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Properties of Mexican Tropical Palm Oil Flower and Fruit Fibers for Their Prospective Use in Eco-Friendly Construction Material

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Abstract: The palm oil industry is the leading source of palm oil waste fibers. The disposal of palm oil waste fibers by burning or dumping causes environmental issues such as the emission of CO_2 and a diminution in soil fertility. Natural fiber reuse in construction materials such as concrete, mortar and adobe bricks as reinforcement provides a possible eco-friendly solution for fiber waste management. Palm oil flower fibers (POFL) obtained from palm oil empty fruit bunches and palm oil fruit fibers (POFR) obtained from palm oil fruit are two important types of palm oil fibers. Valorization of palm oil fibers requires a detailed analysis of their physical, chemical and mechanical characteristics. In this research, tropical palm oil flower and palm oil fruit fibers from Mexico were studied. Fiber extraction, preparation and testing were performed to observe their characteristics, which include water absorption, density, length, section estimation, chemical composition, thermal conductivity, thermal analysis (ATG) and tensile strength. The length, diameter and density of natural fibers have a significant influence on the strength and quality of composite materials. The characteristics of fibers vary with their chemical composition. Mechanical testing of palm oil fibers indicates a large variation in the tensile strength of palm oil flower and fruit fibers. Both palm oil flower and palm oil fruit fibers exhibit bilinear tensile load–deflection behavior associated with the alignment of cellulose along their fiber axis. The thermal characteristics of fibers indicate low thermal stability and thermal conductivity, which are essential for their use in building materials.

Keywords: palm oil flower fibers; palm oil fruit fibers; fiber extraction; strength of fibers

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1. Introduction

Natural fibers have been used in different applications for centuries. Their reuse in composite materials is increasing, as they are low cost, abundant, renewable and have many environmental and economic advantages over synthetic fibers. Natural fibers from plants include jute, hemp, dates, palm, banana spine, sugar cane bagasse and coconut fibers, etc.

Among these natural fibers, palm oil fibers as waste from the palm oil industry are continuously increasing around the world. This waste constitutes 90% of biomass waste available after oil processing activities [1]. A palm tree, including oil and lignocellulosic materials, provides about 230 kg dry weight/year annually [2]. Palm trees are planted on millions of hectares of land worldwide. Large-scale palm cultivation has environmental consequences due to the production of waste fibers. Every year, nearly 30 million tons

of palm waste is generated in the world [3]. Waste fibers are traditionally burned and sometimes left in fields to decay. The burning of fibers pollutes the environment, while on the other hand, the decay of fibers in soil is time-consuming and creates environmental troubles in the form of diseases in fresh plants. Given the large quantities generated, these wastes are a risk to the environment and urgently need to be recycled. Furthermore, availability, price, performance and biodegradable nature are among the favorable factors that promote the recycling of oil palm fibers as value-added products [4]. The possibilities of recycling waste fibers in buildings and other materials are numerous and promising. Financial benefits could be generated from the fibrous waste coming from the palm oil production process as long as recycling takes place close to the production site. The cost of the raw material, i.e., waste fiber, would be null, and its transport for processing would remain limited.

Palm oil waste fibers can be used in building materials in the form of earth bricks, concrete and mortar. Natural fibers used in construction materials reduce CO_2 emissions due to the partial replacement of natural resources.

Palm oil waste fibers are extracted from palm oil empty fruit bunches and palm fruits. Palm oil flower fibers (POFL) were obtained from empty fruit bunches, and palm oil fruit fibers (POFR) were obtained from palm fruit after oil extraction [5], as shown in Figure 1. Natural fiber extraction is usually performed by mechanical processing and retting [6]. The physical, chemical, mechanical and thermal characteristics of fibers are important for their recycling in composite materials. Their physical properties include quantity, length, density, cross-section, water absorption, etc. The quantity of natural fibers in composite materials such as earth bricks ranges from 1% to 7% by mass. It varies with the type of natural fiber and composite material. A higher quantity of fibers decreases the bonding between fiber and matrix [7]. Natural fibers of any size and shape can be used as additives in construction materials. However, the common length of fibers reported in the literature varies from 2 cm to 10 cm. Increasing the length of fibers increases the tensile strength of composite materials [8–11]. For concrete applications, the recommended average fiber length is 2.5 cm [12].

(**a**) (**b**) (**c**)

Figure 1. Palm oil flower bundle with fruits (**a**), palm oil flower bundle waste (**b**) and palm oil fruit fibers (**c**).

The density of natural fibers is another important property. The density of natural fibers increases with the diameter of fibers and ranges from 0.65 to 1.4 g/cc. The low density of natural fibers makes them suitable for building materials [13–15].

Natural fibers are hydrophilic materials. Absorption of water and the presence of humidity decrease the performance and shelf life of fibers in composite materials. Treatment of fiber reduces its water absorption capacity and increases its strength [16].

The morphology of natural fibers shows that these fibers have a lumen (empty tubular structure)-based structure. With an increasing lumen area, the thickness of the wall around it decreases, which reduces the tensile strength of natural fibers.

The mechanical characteristics of natural fibers also have a considerable influence on the tensile strength of construction materials. The mechanical characteristics of fibers include tensile strength, tensile modulus, elongation to break and strain at failure [17]. The tensile strength of palm oil flower fibers ranges from 21 to 400 MPa [10,14,18]. Fibers with a higher tensile strength increase the tensile strength of composite materials and transform brittle failure into ductile failure.

The mechanical properties of fibers are influenced by their physical characteristics such as density and cross-section [19]. The heterogeneous nature, diameter and length variation, morphology and chemical composition of fibers affect their tensile strength. In the case of technical fibers, the tensile strength of natural fibers depends on the diameter of the fiber bundle, which varies with the number of elementary fibers. The diameter of palm oil empty fruit fibers ranges from 150 to 700 µm [13]. The area of natural fibers is estimated by different techniques such as scanning electron microscopy and Vernier caliper.

The chemical composition of fibers is another factor that affects their strength and characteristics. Fibers consist of cellulose, hemicellulose, lignin and pectin. Cellulose is a key element of natural fibers and is responsible for fiber tensile resistance. Natural fibers with higher cellulose content normally have higher tensile strength [7,20–23].

Natural fibers have low thermal conductivity, which makes them suitable for composite materials used for construction [24]. The addition of natural fibers improves the thermal, mechanical and acoustic characteristics of construction materials [25–28]. The addition of fibers increases the tensile strength and post-peak load-bearing capacity of construction materials [12]. In the case of earth bricks, the compressive strength of bricks increases with the addition of natural fibers [29]. Fibers are randomly distributed in composite materials. The uniform distribution of natural fibers in building materials is important to increase the tensile strength of these materials. The longitudinal distribution of fibers along the axis of bricks and mortars improves their tensile strength substantially. As fibers are lightweight, upward movement of fibers takes place during the compaction of building materials, which leads to fiber clusters. The choice of the compaction method is important to stop the movement of the fibers inside the matrix [30]. Compaction of composite materials increases the bonding between fiber and matrix by removing voids. Fiber-reinforced composites have low density and are lightweight construction materials [31].

