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Effect of Fiber Treatment on Mechanical and Physical Properties of Sugar Palm Fiber-Reinforced PLA 3D-Printed Parts

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ABSTRACT

The existence of impurities and other chemical substances on the surface of fibers becomes the barrier in binding the fibers with the polymer matrix. Therefore, the objective of this study is to determine the appropriate fiber treatment of sugar palm fiber (SPF) composite through the investigation of the mechanical and physical properties of the 3D-printed SPF composite parts. This study was carried out by using four different types of fiber which are untreated, silane-treated, alkaline-treated, and combination of alkaline and silane-treated fibers for SPF composites. All the samples undergo tensile and flexural testing and observe under scanning electron microscope to evaluate fiber-matrix adhesion performance. Consequently, the combined treatment for the SPF composite obtained the highest tensile strength and flexural strength that were 22.82 MPa and 28.44 MPa respectively. In physical testing, the composites exhibited improvement in the hydrophobic properties of the SPF composite. The SEM micrograph shows that better interfacial adhesion between the fiber with combination treatment of alkaline and silane and the polylactic acid (PLA) matrix which the composite fracture surface shows fewer voids Based on these results, the combined- alkaline and silane-treated fiber is recommended as fiber preparation method for achieving superior 3D printed SPF composite.

Keywords: Fiber treatment; mechanical properties; physical properties; sugar palm composite; 3D printing

INTRODUCTION

The technology of fused deposition modeling (FDM) is continually evolving and raising much interest among researchers, especially in advanced materials and manufacturing engineering. The advantages of FDM drive the enhancement of this technology in all aspects of the process. The flexibility of FDM depositing materials through the nozzle in three dimensions has allowed as many geometrical shapes as possible and designs to be fabricated through the machine of a 3D printer. Generally, thermoplastic materials are being used as a feedstock of the FDM process as this material has a reversible characteristic where they can be heated and cooled repeatedly. Moreover, thermoplastics have a glass transition temperature which become soft and pliable. Regarding these, it is easy to remold or reshape which is an essential characteristic of FDM feedstock (Arrgo & Frache 2022). To add more value to the thermoplastic filament of FDM, researchers use natural fibers as reinforcing materials of thermoplastic polymer composite and as an element of environmentally friendly.

Natural fibers come in a variety of forms, and in contrast to synthetic fibers, are sourced from plants. Examples of these include flax, hemp, coconut coir and oil palm fiber. There are examples of the bio-composite filament of FDM that have been developed by past researchers such as kenaf/PLA (Haryati et al. (2021), wood/PLA (Yu et al. (2022), and rice straw/PLA (Yu et al. (2021) . These fibers are also renewable, as they can be grown and harvested repeatedly. The advantage of natural fibers is low in density, which can be an advantage in applications where weight reduction is important, such as in the

automotive (Shaharuzaman et al. 2019) and aerospace (Arockiam et al. 2018) industries. These fibers are composed of cellulose and have good strength (Deb & Jafferson 2021) and stiffness (Montalvo Navarrete et al. 2018), which increases the potential to be applied as reinforcement in polymer composites. A wide range of characteristics of natural fibers are caused by the influence of the ground soil, the climate of the area, and the sources of the plants. The composition of natural fibers exhibits their mechanical and physical properties (Ahmad & Zhou 2022). The main constituent of the natural fibers' cell wall is cellulose, and it is the primary source of strength and stiffness of the fiber. The thickness of the secondary layer in the cell wall is mainly attributed to fiber tensile properties. The secondary walls of closely aligned elementary cellulose fibrils form microfibrils, and the microfibrils collectively form a single fiber. Depending on the strength and characteristics of microfibrils, the deformation in the stress-strain curve of a single plant fiber is derived from different stem sections. Therefore, the potential of natural fiber as reinforced materials for polymer filament of FDM has attracted researchers to explore alternative materials for thermoplastic FDM that are more sustainable and reliable. Further investigation is required if the new fiber is going to be employed as filament of FDM. The potential of the new local fiber such as sugar palm fiber to be compounded with polylactic acid (PLA) or polypropylene (PP) or polystyrene (PS) is not new in natural fiber composite. However, the employment of this type of composite in additive manufacturing, specifically FDM is new and creates several research questions that need to be answered. The mechanical and physical properties of the composite need to be determined since the material is going through a repeated thermal extrusion process and the performance of this new composite in terms of the mechanical properties need to be measured.

The FDM process typically involves designing a 3D model using Computer-Aided Design (CAD) software, and then slicing the model into thin cross-sectional layers using slicing software. The 3D printer heats a spool of thermoplastic filament, which is fed into a heated extruder. The extruder melts the filament and deposits it onto a build platform layer by layer, following the path specified by the sliced model. Once the printing is completed, the 3D object is cooled and can be removed from the build platform(Arrgo & Frache, 2022). One of the advantages of FDM technology is that it allows for the creation of complex shapes and intricate geometries that might be difficult or impossible to achieve using other manufacturing methods. FDM also supports a wide range of thermoplastic materials, including ABS, PLA, PETG, and nylon, among others (Bourell et al. 2017). However, FDM printing can result in visible layer lines and surface imperfections, which may require additional post-processing steps such as sanding or painting to achieve a smooth finish. The employment of bio-composite materials as feedstock of FDM has been introduced previously. The printability of the bio-composite filament has been explored by many researchers. The definition of printability is widely understood by researchers in various focus areas including the mechanical behavior of the printed parts. The mechanical behavior of the printed parts includes the tensile, flexural and impact properties. A study done by Liu et al. (2019) showed that through the mechanical testing of pine wood floor/PLA printed part, the tensile strength was 20.7 MPa and improved the pure PLA tensile properties by 74%. On the other hand, Agaliotis et al. (2022) found that the presence of raw fibers in a polymer-based filament of FDM exhibited lower tensile properties as compared with pure PLA 3D printed part. They also agreed that improper preparation of the composite filament implies the lower tensile properties of the 3D printed composite part as particle agglomeration and voids caused degradation in material stiffness. However, Haryati et al. (2021) found that the kenaf/PLA composite printed part has exhibited a slight increase in Young's Modulus with 3wt.% kenaf fiber. Further investigation on the bio-composite printed parts has been conducted by Le Duigou et al. (2020) for its morphological properties. Generally, the excellent tensile strength and modulus of composite materials exhibit good interfacial bonding strength that can be seen through the scanning electron microscope. Challenges in fabricating the composites are getting the optimum composition of the constituents and obtaining good interfacial bonding strength with less fiber pull-out and voids.

