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Investigations into the tensile properties and microstructural features of Coconut fibre (Coir) reinforced Polylactic acid (PLA) biodegradable composites

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Abstract
The study investigated the tensile properties of coconut fibre reinforced Polylactic acid biopolymer. Unidirectional arrays of coir fibres with an average diameter of 0.242 mm and a sample size of about 83 fibres per layer were used to manufacture PLA biodegradable composites by a film stacking technique via hot press. The type A composite was a blend of PLA/PLA/Coir/PLA/PLA while Type B composite was a blend of PLA/PLA/Coir/Coir/PLA/PLA. The manufactured composites were subjected to Instron universal testing machine to characterize their mechanical properties. The tensile strength for composites type A was found to be 11% higher than the tensile strength of composite Type B. However, the Young's modulus for composite type B was 20% higher than the Young's modulus for composite type A. Generally, the values of the tensile properties of the coconut fibre reinforced PLA composite was about 40% higher than the values found in literature for coir composites with matrices other than PLA within the studied range. Their microstructures were observed using scanning electron and optical microscopy, the microstructures of the composite samples show the arrangements of the coir fibres within the PLA matrix. Image analysis of the optical micrographs of the components on an 8-bit grey scale revealed volume fractions of fibres within the layers in Type A and Type B as 33% and 24% respectively. The images of the manufactured composites obtained from scanning electron microscope showed some level of fibre pull-out from the matrix..

Keywords: PLA, composites; coir; tensile properties; volume fraction

1. Introduction

Summarily, composites are used to address certain inadequacies of single materials thereby enhancing their performance levels; this gives room for the development of new and advanced products. The use of polymer as matrix encourages the introduction of different low cost reinforcing fibres thereby reducing the overall cost of production and encourages environmental safety. Natural fibres such as sisal, jute, flax, bamboo and coir have been receiving tremendous attention as reinforcements in composites(Yan *et al.*, 2015; I.Singh *et al.*, 2017; Bai *et al.*, 2021; Madueke, Umunakwe and Mbah, 2022; Richely, Nuez and Biopolym, 2022). Coir is a lignocellulosic fibre that has been characterized by several researchers (Ihueze, Achike and Okafor, 2016; Emeka *et al.*, 2020; Madueke, 2021). Coconut fibre possesses desirable properties such as low thermal conductivity(Veeraprabahar *et al.*, 2022) and hence they are applied in automotive thermal insulation. Coir has good acoustic properties (Muralidharan, 2022) as a result of its porosity, low density(Madueke, Umunakwe and Mbah, 2022) hence they are used in lightweight composites applications. Coir has found application as an eco-friendly inhibitor pigment for organic coatings(Freitas *et al.*, 2022). Other applications of coir are as a replacement for car window regulator handle (Okafor *et al* 2021) and micro-perforated panel(Mullins, 2012). Coconut from where coir fibres are extracted grows extensively in countries such as Indonesia, Sri Lanka, Thailand and some parts of Africa including Nigeria where coir fibres are regarded as waste.

PLA is non-petroleum-based polymer derived from natural resources such as rice, sugar beets and corn(Kamarudin and Abdullah, 2018). The strength, stiffness and elongation of PLA are sufficient in their areas of application such as milk containers, food packaging, medical uses and automotive (Bansal, Ramachandran and Raichurkar, 2017). However, PLA shows some degree of brittleness and low crystallization speed which(Thakur, Thakur and Gupta, 2014) limit its applications. Poor interfacial bond strength has been reported to exist between PLA polymer and natural fibres as a result of their incompatibility. Processing technique employed in the composite manufacture and fibre orientation could lead to improved tensile properties and an overall mechanical properties and hence better commercialization outcome(Hasan, K M; Horvath, PG; Bak, M; Alpar, 2021; Agwu and Ozoegwu, 2022).