The addition of fibers plays a key role in the improvement of thermal and hygroscopic properties as the humidity absorption/evaporation regulation. The addition of fibers to raw earth materials provides good hygroscopic and thermal behaviors with a low economic and environmental cost. The addition of natural fibers reduces shrinkage in building materials [29] and improves their insulation [6].

There are some drawbacks associated with the use of natural fibers in composite materials such as deterioration of the physical and mechanical characteristics of fibers with time due to humidity, weather and bacterial actions. The water absorption of fiber-based composite materials increases due to the hydrophilic nature of natural fibers. The presence of small voids between matrix and natural fiber reduces the effectiveness of composite materials. Chemical and physical treatments are commonly applied to increase the shelf life, strength, durability and thermal stability of fibers [28,32,33]. Alkali treatment of fibers is common. Akali treatment of natural fibers increases their tensile strength and improves their thermal characteristics. Alkali treatment increases adhesion between fiber and matrix and reduces the affinity of fibers for water [34,35]. However, the process of fiber treatment is not eco-friendly and increases the cost, so we focused only on raw palm oil fibers without any treatment.

Many reviews have been conducted on natural fibers in general and a few on palm oil fibers (raw) [5,6]. These studies show the possible recycling of natural fibers in different applications such as polymers, construction materials, geotextiles, automobile parts, etc. However, this paper focuses only on the characteristics of palm oil fibers for their prospective use in construction materials.

The objective of this study is to investigate the physical, chemical, mechanical and thermal characteristics of palm oil flower and palm oil fruit fibers taken from Mexico. These characteristics include length, cross-section, chemical composition, tensile strength, thermal conductivity, etc. Detailed analysis of fiber characteristics helps to determine their suitability for recycling in construction materials such as crude bricks, concrete and mortar.

2. Materials and Methods

2.1. Palm Oil Fibers Preparation

The characteristics of palm oil flower fibers (POFL) and palm oil fruit fibers (POFR) are essential to observe their suitability for use in composite materials. To examine their characteristics, palm oil flower and fruit fibers were taken from the Tabasco state of Mexico, which produces nearly 20% Mexican palm oil. Mexico is the sixteenth biggest palm oil producer in the world, with an annual production of 0.22 million tons in 2020–2021 and an output yield of 2.4 tons/hectare [36]. Palm oil flower fibers are extracted from palm oil empty fruit bunches, which are waste material obtained after palm fruit removal. Palm oil fruit bunches with fruit and after fruit removal are shown in Figure 2a,b respectively. Palm oil flower fibers have less diversity in size and morphology. Most palm oil flower fibers are long. On the other hand, palm oil fruit fibers, extracted from pressed palm fruit after oil extraction, have a short length. Palm oil fruit and POFR fibers are shown in Figure 2c,d respectivley. Samples of oil palm fruits fibers show more marked homogeneity than POFL fibers.

(**a**) (**b**) (**c**) (**d**)

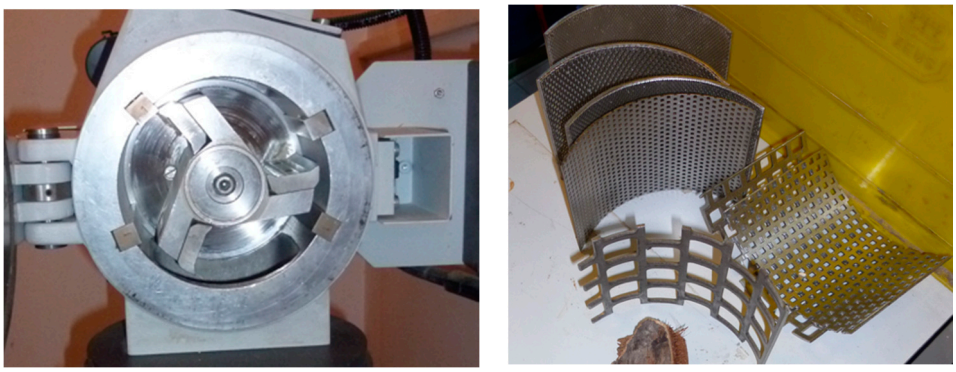
Figure 2. POFL bundle with fruits and after grinning ((**a**,**b**) respectivley), palm oil fruit and POFR short fibers ((**c**,**d**) respectively).

Palm fibers were cleaned manually to remove impurities before using them for testing. Extraction of fibers was performed manually and with machines.

First, each type of fiber was manually isolated one by one to be cut into suitable lengths for the tensile strength test and morphological study. Second, they were mechanically cut to the desired length to recycle them on an industrial scale, for example by incorporating them into composite materials (earth bricks, cementitious materials, etc.). For the tensile strength test, palm oil flower and fruit fibers were cut manually with a knife due to the specific length requirements.

For other tests such as thermal conductivity, density, water absorption, TGA and length distribution, the fibers were extracted with a knife mill, as shown in Figure 3. This machine cuts and grinds the fibers into different lengths. The use of the machine is more practical for the large-scale use of fibers. In a knife mill, the length of fibers can be controlled by using grids of various sizes from a few millimeters to a few centimeters such as 1 cm, 2 cm and 3 cm, etc. The knife mill machine with grids of different sizes is shown in Figure 3.

The different grids allow the fibers to be cut to a maximum length equal to the width of the mesh of the chosen grid. This ensures the availability of fibers of various sizes to incorporate them into different blends for composite materials, as illustrated in Figure 4.



(**a**) (**b**)

Figure 3. The three-knife mill machine (**a**) and various sized steel grids (**b**).

(**a**) (**b**) (**c**)

Figure 4. Raw POFL fibers (**a**) and cut fibers with decreasing length (**b**,**c**).

The length distribution of fibers can be observed manually and with the help of software. As the manual process is time-consuming and impractical on a large scale, the length of fibers, extracted with 2 cm and 3 cm grids, was determined with ImageJ software.

2.2. Physical, Chemical and Thermal Properties of Fibers

The physical properties of fibers include geometrical characteristics such as length and cross-section, morphology, density and water absorption. The morphology and texture of palm oil flower and fruit fibers were observed by scanning electron microscopy (SEM). The cross-sectional and longitudinal views of the fibers along with flatness and unevenness were observed by SEM. The Keyence VHX 6000 digital microscope model was used, in which the microscopic image is processed by computer-assisted drawing software to determine the area of the fiber section [27].

The absolute density of palm oil flower and fruit fibers was determined with the AccuPyc 1330 helium pycnometer from Micromeritics. The density test was repeated three times to obtain the average results. The water absorption of palm oil fibers was found by immersing the fibers in distilled water for 48 h. The water absorption of fibers is an important parameter, as it affects their performance and strength. The chemical composition of fibers is also necessary for their strength, shelf life and decomposition. For the chemical composition test, the fibers were ground and oven-dried at 38 °C for 48 h. The chemical composition of palm oil fibers was obtained with the van Soest method [37] to observe chemical components such as cellulose, lignin, hemicellulose, lignocellulose and pectin, protein, ash and other impurities.