In this study, an eco-friendly filament of FDM was fabricated and the mechanical and physical properties of this new material were investigated. To the author's knowledge, no study on the sugar palm fiber/PLA composite fabricated for the filament of FDM. The fabrication process of the composite until the printing process of sugar palm/PLA composite parts will be explained.

Sugar palm fiber is a natural fiber that is derived from the leaves of the sugar palm tree (Arenga Pinnata), which is native to Southeast Asia. The sugar palm tree is a tall, slender tree that grows up to 20 meters in height and has long, spiky leaves. The fibers are extracted from the tree's leaf sheaths and are used in various applications (Sanyang et al. 2016). Sugar palm fiber has several desirable properties that make it useful in the production of composite materials. These properties include high strength, stiffness, and low density, which make it a suitable candidate for use as a reinforcement material in polymer composites (Sanyang et al. 2016). Sugar palm fiber also has good thermal stability, which can be beneficial in high-

temperature applications (Jumaidin et al. 2017). In addition to its mechanical properties, sugar palm fiber is a sustainable and renewable resource. The sugar palm plant is grown for the plant's sap, which is used to make palm sugar and other products, and the leaves are a by-product of this process. Using the fiber provides an additional use for this by-product, making it an environmentally friendly material (Leman et al. 2008). Sugar palm fiber is a promising natural fiber for use in polymer composites due to its excellent mechanical properties, thermal stability, and sustainability (Bachtiar et al. 2012). As shown in Table 1, the properties of sugar palm fiber are varied with respect to the plant parts.

TABLE 1. Mechanical Properties of Sugar Palm Fiber (Asyraf et al. 2022)

Plant Part	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation at Break (%)
Ijuk	276.6	5.9	22.3
Trunk	198.3	3.1	29.7
Bunch	365.1	8.6	12.5
Frond	421.4	10.4	9.8

Sugar palm fiber composites are materials fabricated by compounding sugar palm fibers with a matrix material, typically a thermosetting or thermoplastic polymer, to create a new resultant material with improved mechanical properties. Studies by Misri et al. (2010) and Atiqah et al. (2017, 2019) have shown that sugar palm fiber composites have several advantages over traditional composites, including lower cost, higher mechanical strength, and better thermal stability. In a study conducted by Ibrahim et al. (2020) the experimental and characterization results showed significant improvement in the performance of the cornstarch (CS) and fibrous residues of both cornhusk (CHF) and sugar palm (SPF) following the hybridization process. The tensile strength and Young's modulus increased from 6.8 MPa to 19.05 MPa and from 61.15 MPa to 1133.47 MPa, respectively. On the other hand, other researchers found that the addition of sugar palm fibers for hybrid composites improved the mechanical properties of the high-density polyethylene (HDPE) matrix, including tensile strength, flexural strength, and impact strength (Ilyas et al. 2022). The resultant composite material was also found to have good water resistance, making it suitable for use in humid environments. Sugar palm fibers are obtained from the leaves of the sugar palm tree (Arenga pinnata) and have been found to have good mechanical performance, like high tensile strength and stiffness, good thermal stability and biodegradability. Azwa et al. (2013) investigated the mechanical properties of sugar palm fiber-reinforced epoxy composites. The study found that the addition of sugar palm fibers improved the tensile strength, flexural strength, and impact strength of the composites. The highest tensile strength was observed at 30% fiber loading, while the highest flexural strength was observed at 20% fiber loading. The study also found that the thermal stability of the composites increased with increasing fiber loading. Moreover, Atiqah et al. (2017a) investigated the mechanical and thermal properties of sugar palm fiber-reinforced polypropylene composites. The study found that the addition of sugar palm fibers improved the tensile strength, flexural strength, and impact strength of the composites. The highest tensile strength was observed at 30% fiber loading, while the highest flexural strength was observed at 20% fiber loading. The study also found that the thermal stability of the composites increased with increasing fiber loading. However, there is a drawback in the properties of the SPF. There are studies from past researchers about the compatibility of sugar palm fibers (SPF) that employed as reinforced materials for polymer composite. The characteristics of the SPF that are naturally hydrophilic caused a challenge in getting uniformity with a polymer that is hydrophobic. Both materials would exhibit poor interfacial adhesion if the fibers were not distributed uniformly within the polymer matrix. A study conducted by Nurazzi et al. (2022) showed that untreated SPF composite exhibited higher water absorption and led to fiber swelling as compared to alkaline-treated SPF. This situation could be avoided if the modification on the fiber surface was performed.

Chemical treatments that modify the fiber's surface can significantly improve the properties of natural fiber composites. After the treatment, impurities are eliminated, the fiber's surface area and roughness are increased and the bonding across the fibers and polymer matrix is strengthened. In addition, good fiber-matrix interlocking conditions occurred after the elimination of some constituents such as wax, lignin and pectin and it allows the optimum stress transfer within the fiber and matrix's molecule. Consequently, the mechanical properties of the composite are strengthened. Some common chemical treatments used for SPF include alkaline treatment and silane treatment. Alkaline treatment involves treating the fibers with a strong alkaline solution, such as sodium hydroxide (NaOH) or potassium hydroxide (KOH). Alkaline treatment has been shown to improve the mechanical properties of natural fiber composites. Bachtiar et al. (2010) have proven that the alkaline-treated sugar palm fiber composite exhibited higher flexural strength by 24.41% and higher tensile modulus by 148% than untreated sugar palm fiber composite in their study. Radzi et al. (2019) also agreed in their study that the fiber surface modification through an alkaline solution could improve the mechanical behavior of the sugar palm fiber composite

and could be beneficial for the application in automotive component design. Alkaline treatment on the fiber would react with hydroxyl (-OH) in natural fibers and remove thermally unstable elements such as hemicellulose and lignin partially (Nurazzi et al. 2021). Consequently, the percentage of cellulose is increased, and the study proves that the highest tensile strength was obtained with a soaking time of 60h of fibers at 5% NaOH concentration (Kathirselvam et al. 2019). However, the challenge is dealing with the hydroxyl group would lead to moisture absorption within the fibers. A higher content of moisture or water absorption would limit the application of the composite. For 3D printed composite based objects, to widen the application of the printed objects, water resistance is preferred.

