values of bio-composite. When comparing PFL15 and FL15MAH/L samples, it can be seen that the tensile and flexural elongation at break values are increase by about 50% and 200%, respectively.

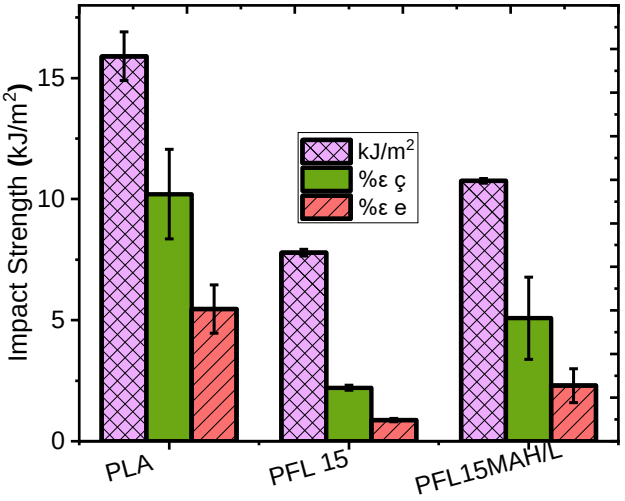


Figure 4. Impact strength and elongation at break of PLA and bio-composites

However, the highest elongation at break value is still lower than pure PLA. This reduction can be explained by process parameters need to optimize both extrusion and injection process. There is one more possible reason that bio-composites might have a poor orientation of the fibers in the matrix. Deboning, fracture, and pull-out of the fibers are the main mechanism of energy absorption under dynamic loads. If there is poor adhesion between matrix-fiber leads to a higher energy absorption thus, IS values of bio-composites could decrease (Bax ,2008:04).

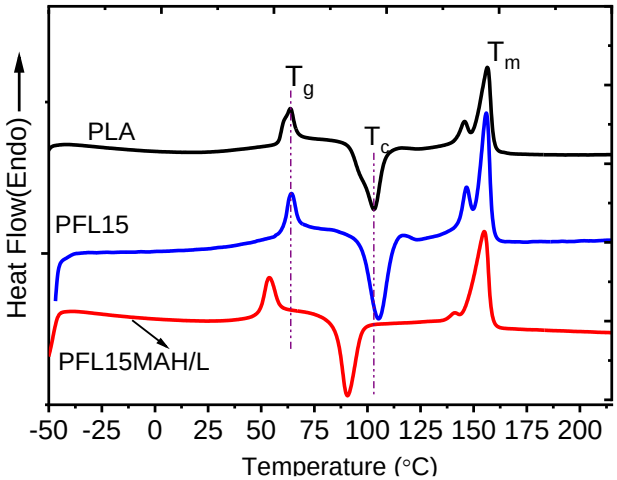


Figure 5. DSC heating thermographs of PLA and bio-composites

DSC analyses were performed to determine the crystallization behavior of the PLA and bio-composites since the mechanical properties (impact, tensile and flexural strength) of the polymers depend on the

crystallization rate. Figure 5 shows DSC curves of the specimens. Differential scanning calorimetry (DSC) was also utilized to find out transition temperatures which are melting temperature (T_m), glass transition temperature (T_g), and cold crystallization temperature (T_c) of the specimens.

Table 3. DSC parameters of PLA and the compounds

Samples	T_g (˚C)	T_c (˚C)	T_m (˚C)	ΔH_m (j/g)	ΔH_c (j/g)	X_c (%)
PLA	62,4	106,3	155,06	24,9	9,3	15,9
PFL15	63,8	102,3	156,4	22,4	11,4	13,9
PFL15MAH/L	53,81	90,6	154,96	18,77	15,6	6,8

As shown, in Table 3, the glass temperature of the PFL15 is 63.8 °C which is slightly more than pure PLA (62.4 °C). The addition of internal lubricant and coupling agent into the bio-composite shows a decrease by about 10 °C compared to PFL15. The crystallinity slightly decreased with the addition of flax fiber into the PLA at about 2%. The addition of internal lubricant accelerates the reduction by about 9% compared to pure PLA. Moreover, the cold crystallization temperature decrease with the addition of flax fiber due to cellulose fibers act as efficient nucleating agents for the crystallization of PLA.