Thermal characteristics include thermal conductivity (λ), which is steady-heat flow from the unit area of the fiber, and thermogravimetric analysis. The thermal conductivity of the fibers was measured with the heat flow method with a porcelain mold of size $15 \times 15 \times 3$ cm^3.

The thermal stability of fibers is their resistance against extreme temperatures. The thermal stability of natural fibers is measured with thermogravimetry and differential thermal analysis [38]. The thermal stability of the palm oil flower and palm oil fruit fiber bundle was measured with the TGA 295 F1 Libra thermogravimetric analyzer (Netzsch). The applied temperature ranged from 25 °C to 800 °C, with a heating rate of 10 °C/min. Temperature increases at a constant rate in thermogravimetric analysis and decomposition were noted by a change in mass with respect to temperature and time. TGA has been performed for natural fibers with the same approach by different researchers [19,39].

2.3. Mechanical Properties of Fibers

The mechanical properties of fibers play a vital role in their use as reinforcement. Fibers with good tensile strength increase the post-peak load-bearing capacity of composite materials. The mechanical characteristics of fibers include tensile strength, strain and modulus of elasticity.

The tensile strength of palm oil flower and fruit fibers was determined with the ASTM standard [40]. The Shimadzu AGS-X universal testing machine was used with 200 N sensors at a displacement rate of 0.5 mm/min. The tensile strength test was performed on the bundle of fibers extracted manually from empty fruit bunches and fibers extracted from pressed palm fruits. The fibers were sun-dried on site and oven-dried in the laboratory before testing to remove absorbed moisture. Fibers of length 3 cm were protected from bending by gluing them with a 4 cm × 4 cm cardboard paper frame. The free length of fibers inside the frame was 2 cm. The cardboard was gripped by the machine and cut from the sides to start the test. The tensile test was performed on 10 palm oil flower and 10 palm fruit fiber samples. The frame used for straightening the fibers and testing the installation is illustrated in Figure 5.

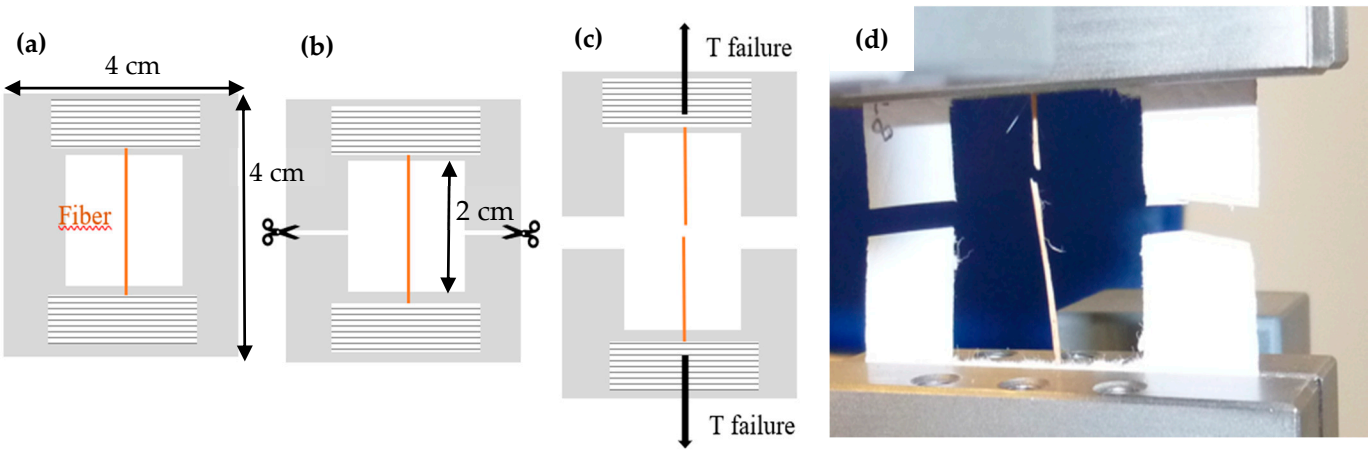


Figure 5. Natural fibers installation on a cardboard frame (**a**), cut of the frame just before testing (**b**) and failed fiber after tensile strength test (**c**,**d**).

Figure 5a shows the fiber attached with cardboard for straightening. Figure 5b shows the cutting of cardboard just before the start of the test at the center so the tensile strength was applied only on fiber. Figure 5c shows the failure of the fiber under tensile load, and Figure 5d shows the failed fiber after the tensile strength test.

3. Results and Discussion

3.1. Analysis of Fibers Physical Characteristics

3.1.1. Length Distribution of Fibers

Palm oil flower fibers extracted with the knife mill machine with 2 cm and 3 cm grids have a wide range of fiber length distributions. The aspect of fibers after mechanical cutting is shown in Figure 6a,b with the size distribution of fibers cut with 2 cm and 3 cm grids.