Meanwhile, silane treatment involves modifying the natural fibers with a silane coupling agent, such as gamma-aminopropyltriethoxy silane (APTES) or vinyltriethoxy silane (VTES). This treatment introduces silane groups on the fiber surface, which can react with the polymer matrix to improve the adhesion between the fibers and the matrix. In comparison with untreated fibers, natural fibers that undergo surface modification by silane treatment exhibit an improvement in the mechanical and physical properties of natural fiber composites. As stated in Atiqah et al. (2018) study, the tensile strength of silane-treated sugar palm fiber composite exhibited the highest value (156.79 MPa) compared with untreated composite (133.28 MPa). Similar results were also obtained from Zahari et al. (2015) study where silane treatment that works as a coupling agent between the fiber and polymer matrix has improved the interfacial strength and consequently, the tensile strength of the composite is improved from the neat polymer. In general, fiber treatment that modified the fiber's surface can improve the adhesion between the natural fibers and the polymer matrix, increase the mechanical properties, and enhance the moisture resistance of natural fiber-reinforced composites. This can make these composites more competitive with traditional composites and increase their potential use in various applications, including construction, automotive, and packaging. Therefore, this study was conducted to examine the influence of fiber treatment on the mechanical and physical properties of composite materials.

METHODOLOGY

The execution of mechanical and physical tests and morphological observation will contribute significant data on fabricating SPF/PLA composite filament. From these experimental studies, the selection of suitable fiber treatment would assist the researcher to decide which treatment would bring a better effect in terms of strength, flexibility, and ductility of the composite filament. Hence, the flow process of this study is shown in Figure 1.

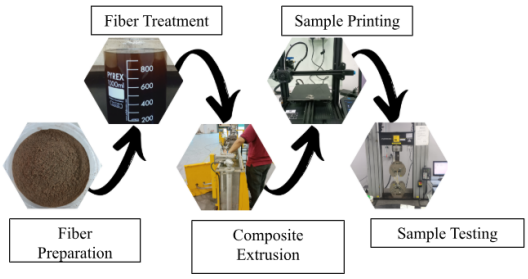


FIGURE 1. Flow Process of Investigation

MATERIALS PREPARATION

Sugar palm fiber supplied by a local supplier was obtained from the western peninsular of Malaysia, Jempol, Negeri Sembilan. While, the polymer matrix, polylactic acid (PLA) with a density of 1.24g/cm3, Ingeo TM Biopolymer 2003D pellets (100% PLA pure) were supplied by Mecha Solve Engineering. Besides, acetic acid(glacial), methanol, sodium hydroxide pellets for analysis Emsure R, and (3-aminopropyl) triethoxysilane, were obtained from Polyscientific Enterprise Sdn Bhd (Melaka). Sugar palm fibers were separated from the sugar palm tree and carefully cleaned to eliminate any dust and dried leaves, ensuring their purity. The SPF was kept in the open air and dried for 72 hours. The long fiber was cut into lengths of 1 to 3 cm and then rinsed with tap water to eliminate any impurities such as dust, dried leaves and sticks. The SPF was left to air-dry and subsequently underwent drying in an air-circulating oven at 60 ^{o}C for 48 hours before being crushed with a high-speed crusher. An industrial sieve machine was used to obtain particle-sized fiber with an average size of 125 to 250 µm.

FIBER TREATMENT

The fiber's surface modification through chemical treatment applied to SPF aimed to enhance the adhesion between the fiber and the matrix. Three (3) treatments have been performed which are alkaline, silane, and the combined- alkaline and silane treatment. During alkaline treatment, sodium hydroxide (NaOH) pellets for analysis Emsure R were used. The 6% concentration of alkaline solution was prepared. Later, the SPF was immersed in the solution for 3 hours. Following the rinsing process with distilled water until achieving a neutral pH, the fibers were subjected to drying in an oven operating at 60 ^{o}C for a

duration of 48 hours. After this drying phase, the surface of the fiber was treated with (3 -Aminopropyl) triethoxysilane(APS). This silane treatment procedure involved immersing the SPF particles in a solution of APS with a concentration of 2% for a duration of three hours. The preparation of the 2wt% silane solution entailed the mixing of APS with a methanol and distilled water mixture in a ratio of 90/10 w/w respectively. The pH of the solution was adjusted to 3.5 using acetic acid and stirred continuously for 10 minutes. The SPF was immersed in the solution for 3 hours. Then, rinse it until it is neutral. Next, oven-dried the treated SPF was performed at 60 °C for 72 hours to remove the existence of moisture. Meanwhile, for the combination of alkaline and silane treatment, the SPF was treated with a 6% concentration of NaOH solution and continued with a 2% concentration of silane solution, as the second procedure and the drying procedure are like the previous.

SAMPLE FABRICATION PROCESS

The beginning of SPF/PLA composite filament preparation involves a fiber loading of 2.5%. Furthermore, untreated SPF composites were extruded to establish the baseline for comparative analysis across different composite treatments. As illustrated in Figure 2, the process involves the introduction of PLA pellets and SPF particles into the hopper of a twin-screw extruder. Subsequently, thorough mixing of the PLA pellets and SPF particles occurs prior to their entry into the twin screw's inlet. This facilitates their conveyance into the melting chamber and subsequent extrusion through a die. The resultant composite filament is shown in Figure 3.

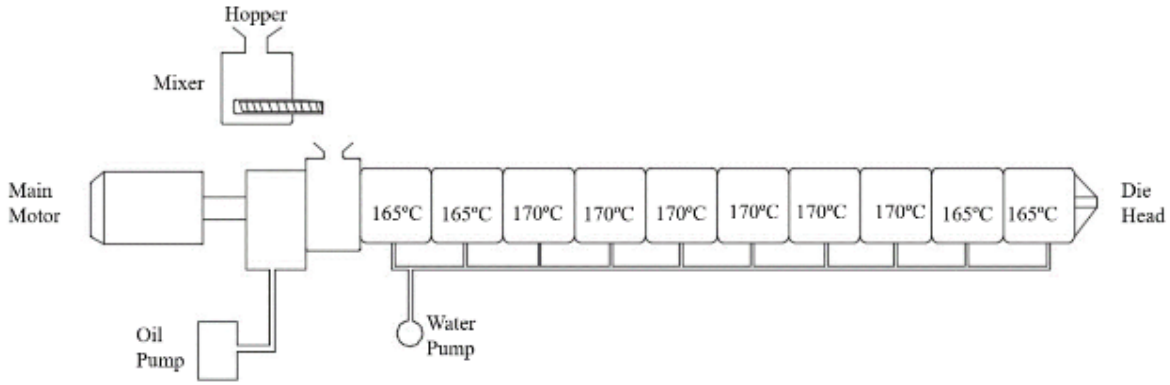


FIGURE 2. Diagram of a twin-screw extruder.