Fibre-reinforced PLA composites are being developed and investigated to reduce the use of synthetic materials. PLA/natural fibre composites that have been manufactured and investigated include; PLA/kenaf (Kamarudin and Abdullah, 2018), PLA/jute (Sanivada *et al.*, 2020). Flax fabric has also been used with PLA via hot press to manufacture PLA/Flax composite(Nassiopoulos and Njuguna, 2015), and PLA/Coir (Dong *et al.*, 2014). However the tensile properties were not characterised. Increase in crystallinity index and thermal stability have been reported on treated coir reinforced PLA composites(Monroy *et al,*. 2023),). For coir/epoxy composites prepared with the fibres randomly oriented; the tensile strengths obtained have been in the range of 17.31-20.2 MPa(Yan *et al.*, 2016; Hemanth *et al.*, 2017; Cesar *et al.*, 2018). For coir/polyester manufactured via compounding, a low tensile strength of 13.05MPa has been recorded(Naveen.P; Yasaswi, 2013) and with coir/PP, a tensile strength of 15.02MPa (Ichim *et al.*, 2022) was observed, for the manufactured coconut fibre reinforced high density polyethylene composites, the tensile strength obtained was 26.6MPa with 20% coir volume fraction and a Young's modulus of 0.65GPa (Ihueze, Achike and Okafor, 2016). For enhanced applications of coir fibre composites, a significant improvement in the tensile strength is imperative hence a diversion in the matrix, fibre volume fraction, the arrangement of the fibres in the matrix and the manufacturing techniques are necessary. Moreover, not much work has been reported on the tensile properties of coir/PLA composites with fibres unidirectionally oriented and stacked in layers and composites manufactured using hot press compaction, hence this research work.

2.0 Material and methods

2.1 Materials

Matrix: Polylactic-acid (PLA) film of length 400 mm, width 300 mm, weight 3.76 g and thickness of 0.025 mm was supplied by Vegware Ltd UK. PLA film was dried at 80^{o}C for 6 hours and stored in a desiccator prior to use. The dried PLA film was cut in double layers of dimensions; 20 mm x 120 mm.

Reinforcement: As-received coir fibres are as shown in Figure 1. The average diameter of the selected fibres was 0.242 +/- 0.08 mm. The coir fibres were washed three times in warm water (50°C) for 15 minutes per wash and dried at room temperature for two days. These coir fibres were unidirectionally-aligned on a polytetrafluoroethylene (PTFE)-coated metal plate on which thin strips of adhesive were placed at the opposite ends to form single layers of 20 mm width by 130 mm length samples.

Figure 1: Selected coir fibres

2.2 Method

Combined geometry and orientation of the fibres exert significant influence on the performance of fibre-reinforced composites(Agwu and Ozoegwu, 2022).Tensile properties of composites containing longitudinally-oriented closely packed fibres have been proved to be higher than those with fibres transversely-oriented, as close packing leads to increase in the density of material and hence the secondary intermolecular forces and strength are subsequently increased(Summerscales and Grove, 2014; Manalo *et al.*, 2015). Fibres are better and evenly distributed in the matrix and fibre damage is grossly reduced when they are stacked in layers (Madueke, 2021). Given the degradation temperature of cellulose (natural fibres) to be approximately 200 ^oC, it is imperative to consider polymer matrix that can accommodate temperature of less than 200 ^oC. PLA has a glass transition temperature (Tg) of 55-65^oC and melting temperature (Tm) of 120-170^oC. Therefore, the choice of using the following technique (including the processing parameters) to manufacture PLA/coir composites are based on these factors.

Double layers of the PLA film with the specified dimensions were placed on the steel plate covered with PTFE. The aligned single coir fibres were placed over the PLA, second double layers of PLA film was placed over the coir fibre layer and covered with PTFE sheet to form the Type A sandwich structure of PLA/PLA/Coir/PLA/PLA. The layout is as shown in Figure 2. The set up was then introduced onto a conventional hydraulic press (Moore Max) with a maximum press capacity of 25 tons. The degradation temperature of natural fibres is about 200 ^oC, therefore the compaction temperature must not exceed 200^oC. The required temperature of 165^oC was set for the upper and lower platens. When the set temperature was attained, a pressure of 2 MPa was applied for 5 minutes. The power was turned off on attaining the set time and the pressure was maintained as the platens cooled to about 45^oC. The pressure was then released and the steel plate was removed and left to cool to room temperature for 1 day. The Type B was prepared in similar way but with double layers of coir fibre to form PLA/PLA/Coir/Coir/PLA/PLA. All specimens were manufactured using hot compaction as illustrated in Figure 3. The processing parameters of the hot press for the two samples are the same; Temperature of 165 ^oC, pressure of 2MPa and hot-pressing time of 5 minutes.