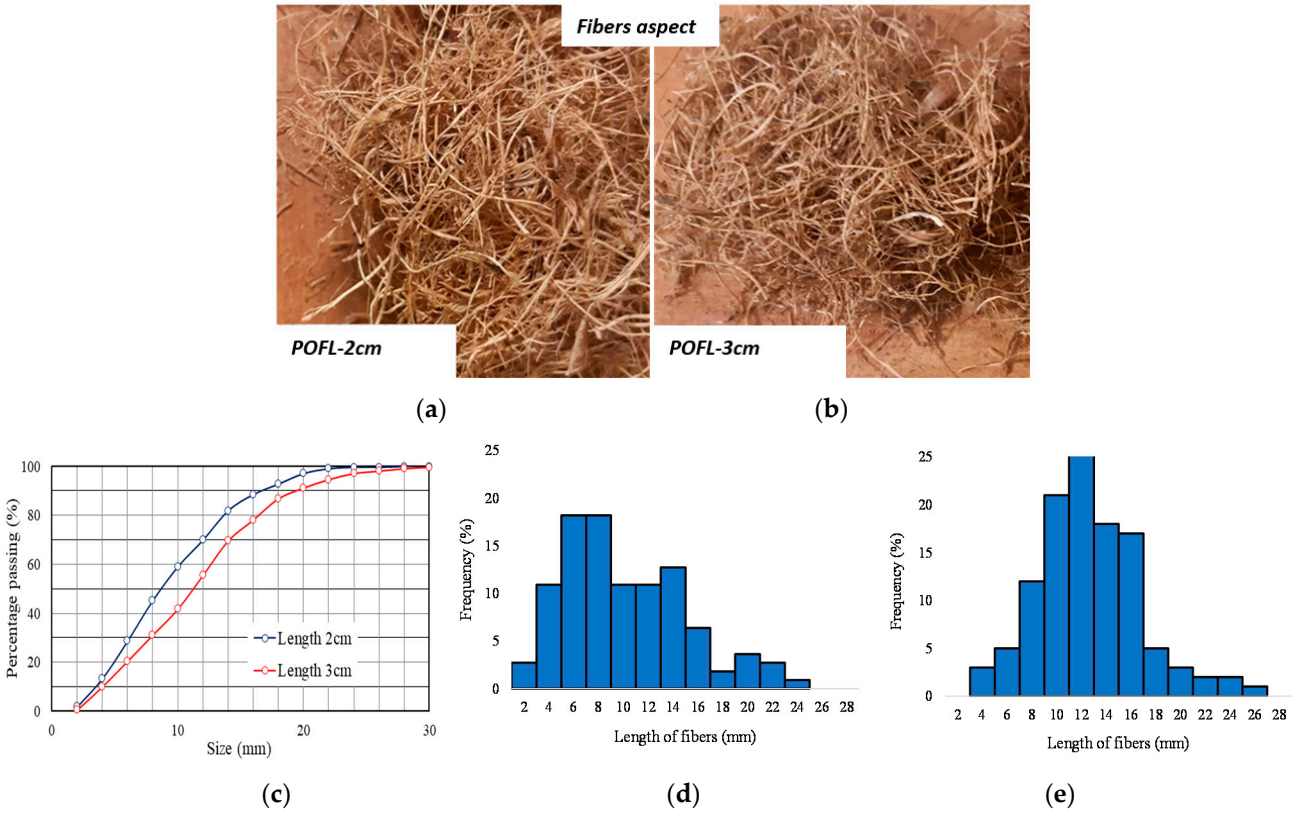


Figure 6. POFL fibers aspect (**a**,**b**), cumulative length distribution (**c**) and individual length distribution for 2 cm long and 3 cm long fibers ((**d**,**e**) respectively).

It can be observed in Figure 6c that there are both short and long fibers, but the percentage of fine-sized particles is higher with the 2 cm grid. Figure 6d,e show that for most of the fibers, the length is varied between 2 mm and 24 mm for POFL fibers from 2 cm grids and 2 mm to 26 mm for POFL fibers from 3 cm grids. The length distribution of the fibers is not uniform. The average length and maximum length of palm oil flower fibers after cutting with a grid of 2 cm (G-2 cm) and a grid of 3 cm (G-3 cm) are summarized in Table 1.

Table 1. Average and maximum length of palm oil flower fibers.

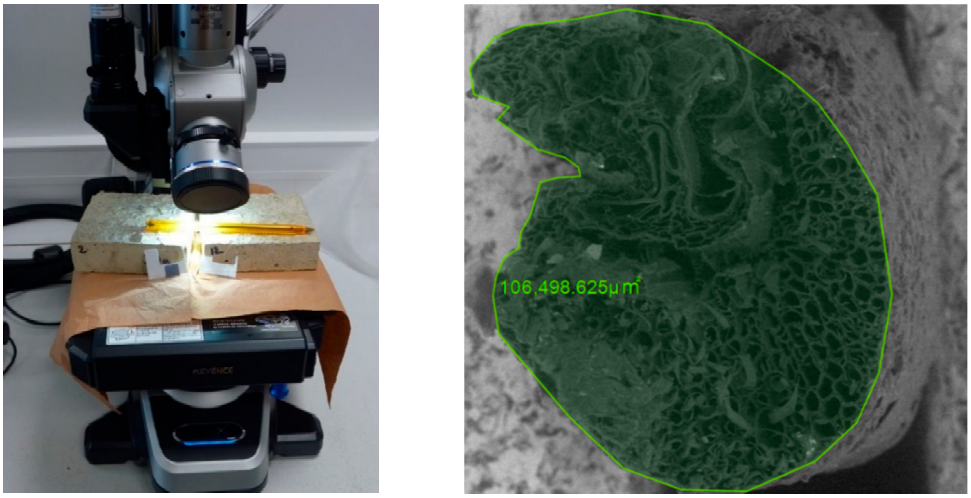
Grid Size	Average Length (mm)	Maximum Length (mm)
2 cm	9.48	24.05
3 cm	11.5	32.96

The length distribution of palm oil fibers in Figure 6 and Table 1 shows that palm oil flower fibers can be classified as long fibers, as their lengths range between 5 mm and 50 mm [41].

The natural length of fibers is essential for concrete and earth bricks for reinforcement, as they play a critical role in increasing the toughness of the material.

3.1.2. Cross-Section of Fibers

The area of palm oil flower fibers was found with the digitization of an electronic image captured either by an optical microscope (DEM image), as shown in Figure 7a, or by an environmental scanning electron microscope (SEM image). An SEM image of a typical section of a palm oil flower is given in Figure 7b. The section of fibers is estimated by considering the natural fibers as elliptical. The area of fibers can also be found with a Vernier caliper, albeit with less accuracy in the results.



(**a**) (**b**)

Figure 7. Digital microscope used after tensile test for estimation of area (**a**) and area estimation of palm oil flower fiber from SEM image (**b**) after pull-out test.

The actual area of natural fibers is always smaller than the measured area due to the presence of a porous structure, as presented in Figure 7b, inside the natural fibers [20]. The average area of palm oil flower and palm oil fruit fibers determined with microscopic analysis is shown in Table 2.

Table 2. Total average area of palm oil fibers.

Method	POFL Area (mm^2)	POFR Area (mm^2)
Microscopic analysis	0.070	0.027

Table 2 shows that the total area of palm fruit fibers is smaller than the area of palm oil flower fibers. Palm fruit fibers are thin and small in length. Moreover, the area of technical fibers changes with the number of elementary fibers. POFL fibers include both coarse and fine fibers, as observed in Figure 6. The heterogeneous nature of fibers, assumption of a circular or elliptical shape of fibers and apparatus limitations contribute towards bias in section estimation.

3.1.3. Water Absorption of Fibers

Palm oil flower and palm fruit fibers are hydrophilic in nature, which is associated with cellulose and lignin content. The presence of a hydroxyl group in cellulose and lignin increases water absorption in palm oil flower and fruit fibers [15]. The average water absorption coefficient for palm oil flower and fruit fibers is shown in Table 3.

Table 3. Water absorption of palm oil fibers.

Fiber Type	Water Absorption (%)
Palm oil flower POFL	235
Palm oil fruit POFR	258

The water absorption of natural fibers increases with the increase in the number of pores present in the natural fibers. The swelling of fibers takes place with the absorption of water. The higher water absorption of natural fibers makes them vulnerable to weathering and climate effects, and their strength decreases. Palm oil fibers contain 10% water-soluble content [38]. Therefore, precautions must be taken for their application in humid environments. Treatment of fibers decreases their water absorption.