FIGURE 3. SPF/PLA filament composite with different treatment

All printed samples were fabricated using 3D printer, Creality Ender 3 as referred to in Table 2. The drawing tensile and flexural testing was done by performing Solidworks software and then a slicing software which is Creality Slicer to convert the STL file of the drawing into a G-code. The desired parameter of the composite filament printing process can be set in the slicer. The parameters used for the FDM machine to print out the specimen (according to ASTM D638 and ASTM 790) of the tensile test and the flexural test are shown in Table 3 and the printed composite samples are shown in Figure 4.

TABLE 2. List of the samples

Name of Sample	Sample Type
UT_SPF/PLA	Untreated Sugar Palm Fiber/Polylactic Acid
N_SPF/PLA	Alkaline treated Sugar Palm Fiber/ Polylactic Acid
S_SPF/PLA	Silane treated Sugar Palm Fiber/ Polylactic Acid
NS_SPF/PLA	Combined Alkaline-Silane Treated Sugar Palm Fiber/Polylactic Acid

TABLE 3. Printing parameters for SPF/PLA composite filament.

Index	Parameters	Description
1.	Infill	100 %
2.	Infill type	Lines
3.	Layer height	0.18mm
4.	Nozzle temperature	190 °C ~ 200 °C
5.	Bed temperature	80 °C ~ 85 °C
6.	Nozzle Size	0.8 mm



FIGURE 4. Printed sample of (a) flexural test (rectangular shaped) and (b) tensile test (dog-bone shaped)

TENSILE TEST

A tensile test was applied to examine the mechanical properties of the 3D printed SPF/PLA composites. Following the completion of the tensile test, various material properties can be assessed, including but not limited to yield stress, maximum elongation, tensile strain and Young's modulus. Conforming to the specifications outlined in ASTM D638, type 1 geometry, characterized by a "dog bone" shape, was adopted for the specimen. This geometry entails a span length of 50 mm and employs a load cell with a capacity of 5 kN. The standardized crosshead speed for this testing procedure is set at 1 mm/min. An assessment of the tensile properties of the composite material was conducted utilizing an Instron 5585 Universal Testing Machine. The tensile strength of the filament composite can be calculated using Equation (1).

$$\sigma=FA \quad (1)$$

where is the tensile strength of the composite (Pa), F is the maximum force at break (N) and A is the area of the cross-section (m^2).

FLEXURAL TEST

The flexural test was conducted in accordance with the ASTM D790 standard, employing a three-point bending configuration and employing the Universal Testing Machine model Instron 5585 (manufactured in United States). Consistent with standard procedure, the crosshead speed was set at 1 mm/min with a 5kN load cell applied. Adhering to ASTM specifications, the dimensions of the sample were 125 mm X 12.7 mm X 3.2 mm, and the span length was maintained at 50 mm. Utilization of Equation (2) enables the computation of the flexural test outcomes for the composite sample.

$$\sigma=3PL2bd2 \quad (2)$$

Where σ is the flexural strength of the composite (Pa), P is the maximum force at break (N), L is the support span (mm) b is the width of the beam tested (mm), and d is the depth of the beam tested (mm).

MOISTURE CONTENT

The 10 mm x 10 mm x 3 mm composite samples were fabricated for five replications. Every sample was dried at 105 °C for a full day. To determine the moisture content, the weight of the samples was measured both before and after the heating process (W_i and W_f). Equation (3) was applied to calculate the moisture content.

$$\text{Moisture content (\%)} = Wi\text{-}WfWi \times 100 \quad (3)$$

WATER ABSORPTION

After being dried at 105 °C for 24 hours, five samples with sizes of 10 mm x 10 mm x 3mm were immersed in water at an ambient temperature of 23 °C for half and two hours to eliminate any residual moisture. The samples were

weighted both before and after the immersion, and the amount of water that was absorbed was calculated using W_i and W_f as in equation (4):

$$\text{Water absorption (\%)} = Wi\text{-}WfWi \times 100 \quad (4)$$

THICKNESS SWELLING

The percentage of thickness swelling was calculated using a similar testing setup as indicated in the section on flexural testing. Samples were measured both before (T_i) and after (T_f) immersion using a digital vernier (Model: Mitutoyo) with an accuracy of 0.01 cm. To find the swelling thickness ratio, the following equation (5) was applied:

$$\text{Thickness swelling (\%)} = Ti\text{-}TfTi \times 100 \quad (5)$$

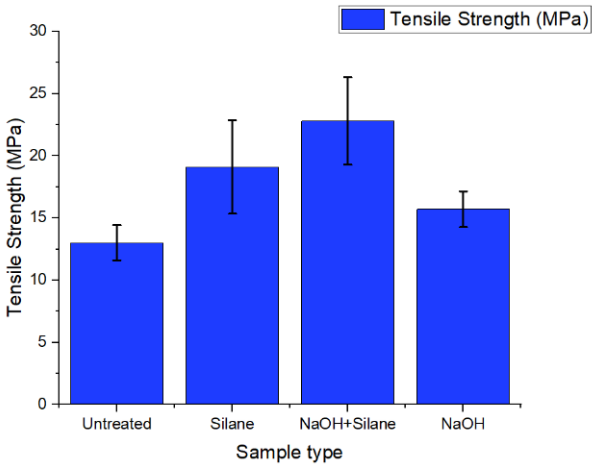
SCANNING ELECTRON MICROSCOPE (SEM)

Morphological studies were performed in detail on the fractured surface of the tensile test sample using a Scanning Electron Microscope (SEM), JSM-6010PLUS/LV, manufactured by JEOL Ltd (Japan). The four different samples taken from the tensile specimen were tested; untreated composite, alkaline, silane, and a combined alkaline-silane treated composite.