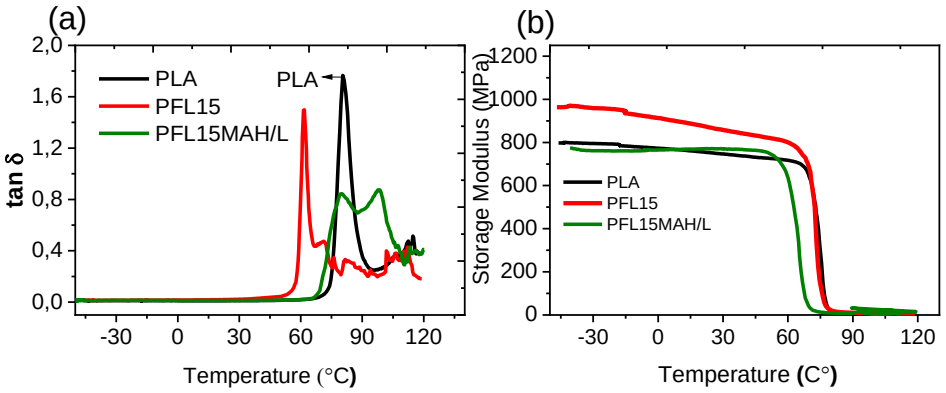


Figure 6. (a) Loss factor (tan d) values of pure PLA and bio-composites; (b) storage modulus values of PLA and PLA/flax composites

Figure 6 shows the storage modulus and damping factor of pure PLA and bio-composites as a function of temperature. The addition of the internal lubricant and coupling agent significantly affects the dynamic mechanical properties of the bio-composite. While PLA has a 770 MPa storage modulus at around 25 °C, the FPL15 has a 900 MPa storage modulus. The addition of MAH and internal lubricant leads to a reduction of about 140 MPa in the storage modulus. While PLA has the highest damping factor of about 1.75, this drops to values of between 0.8 and 1.4 for the composites. According to the DMA results, although the glass transition temperature of PLA was affected by the addition of flax fiber. The damping factor was also reduced in the PFL15 and PFL15MAH/L.

Figure 7 shows the Thermogravimetric curves of PLA and composites. Compared to pure PLA, the degradation temperature of bio-composite decreases by about 40 °C due to flax fiber has a lower thermal degradation temperature at about 310°C. For pure PLA, PFL, and PFL15MAH/L thermal degradation temperatures were measured as 364, 310, and 320°C, respectively. It means that compared to pure PLA the thermal degradation temperature of the bio-composite starts earlier.

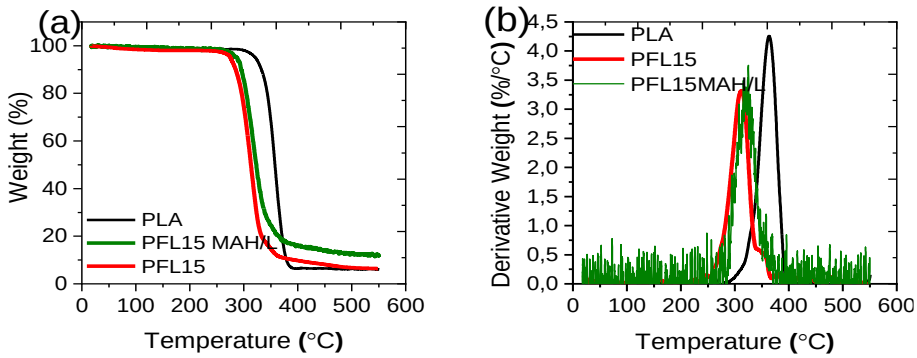


Figure 7. (a) Thermogravimetric curves, (b) DTG curves of PLA and bio-composites

The mass residues at 550°C are measured as 6.00, 6.38, and 12.15% for PLA, PFL15, and PFL15MAH/L, respectively. This can be explained as being because, at high temperatures, flax fiber composites show better thermal stability than pure PLA.

CONCLUSION

In this study PLA short flax fiber bio-composites with 15% wt. flax fiber content was manufactured by extrusion and injection molding process, respectively. The current study is aimed at investigating the thermal, mechanical influence of internal lubricant and MAH on the mechanical, and thermal properties of bio-composite and pure PLA. The bio-composite made of PLA and flax fibers exhibit promising mechanical properties, in particular stiffness of the PLA which are YM and FM values. Owing to their mechanical and thermal properties, possible fields of application could be the automotive and construction industry. However, the addition of an internal lubricant rate should be optimized to improve some mechanical properties. Moreover, the flammability properties of this bio-composite should be improved to use in the automotive industrial applications. Tensile and flexural elongation at break of the composite with the internal lubricant and MAH exhibited a notable improvement of about 50 and 200%, respectively. The addition of internal lubricant and MAH also improved TS and FS values of the bio-composite. Whereas the addition of flax fiber into PLA caused reductions in TS and FS values. The addition of internal lubricant and MAH into PLA led to reduction TGA and DMA values and T_g transition temperature of the composites were affected favorably.

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