3.1.4. Density of Fibers

Low-density natural fibers are important for their use in lightweight building materials. The measured density of palm oil flower fibers and palm oil fruit fibers is shown in Table 4, and the density of palm oil flower fibers reported in the literature is also shown. The addition of fibers decreases the density of composite materials, which is important for building structures [15].

Table 4. Density of palm oil fibers.

Fiber Type	Measured Density (g/cm^3)	Literature Density (g/cm^3)	References
Palm oil flower POFL	1.37	1.15−1.40	Fang et al., 2017
Palm oil fruit POFR	1.36		Karina et al., 2008

3.1.5. Morphology of Palm Oil Fibers

The morphology of fibers was observed by scanning electron microscopy. Palm oil flower and fruit fibers have different lengths and thicknesses, as mentioned above. Palm oil flower fibers have high morphological heterogeneity. Figure 8A,B shows an aligned, nonwoven and irregular structure of the bundle of fibers due to the presence of noncellulosic particles such as lignin, pectin etc. The cross-section of elementary palm fruit fibers can be seen in Figure 8D, in which the alignment of fibers is like a honeycomb structure that shows strong binding. Porous surfaces in the honeycomb structure of fibers are usually considered circular or cylindrical, but Figure 8D shows that there is variability in the size and shape of these structures. Small-diameter hollow structures are at the boundaries, and coarse-diameter hollow structures are situated in the center of the fiber. Clusters of small-diameter tubes are the majority. Similar observations for coir, jute and sisal fibers are reported in the literature [20]. The presence of hollow tubes at the surface of natural fibers increases the roughness of its surface, which is important for composite materials, as it contributes to the bonding of fiber and matrix [42]. The geometry of the cell also has a significant impact on the tensile strength of plant fibers [16].

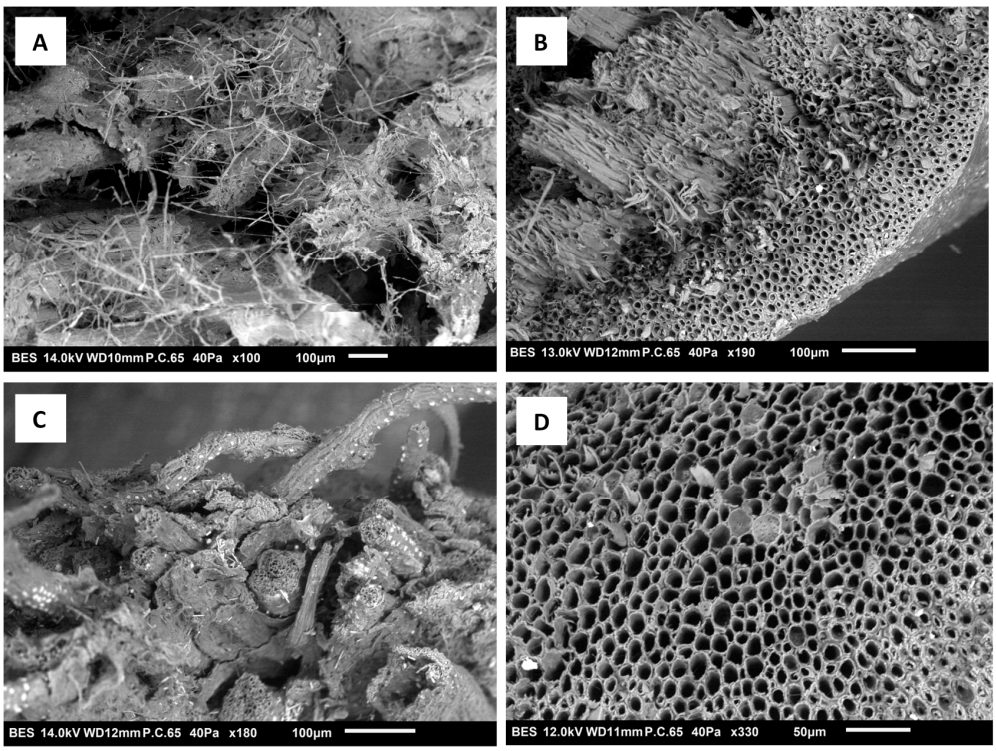


Figure 8. SEM images of palm oil flower (**A**,**B**) and fruit fibers (**C**,**D**).

3.2. Chemical Characteristics Analysis

Chemical Composition of Fibers

Chemical composition plays an important role for the shelf life and the decomposition of natural fibers. Palm oil fibers consist mainly of cellulose, hemicellulose, lignin and water-soluble compounds. The chemical composition of palm oil flower and fruit fibers from Mexico is shown in Figure 9. Palm oil flower fibers contain 48.84% cellulose, 23.32% hemicellulose, 11.61% lignin and cutins and 16.23% water-soluble compounds. Similarly, the chemical composition of palm fruit fibers can be observed in Figure 9.

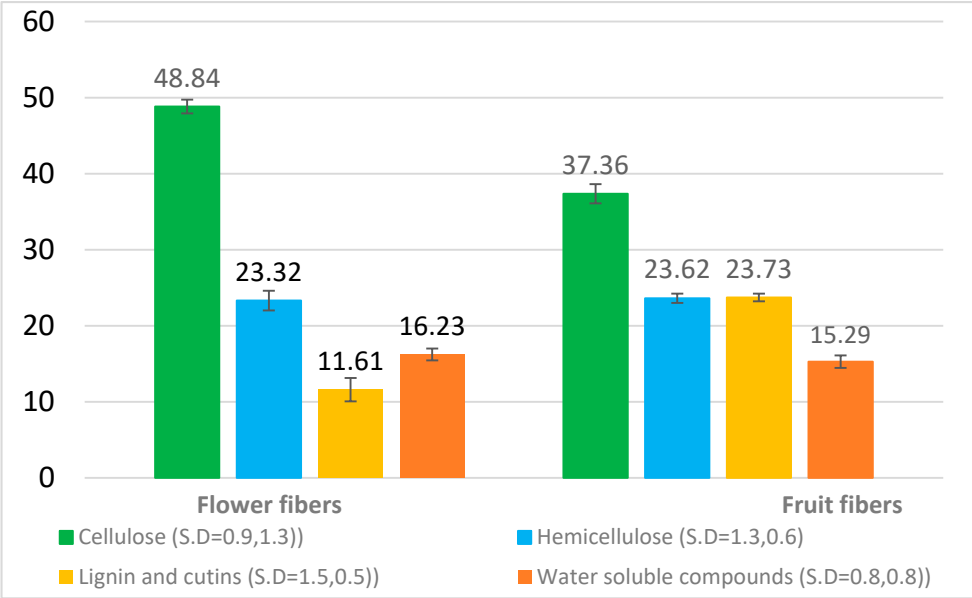


Figure 9. Biochemical fraction of POFL and POFR fibers. Note: S.D = standard deviation for palm flower fibers and fruit fibers, respectively.

The chemical composition of palm oil flower fibers reported by different researchers is presented in Table 5. There is significant variation in cellulose, hemicellulose and lignin content of palm oil flower fibers reported in different studies.