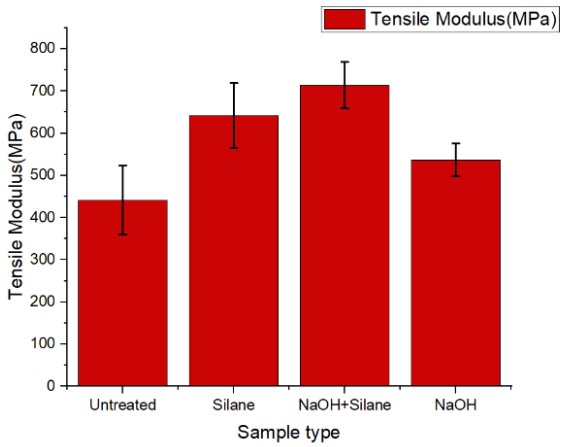
RESULTS AND DISCUSSION

TENSILE PROPERTIES

To investigate the effect of the chemical treatment of SPF on the properties of 3D printed SPF/PLA composite, mechanical testing was conducted in this study. As illustrated in Figure 5, the tensile strength of the untreated fiber composite was 13.23 MPa, which was implied as the lowest value obtained as compared to the other sample of SPF/PLA composite. The result shows the values of tensile strength increased to 15.73 MPa for alkaline treated composite, 19.11 MPa for silane-treated composite, and 22.82 MPa for the combined- alkaline and silane-treated SPF/PLA composite. The highest tensile strength for the 3D printed SPF/PLA composite filament was found for the combined- alkaline and silane-treated SPF/PLA composite. The tensile strength was improved by 75% as compared with untreated SPF/PLA composite. The higher tensile strength value is due to great interfacial bonding between the treated SPF and PLA matrix. The changes in the SPF surface after chemical treatment improved the properties of the composite, where more effective stress transfer occurred between the matrix and fibers in the composite structure (Hosseini et al. 2023). The removal of constituents from the surface of the fibers could encourage the fibers to well blend with the polymer matrix where the rough surface of the fibers can adhere to the molecules of the matrix. Moreover, improved strength is exhibited when voids within the composite structure are decreasing, hence improving the adhesion between the fibers and the matrix (Sánchez et al. 2020).The inclusion of treated SPF within the composite resulted in a reduction in polymer chain mobility, consequently enhancing the stiffness of the material. Notably, when contrasted with both untreated and differently treated composite, the composite comprising SPF/PLA treated with a combination of alkaline and silane exhibited the highest tensile modulus. This enhancement primarily stems from reinforced adhesion mechanisms.



(a)



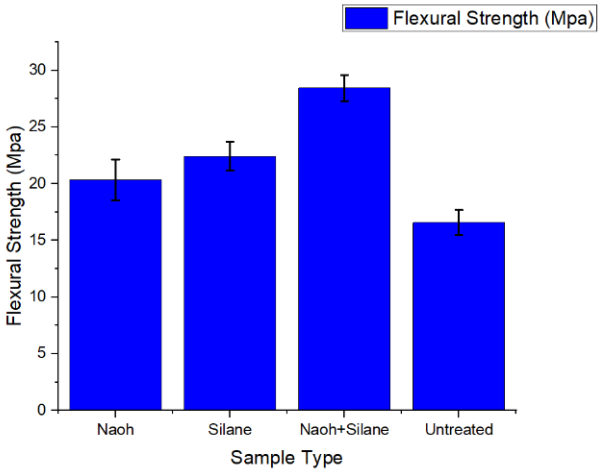
(b)

FIGURE 5. (a) Tensile strength and (b) Tensile modulus of 3D printed SPF composite with different fiber treatment

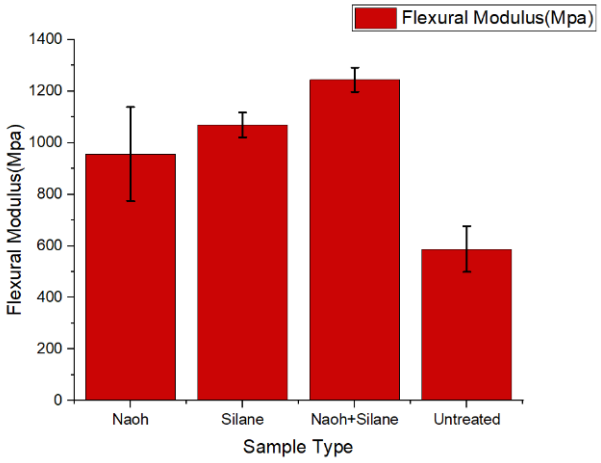
Izwan et al. (2020) where they found that 15 minutes of soaking time for chemical treatment exhibited the best tensile properties of SPF to be used as reinforcement in composites. It is also supported by the findings from Sherwani, Sapuan, et al. (2021; Sherwani, Zainudin, et al. (2021) where the highest value of tensile properties of SPF exhibited from chemically treated SPF.

FLEXURAL PROPERTIES

The type of chemical treatment exerted discernible effects on both the flexural strength and flexural modulus of the 3D-printed SPF/PLA composite, as depicted in Figure 6. Notably, the composite exhibited enhanced flexural properties when subjected to a combined treatment involving NaOH and silane, leading to improved wetting characteristics. This enhancement can be attributed to the optimization of adhesion between SPF and PLA facilitated by the reduction in the thickness of the SPF's cell wall through the NaOH and silane treatment. In contrast, the untreated SPF composite displayed the lowest flexural strength, measuring at 16.59 MPa, when compared to other treated SPF composites. According to the findings, the flexural strength exhibited distinct enhancements across various treatments: the alkaline treated SPF composite demonstrated a strength increase of 20.33 MPa, the silane treated SPF composite exhibited an increase of 22.43 MPa, and the composite treated with a combination of alkaline and silane showcased the highest enhancement at 28.44 MPa. Consequently, it was observed that the SPF/PLA composite filament subjected to combined alkaline and silane treatment attained the highest flexural strength. Moreover, a comparative analysis between treated and untreated SPF/PLA composites revealed a notable 71% increase in flexural strength. A similar finding was also found in Bachtiar et al. (2012) study where the treated SPF composite exhibited the highest value of flexural strength with 24% improvement from untreated SPF composite. Mukhtar et al. (2019) study also shows that improvement in flexural properties of chemically treated SPF composite as compared to untreated SPF composite by 16%. Another study from Widodo et al. (2020) has shown similar results of flexural strength of treated SPF composite as much as 24.03 MPa and 4.01 MPa of flexural modulus.