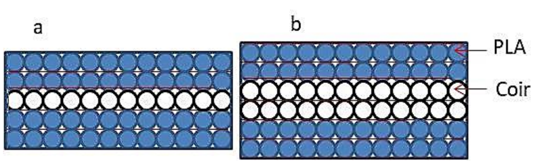

Figure 2 Layout of (a) PLA/PLA/Coir/PLA/PLA (b) PLA/PLA/Coir/Coir/PLA/PLA

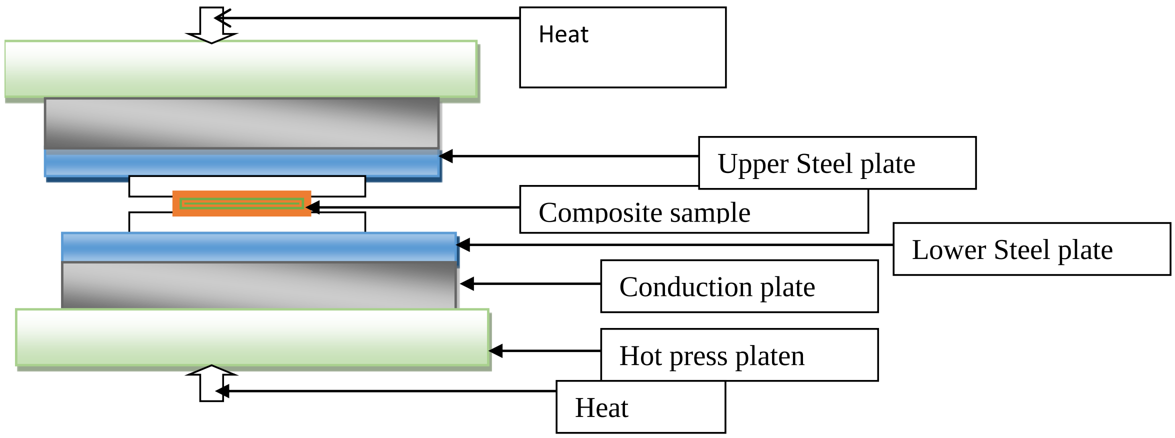

Figure 3 Schematic illustration of the composite sample within the hot press

2.3 Tensile Tests

Tensile testing of the composites were carried out on Instron 5566 universal testing machine with the software recordings at a cross-head speed of 1 mm/minute in accordance with ASTM D638. The relative humidity and the temperature of the testing environment were 45% and 24^{o}C respectively. The Young's modulus was calculated from the initial linear portion of the slope of the stress-strain curve.

2.4 **Scanning electron microscopy and optical microscopy**

The microstructure was characterized using Hitachi S4700 Table top Scanning Electron Microscope (SEM) 3030 and Zeiss Axioskop 2 HAL 100 optical microscopy model. Samples were sectioned and potted. The potted samples were grinded and polished using standard metallographic technique in order to obtain crisp images for optical microscopy. Sectioned samples for scanning electron microscopy were sputter-coated with gold. The sputter-coated samples were taken to the table top SEM to observe the microstructure.