Table 5. Chemical composition of natural fibers.

Reference	Type of Fiber	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Ash (%)
[13]	OPEFB	59	2.1	25	3.2
[20]	OPEFB	49.44	23.19	12.56	-
[36]	OPEFB	65	-	19	2

Note: OPEFB = oil palm empty fruit bunch.

3.3. Analysis of Fibers Thermal Characteristics

3.3.1. Thermogravimetric Analysis

Thermogravimetric analysis (TGA) was performed for palm oil flower and palm oil fruit fibers to observe the mass loss of fibers with temperature. Mass loss occurs due to different reactions at high temperatures. Water evaporation takes place from natural fibers at a temperature between 100 and 200 °C. This is followed by the burning of organic matter at a temperature of 300 to 500 °C. The organic matter of fibers consists mainly of cellulose and lignin.

Figure 10 shows the TGA curve of one sample test on palm oil flower fibers. The test was repeated three times to obtain an average value. Average mass loss at three different temperatures is shown in Figure 11. Precisely 5.30% of mass loss happens at 29 °C to 180 °C, which is mainly associated with the dehydration of fibers. In the second stage, up

to 460 °C, 65.1% mass loss occurs, which is associated with the decomposition of cellulose, hemicellulose, lignin and pectin content. From 460 °C to 600 °C, there is a slight mass loss, followed by constant behavior, which shows the mass stabilization due to remaining ash and impurities.

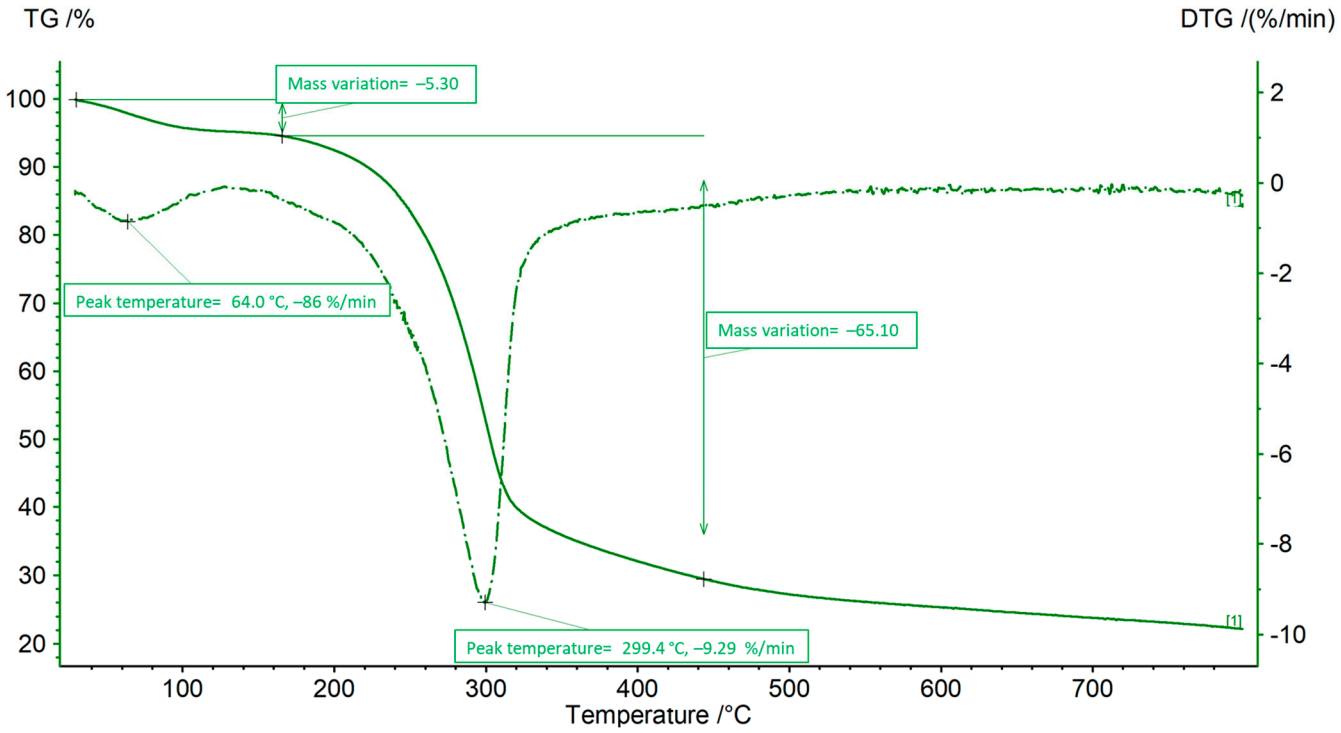


Figure 10. POFL fiber mass loss with temperature.

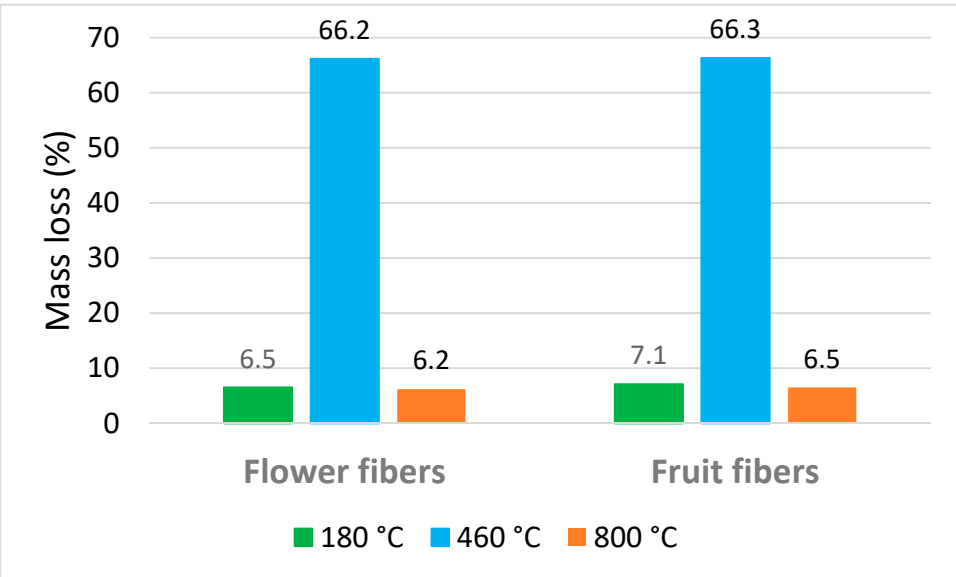


Figure 11. POFL and POFR fiber mass losses at different temperature ranges.

The average mass loss at different temperatures for palm oil flower and palm oil fruit fibers is shown in Figure 11.