(a)



(b)

FIGURE 6. (a)Flexural strength and (b) Flexural modulus of 3D printed SPF composite with different fiber treatments.

According to both results of tensile and flexural testing, it is found that the improved properties of combined-alkaline and silane-treated SPF composite were obtained. A similar trend was exhibited in both tests where combined- alkaline and silane treatment showed the highest value compared to others. The reason behind the findings is the higher surface area of fibers that allows the raising of a degree of cross-linking in the interface region and enables the stronger bonding condition between fiber and matrix.

The flexural modulus of combined- NaOH and silane also shows the highest result compared with other untreated and treated composites. The chemical treatments of SPF enable more chemical bonds to form among the hydroxyl groups on the silane molecules and fiber surface which are attributed to the highest flexural modulus among others. This finding was similar to other research on SPF and thermoplastic polyurethane which also executes the

combination of NaOH and silane treatment that results in improved flexural properties of the composite (Atiqah et al. 2017a).

The effect of fiber treatment on PLA composites can be significant in terms of improving their mechanical properties. Fiber treatment can modify the surface of the fibers, which can lead to better adhesion between the fiber and the PLA matrix, resulting in improved mechanical properties of the composite. The combination of alkaline and silane treatment improved the interfacial adhesion between the SPF and the PLA matrix. It contains molecules that have both organic and inorganic groups and can react with the fiber surface and the polymer matrix, forming a covalent bond between them. This can lead to improved tensile strength and flexural strength, the PLA composite. In addition, the resistance of chain deformation was exhibited through the higher modulus of 3D printed composite.

Hence, fiber treatment can significantly improve the mechanical properties of PLA composites, and the specific type of treatment used depends on the type of fiber and the desired properties of the composite. On the other hand, other factors may influence the mechanical properties of the 3D-printed composite parts. Although the printed samples undergoing similar printing process parameters, the interlayer adhesion between the printed layers would differ as the condition of the composite filament composition is different. In addition, fiber distribution also would likely influence the quality of the printing part. The extrusion process to fabricate the composite filament is a highly crucial process where the orientation and dispersion of the fibers are uncontrollable. As in Figure 7, the fibers are not well distributed evenly. During the printing process, adhesion between the two printed layers would likely be poorer when the filament that was deposited through the nozzle was packed with fibers. For example, as in Figure 8, it is more likely to imply that the untreated SPF composite filament is more porous by the presence of the voids as compared to the treated SPF composite filament. Therefore, during the printing process, the porous filament would produce weak interlayer adhesion and influence the mechanical properties of the printed samples.

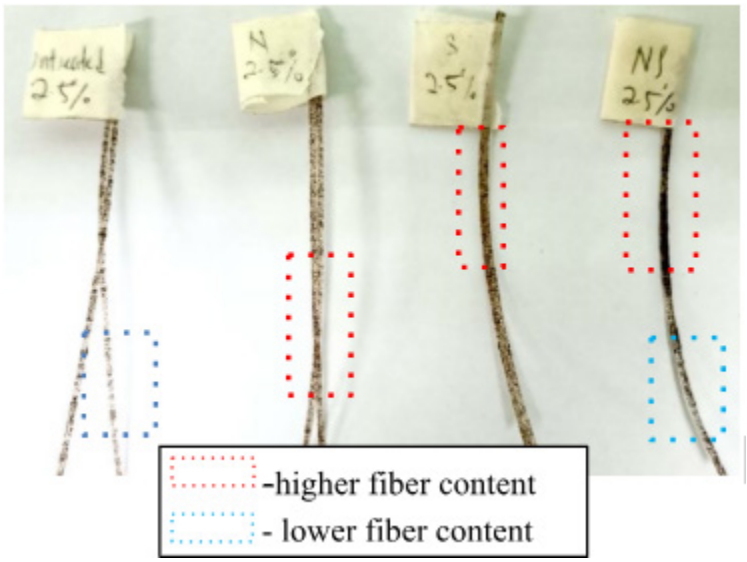


FIGURE 7. Sample of filaments

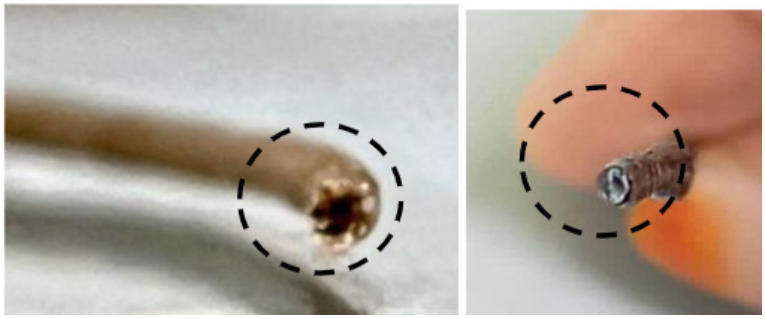


FIGURE 8. Image of (a) untreated and (b) treated SPF composite filament' cross-section

PHYSICAL PROPERTIES

As discussed in the previous section, treatment on the surface of SPF is significantly important to overcome several drawbacks and limitations of SPF as reinforced material in composite. Poor interfacial bonding of fibers and matrix would lead to the presence of voids and have more tendency to absorb a lot of water. Consequently, poor mechanical and thermal properties will be exhibited. Therefore, researchers suggested conducting fiber treatment to improve the hydrophobic properties of the SPF composite. In Table 4, it is shown that a similar trend was observed for the thickness swelling, water absorption and moisture content. Hence, combined- alkaline and silane-treated SPF composite could improve the hydrophobic properties of SPF composite by having less water uptake tendency.

TABLE 4. Results for thickness swelling (TS), water absorption (WA) and moisture content (MC) of the 3D printed SPF composite.