3. Results and discussions

3.1 Tensile tests

The stress strain curves of the composites samples are shown in Figure 4. The tensile properties of the composites are shown in Table 1 and displayed in Figures 5 and 6. The average tensile strength, Young's modulus and elongation at break obtained with single layer of coir in PLA are 49.73 ± 5.26 MPa, 10.56 ± 0.92 GPa and 1.64 ± 0.00% respectively while those obtained with double layers of coir in PLA are 44.38 ± 3.67 MPA, 11.36 ± 1.06 GPa and 1.55 ± 0.09% respectively. Improved tensile properties and an increase in the biodegradability of the composites have been recorded when PLA was reinforced with coir fibre using hydraulic press at a pressure of 1.5MPa for 15 minutes at room temperature, however a decrease in the glass transition temperature was observed(Dong *et al.*, 2014). The mechanical properties of composites produced by hot compaction depends on the compaction temperature and other constituent materials used in the composite(Foster *et al.*, 2016). Higher compaction temperature leads to better bonding between the layers, as a result of the higher cohesive strength of the matrix, however when the compaction temperature exceeds the optimum, a decrease in the tensile strength can be observed. The tensile strength obtained from literature for epoxy reinforce coir fibres with fibres in random orientation are in the range of 17.31 to 20.2MPa (Yan *et al.*, 2016; Hemanth *et al.*, 2017; Cesar *et al.*, 2018). The tensile strength of PP/coir obtained from literature was 15.02 MPa (Ichim *et al.*, 2022), while that of Coir/PU via HLU was 4.13MPa (Faria *et al.*, 2023), the higher tensile strength in the current study can therefore be attributed to the matrix used, fibre orientation, manufacturing techniques and fibre volume fraction. PLA/sisal composite manufactured using hot press via stacking at compaction temperature of 180^{o}C, 4MPa and for 8 minutes showed improved wear behaviour and low coefficient of friction due to the incorporation of the fibre and the fibre orientation(Kumar, Singh and Madaan, 2013). The interfacial bond strength between the PLA matrix and the coir fibre can be improved via fibre treatment (Amroune, Belaadi and Mohamad, 2018)(Ghori, Rao and Rajhi, 2023) to further improve the mechanical properties of the composites.

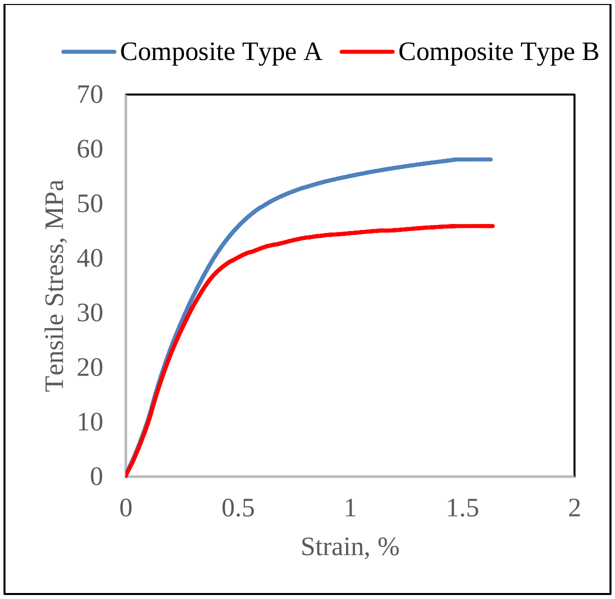

Figure 4: Stress/strain curves of the composites samples

Table 1: Tensile properties of the manufactured composites

	Parameter		
Composite type	Tensile Strength (MPa)	Young's modulus (GPa)	Elongation at break (%)
A	49.73 ±5.26	9.04 ± 0.92	1.63 ±0.00
B	44.38 ±3.67	11.36±1.06	1.55 ±0.09