The average mass loss of palm oil flower fibers for the entire temperature range, i.e., from 0 °C to 800 °C, is around 79%, while for palm fruit fibers, the average mass loss for this temperature range is around 80%. Mass loss is nearly similar in both types of

fibers, and this is because both fibers come from the same plant with similar chemical compositions. The remaining mass of nearly 20% belongs to ash and impurities and other chemical components.

3.3.2. Thermal Properties of Fibers

The average thermal conductivity and thermal resistivity of palm oil flower and palm oil fruit fibers are shown in Table 6.

Table 6. POFL and POFR fiber measured thermal conductivity and resistivity.

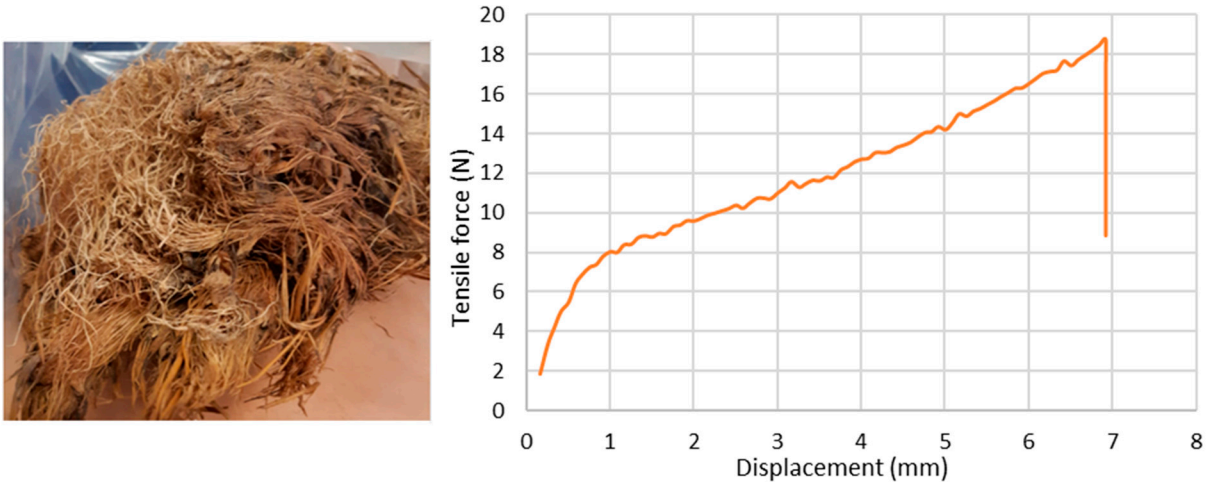
Type of Fiber	Thermal Conductivity (W/mK)	Thermal Resistivity (m^2K/W)
POFL fiber	0.058	0.80
POFR fiber	0.055	0.84

Both flower and fruit have low thermal conductivity. The thermal conductivity of natural fibers is usually low, i.e., coconut fibers have a thermal conductivity of 0.078 W/mK. Due to the low thermal conductivity of natural fibers, the thermal conductivity of composite materials such as earth bricks is also low, which makes fibers suitable for their use in thermal insulation materials used in buildings [29].

3.4. Analysis of Fibers Mechanical Characteristics

Fibers exhibit two sorts of behavior on loading, which are elastic and elastoplastic behavior. Elastic behavior follows Hooke's law. Glass and metal elements such as steel fibers follow this behavior. Most natural fibers such as palm oil flower fibers show elastoplastic behavior. This is due to the presence of cellulose, which is a chain-like structure. In the tensile test, when one end of natural fiber is fixed and the other is pulled, the cellulose aligns itself along the fiber axis and shows elastic behavior at a certain load. After that, the behavior transforms into plastic deformation.

The tensile load–deflection curve for a palm oil flower fiber is shown in Figure 12, which shows that palm oil flower fibers exhibit elastoplastic behavior. Both palm oil flower and fruit fibers show elastoplastic behavior.



(**a**) (**b**)

Figure 12. POFL fibers (**a**) and typical POFL fiber load–deflection curve (**b**).

The zone of influence of tensile force for low strength and high strength for palm oil flower and fruit fibers is shown in Figure 13.

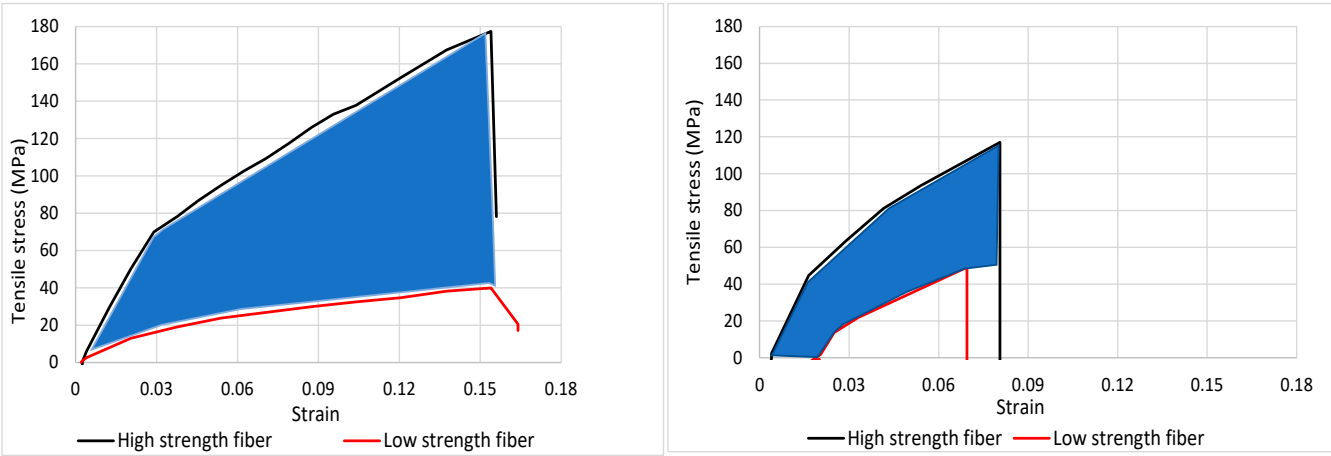


Figure 13. Palm oil flower (**a**) and fruit (**b**) fibers stress–strain zone of influence.

The overall tensile load-bearing capacity of palm oil flower fibers is greater than the palm oil fruit fibers. This is because palm oil flower fibers are smooth fibers, while palm oil fruit fibers are obtained by crushing the palm fruit after palm oil extraction. Moreover, the cellulose content of POFL fibers is greater than the cellulose content of POFR fibers, as natural fibers with higher cellulose content have higher tensile strength [20]. The average tensile stress of palm oil flower and fruit fibers is summarized in Table 7. The average initial tensile modulus (E_{t1}) for palm oil flower and fruit fibers determined from the elastic part of the tensile stress–strain curve before hardening and tensile modulus (E_{t2}) for the plastic phase is also presented in Table 7. The average ultimate tensile strength of 10 POFL and POFR fibers was determined. The minimum, maximum and average ultimate tensile stress observed for each type of fiber is summarized in Table 7.