Samples	TS (%)		WA (%)		MC (%)
	0.5 h	2.0 h	0.5 h	2.0 h	
UT_SPF/PLA	0.81 ± 0.38	0.77 ± 0.45	4.04 ± 1.84	4.24 ± 1.69	0.94 ± 0.67
N_SPF/PLA	0.36 ± 0.19	0.36 ± 0.36	3.05 ± 0.96	3.57 ± 1.02	1.33 ± 0.44
S_SPF/PLA	0.28 ± 0.21	0.48 ± 0.34	1.11 ± 0.68	1.36 ± 0.64	1.48 ± 0.39
NS_SPF/PLA	0.30 ± 0.10	0.45 ± 0.34	0.93 ± 1.03	1.31 ± 0.91	0.52 ± 1.29

As exhibited in the results in Figure 9, the moisture content of the combined- alkaline and silane-treated SPF /PLA composite was the lowest where it is reduced by as much as 45% compared with untreated sugar palm composite. The quantity of moisture in the natural fiber could affect its mechanical properties, dimensional integrity and swelling behavior. The fibers that underwent alkaline or silane treatment show a higher value of moisture content compared with untreated fiber composite. This may be caused by the modification of its surface during the treatment where the linkage of lignin and polysaccharides were broken and increased the amount of cellulose content. Therefore, the high cellulose fiber would increase the moisture content of the composite (Mohammadi et al. 2024). It is caused by the high percentage of hydroxyl group were found in plant fiber cells and the hydrogen bond was splitted as H_2O encountered sugar palm fiber, and the -OH group formed a new hydrogen bond with H_2O molecules (refer to Equation 6) (Sherwani, Sapuan, et al. 2021). With regards to the results from mechanical properties, the combined- alkaline and silane treatment of the sugar palm fibers exhibited better tensile and flexural properties due to good interfacial bonding between the fibers' surface and PLA molecules. The strong bond implies improved hydrophobic characteristics by lessening the interaction of the hydroxyl group.

$$SPF\text{-}OH+NaOH \rightarrow SPF\text{-}O\text{-}Na+H2O \quad (6)$$

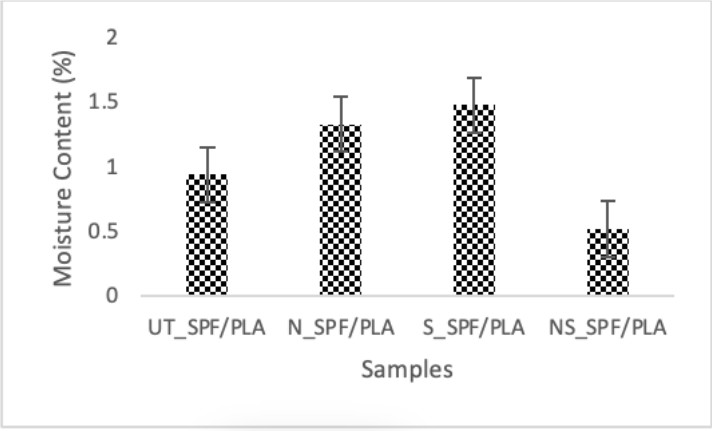


FIGURE 9. Results of moisture content of 3D printed SPF composite with different fiber treatments.

Concerning the finding on moisture content, results on water absorption and thickness swelling should follow the same trend as moisture content theoretically. Findings from water absorption would imply the sensitivity of the composite when exposed to water. As shown in Figure 10, the results agree that the combination of fiber treatment

method is effective in improving the hydrophobic characteristics of the composite where the percentage of improvement for water absorption characteristics is by 77% and 69% after half an hour and two hours immersed in water respectively in comparison with untreated fiber composite. According to Table 4, the water uptake for the combined- NaOH and silane-treated fiber composite is only about 0.93% and 1.31% for two different durations. With the same duration, untreated fiber composite exhibits as much as 4.04% and 4.24% water absorption. Hence, the treatment of the fiber through combined- NaOH and silane effectively improved the bonding condition between the fibers and molecules of the polymer where fewer water molecules are being absorbed by the composites because there are fewer voids and cracks within. Through the examination of the thickness swelling, sugar palm fibers that had undergone both alkaline and silane treatment have shown the lowest value of thickness swelling as compared to other composites with different single treatments as in Figure 11. This physical testing was done to examine the dimensional stability changes of the 3D-printed SPF/PLA composite after exposure to water or humid conditions. Characteristic of thickness swelling of the composites is significantly related to water absorption properties where the increase of water uptake within the composite structure would expand the dimension of the part.

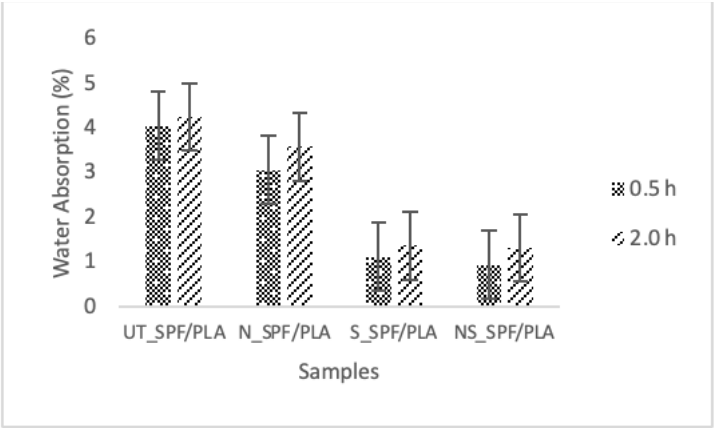


FIGURE 10. Results of water absorption of 3D printed SPF composite with different fiber treatments

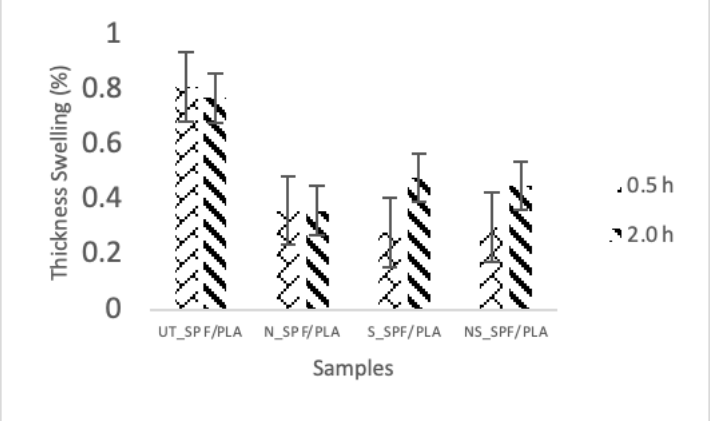


FIGURE 11. Results of thickness swelling of 3D printed SPF composites with different fiber treatments

In summary of physical testing, it is observed that fiber treatment is effective in improving the hydrophobic properties of the 3D-printed SPF/PLA composites. The order of decreasing the value of the water absorption and thickness swelling 3D printed SPF/PLA composites are as follows: UT_SPF/PLA> N_SPF/PLA> S_SPF/PLA> NS_SPF/PLA. The most significant improvement of both characteristics is combined- alkaline and silane treatment as compared to single fiber treatment. This finding also agrees with (Atiqah et al. 2019) As the natural fiber has hydrophilic characteristics by nature, the concern on the application of it in the 3D printing industry has been raised. The ability of 3D printed natural fiber composite to be applied for outdoor and high humidity or water exposure

conditions is being questioned. Therefore, with this finding, the formulation of a suitable natural fiber composite filament for FDM could be determined. Research should be done to extend the application range of this type of material and improve its water resistance and fire resistance characteristics.