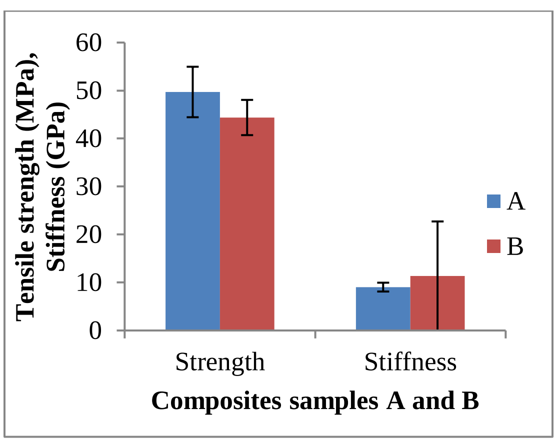

Figure 5: Tensile strength and Young's modulus of the manufactured composites

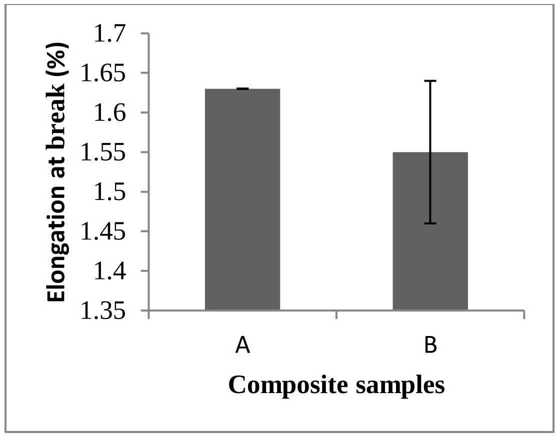

Figure 6: Elongation at break of the manufactured composites

3.2 Microstructure

The micrographs of the as-received coir fibres are shown in Figure 7 (a and b) showing the multicellular nature of coir and the presence of tyloses and lumen in the microstructure as previously observed(Tran *et al.*, 2015). The micrographs of the composites are shown in Figures 8 and 9 repectively. Figure 10 shows fractured PLA/coir surfaces while Figure 11 is the SEM image depicting the pull out of coir fibre from PLA matrix, similar observation has been made by (Li Zhang, Zhihui Sun, Duoping Liang, Jing Lyn, 2017). This pull out can be effectively reduced via fibre treatment such as the use of silane and alkali as this will enhance the bonding of coir to PLA matrix (Asim *et al.*, 2016), hence narrowing the gap between PLA and coir within the composite. The optical images of the polished samples was obtained using Zeiss Axioskop-2 microscope. These images were used to determine the volume fraction of the fibres in the composites samples. ImageJ software was adopted to analyse the micrographs. Each sample was analysed for five times and the average taken. The average volume fracton of the fibres for single and double coir layers in PLA were obtained. The Type A and Type B composites showed fibre volume fractions of 33% and 24% respectively, this can be attributed to the closer contacts of the fibres as shown in Figure 8. The reduction in the volume fraction of the double layer can be attributed to the gaps inbetween the fibres and inbetween the layers. Coir fibre volume fractions of 25, 30 and 22% have been used for Polypropylen, Polyethylene and epoxy matrix respectively(Yan *et al.*, 2016; Cesar *et al.*, 2018; Ichim *et al.*, 2022). The volume fraction of the fibre has a maximum limit above which yields a negative effects on the composites(Raghavan).