SCANNING ELECTRON MICROSCOPE

Figure 12 shows the SEM images from fractured samples of 2.5% SPF/PLA composite which are untreated, NaOH, silane, and a combination of NaOH and silane-treated SPF. There was a clear observation between the treated fiber and untreated fiber which can be concluded that treated fibers gain surface roughness improvement and provided an uneven surface topography. In the untreated sample, more fibers were pulled out than in other samples, creating more holes and resulting in poor fiber-to-matrix adhesion. Chemical treatment was used to eliminate the fragments and contaminants that were present on the surface of the untreated SPF, as shown in Figure 12 (a). Untreated fibers had a smooth surface with contaminants on it. As seen in Figure 12 (b), (c), and (d), the PLA matrix covering the surface of the SPF samples demonstrated strong interfacial bonding. This was due to the SPF's surface being modified by numerous treatments. The appearance of more nodes and fibrils on the surface of SPF had occurred due to the chemical reaction from combination of NaOH and silane treatment, which produced deeper indentations within the SPF. A similar finding is found in (Kumar et al. 2023)'s study where the groove occurred on the surface of the fibers after the chemical treatment removed all the impurities. The surface becomes rougher and the bonding condition within the fiber and polymer matrix is improved for treated fiber composites. The mechanical testing's result and the SEM observation of different treated SPF/PLA composites agreed, showing that surface treatment enhanced the natural fiber composite's properties. Moreover, it was observed that poor interlayer adhesion 3D printed layers were exhibited in untreated SPF composite as in Figure 12 (a) where it is more porous and non-homogenous than the other samples. SEM image in Figure 12(b) where the fibers were treated under combined treatment reveals that fibers well embedded within the polymer matrix indicate the presence of strong interfacial adhesion between the reinforcing fibers and the matrix. Good interfacial bonding was observed and applied tensile loads are more uniformly distributed across the fiber–matrix interface, thereby reducing interfacial debonding and fiber pull-out. Consequently, enhanced stress transfer efficiency contributes to the observed improvement in tensile strength and tensile modulus.

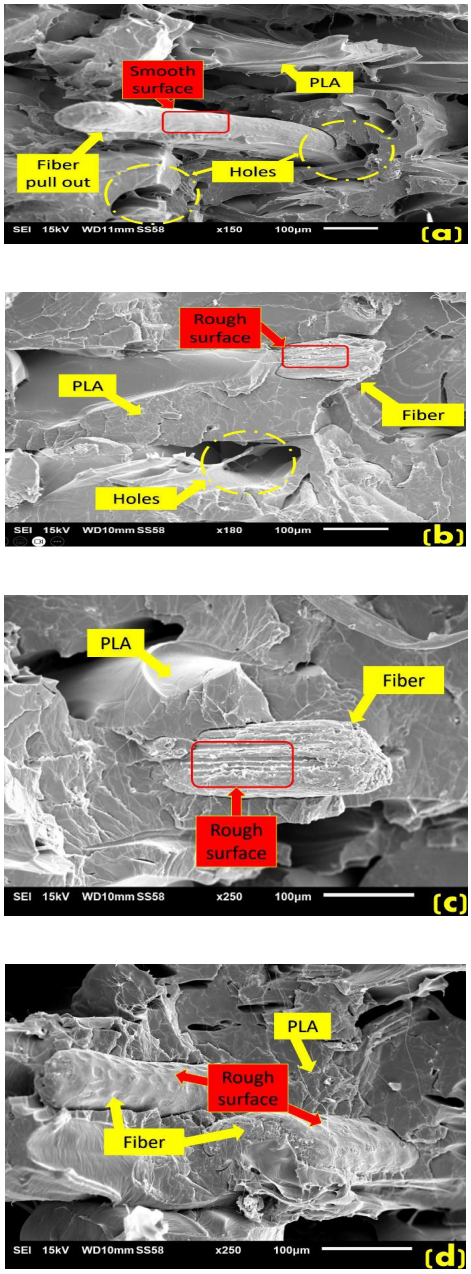


FIGURE 12: SEM images of a fractured surface of 3D printed (a) Untreated SPF/PLA, (b) Combined- NaOH and Silane treated SPF/PLA (c) Silane-treated SPF/PLA, (d) NaOH-treated SPF/PLA.

CONCLUSION

In conclusion, the study has achieved its objective of determining the appropriate fiber treatment of SPF composite that would result in greater mechanical and physical properties. The SPF composite that employed combined-NaOH and silane-treated fibers showed greater tensile strength and flexural strength by 75% and 71% improvement for both properties respectively. In physical

testing of the 3D printed SPF composite, untreated SPF composite showed the highest moisture content, water absorption, and thickness swelling as compared to treate SPF composite. The lowest value of these properties was obtained by the combined-NaOH and silane-treated SPF composite where only 1.31% of dimensional differences as compared to the untreated SPF composite due to the swelling by the moisture within the composite structure. For moisture content, 0.52% differences in weight for the combined-NaOH and silane-treated SPF composite after being heated to determine the moisture content of the composite. In SEM images, it is found that the combined-NaOH and silane-treated SPF composite has less occurrence of fiber-pull out and void as an indication of good interfacial bonding between the fibers and polymer. The surface of the fibers that appeared to be uneven and rough is due to the elimination of wax and other impurities after being soaked in NaOH and silane solution. Hence, combined-NaOH and silane treatment is recommended to be applied for SPF composite filament for FDM and further investigation is required to determine the printability of the SPF filament that should comply with process requirements of FDM 3D printing.

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DECLARATION OF COMPETING INTEREST

None.

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