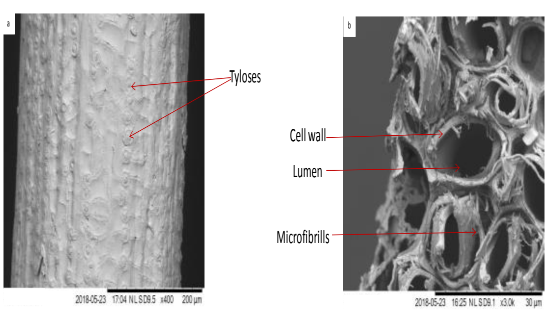

Figure 7: (a) longitudinal and (b) cross section of coir fibre

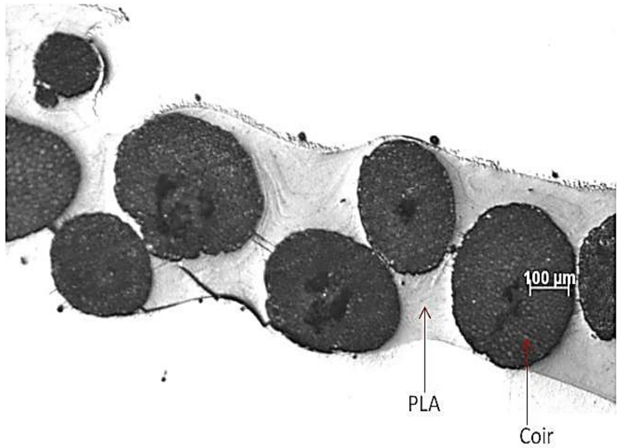

Figure 8: Optical micrograph of the cross-section of PLA/PLA/Coir/Coir/PLA/PLA

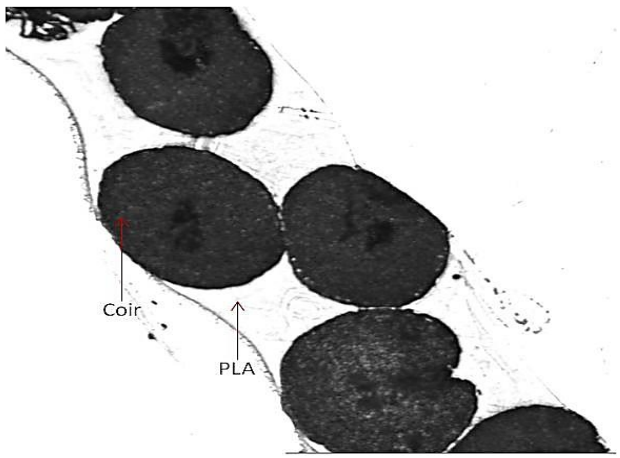

Figure 9: Optical micrograph of the cross-section of PLA/PLA/Coir/PLA/PLA

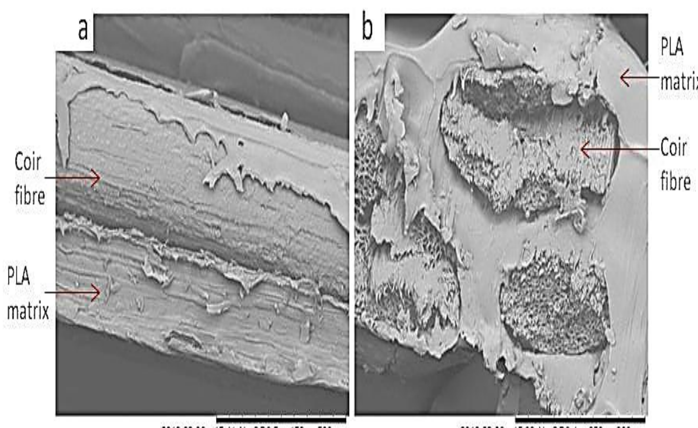

Figure 10: SEM images of the PLA/coir (a) surfaces (b) cross-section

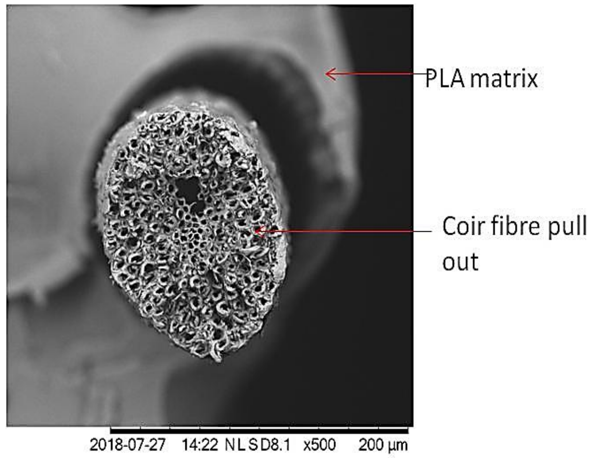

Figure 11: SEM image showing coir fibre pull out from the PLA

4. Conclusions

Reinforcing PLA with unidirectionally-oriented coir fibre has led to an improvement in the mechanical properties such as the tensile properties of the composites. The tensile strength and elongation at break of single layer coir/PLA composites (Type A) were found to be higher than those of double layer coir/PLA composites (Type B) by 11%..The stiffness of the double layer coir/PLA composites (Type B) was found to be 20% higher than that of single layer coir/PLA composites (Type A). The tensile properties obtained are way higher than the tensile properties of coir composites of matrices other than PLA obtained from literature within the studied range. Higher and improved mechanical properties can further be achieved when coir is chemically treated with alkali such as NaOH and silane coupling agent. These treatments will lead to better bonding between the fibre and the matrix and improved mechanical interlocking of the fibre and the matrix. Secondly, from the results obtained, the mechanical properties of PLA/coir can be made to meet specific applications with a wide range of compaction temperature and stacking sequence. Therefore future work will vary the temperature, pressure and stacking sequence and assess their influences on the mechanical properties of the composites.

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