Table 7. Ultimate tensile stress values and E_t tangent tensile deformation modulus.

Type of Fiber	$\sigma_{max.}$ (MPa)	$\sigma_{min.}$ (MPa)	$\sigma_{average}$ (MPa)	E_{t1} (GPa)	E_{t2} (GPa)
POFL	181.33	31.72	104.33	1.3	0.38
POFR	117.64	49.09	85.00	3	0.72

Table 7 shows that palm oil flower fibers have good tensile strength. The tensile strength of palm oil fruit fibers is comparatively low. POFR fibers are obtained by crushing the palm fruit, which affects their strength.

The tensile strength of fibers varies with the type of fibers and diameter of fibers. Long-size natural fibers are weak and break easily, as the tensile strength of long fibers is affected by the presence of weak links inside the fibers [17]. For the traction test, we only select fibers of a 3 cm length, and fibers used for the tensile test are free from knots. These changes may lead to a bias for future interpretation of mechanical characteristics. Another problem associated with the tensile strength test is the use of palm fruit fibers of sufficient size, i.e., fibers of a 3 cm length for the tensile strength test, which is sometimes difficult. Palm fruit fibers of a 3 cm length represent only about 2% of the available fibers.

The deformation modulus and tensile strength of fibers are directly proportional to the cellulose content of natural fibers. The deformation modulus of natural fibers ranges from 0.57 to 9 GPa [17,18]. The values of tensile strength of natural fibers, found in the literature, are shown in Table 8.

Table 8. Mechanical properties of palm oil and other natural fibers.

Reference	Type of Fibers	Tensile Strength (MPa)	Tensile Modulus (GPa)
[10]	Palm fruit fibers	110	0.95
[36]	Palm flower and fruits	248	2
[10]	Coconut	162	2.49
[8]	Date palm	170–290	0.232

4. Conclusions

In this study, the characteristics of palm oil flower and fruit fibers were studied. It is observed that the length of palm flower fibers is higher than palm fruit fibers and meets the requirements of the construction industry. Tensile strength testing of fibers shows that palm oil fruit fibers have higher tensile strength compared to palm oil fruit fibers. Tensile load–deflection curves show that palm oil flower fibers and palm fruit fibers exhibit elastoplastic behavior. The average area of palm oil fruit fibers is around 0.07 mm^2. Based on the different characteristics of palm oil flower and fruit fibers such as length, diameter, thermal conductivity and tensile strength and their comparison with the properties of natural fibers used in the literature, it can be concluded that palm oil flower and fruit fibers can be used for composite materials such as concrete, mortar and earth blocks in the construction industry.

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References

1. Dungani, R.; Aditiawati, P.; Aprilia, S.; Yuniarti, K.; Karliati, T.; Suwandhi, I.; Sumardi, I. Biomaterial from Oil Palm Waste: Properties, Characterization and Applications. *Palm Oil* **2018**, *32*. [CrossRef]
2. Sumathi, S.; Chai, S.; Mohamed, A.R. Utilization of oil palm as a source of renewable energy in Malaysia. *Renew. Sustain. Energy Rev.* **2008**, *12*, 2404–2421. [CrossRef]
3. Hasamudin, W.; Soom, R.M. *Road-Making Using Oil Palm Fiber (BIT5)*; Malaysian Palm Oil Board Information Series; Malaysian Palm Oil Board, Ministry of Primary Industries, Malaysia: Kuala Lumpur, Malaysia, 2002; Volume 171, 4p, ISSN 1511-7871.
4. Liew, W.L.; Muda, K.; Kassim, M.A.; Affam, A.C.; Loh, S.K. Agro-industrial waste sustainable management—A potential source of economic benefits to palm oil mills in malaysia. *J. Urban. Environ. Eng.* **2017**, *11*, 108–118. [CrossRef]
5. Alhijazi, M.; Zeeshan, Q.; Safaei, B.; Asmael, M.; Qin, Z. Recent Developments in Palm Fibers Composites: A Review. *J. Polym. Environ.* **2020**, *28*, 3029–3054. [CrossRef]
6. Kicińska-Jakubowska, A.; Bogacz, E.; Zimniewska, M. Review of Natural Fibers. Part I—Vegetable Fibers. *J. Nat. Fibers* **2012**, *9*, 150–167. [CrossRef]

7. El Azhary, K.; Chihab, Y.; Mansour, M.; Laaroussi, N.; Garoum, M. Energy Efficiency and Thermal Properties of the Composite Material Clay-straw. *Energy Procedia* **2017**, *141*, 160–164. [CrossRef]
8. Hakkoum, S.; Kriker, A.; Mekhermeche, A. Thermal characteristics of Model houses Manufactured by date palm fiber reinforced earth bricks in desert regions of Ouargla Algeria. *Energy Procedia* **2017**, *119*, 662–669. [CrossRef]
9. Purnomo, H.; Priadi, D.; Lumingkewas, H.R.; Lumingkewas, R.H. Strength Improvement of Early Age Unfired Soil Lime Bricks. *Adv. Mater. Res.* **2013**, *689*, 299–303. [CrossRef]
10. Danso, H. Use of Agricultural Waste Fibres as Enhancement of Soil Blocks for Low-Cost Housing in Ghana. Ph.D. Thesis, University of Portsmouth, Portsmouth, NH, USA, 2015; 258p.
11. ASTM D7357–07. *Standard Specification for Cellulose Fibers for Fiber-Reinforced Concrete*; ASTM International: West Conshohocken, PA, USA, 2012.
12. Hussain, M.; Levacher, D.; Leblanc, N.; Zmamou, H.; Djeran Maigre, I.; Razakamanantsoa, A. Influence of palm oil fibers length variation on mechanical properties of reinforced crude bricks. In Proceedings of the 4th International Conference on Bio-Based Building Materials ICBBM 2021, Barcelona, Spain, 16–18 June 2021; pp. 628–633.
13. Fang, T.W.; Malaysia, M.U.S.; Asyikin, N.S.S.N.; Shawkataly, A.K.H.P.; Kassim, M.H.M.; Syakir, M. Water Absorption and Thickness Swelling of Oil Palm Empty Fruit Bunch (OPEFB) and Seaweed Composite for Soil Erosion Mitigation. *J. Phys. Sci.* **2017**, *28*, 1–17. [CrossRef]
14. Ismail, S.; Yaacob, Z. Properties of laterite brick reinforced with oil palm empty fruit bunch fibres. *Pertanika J. Sci. Technol.* **2011**, *19*, 33–43.
15. Karina, M.; Onggo, H.; Abdullah, A.D.; Syampurwadi, A. Effect of Oil Palm Empty Fruit Bunch Fiber on the Physical and Mechanical Properties of Fiber Glass Reinforced Polyester Resin. *J. Biol. Sci.* **2007**, *8*, 101–106. [CrossRef]
16. Bledzki, A. Composites reinforced with cellulose based fibres. *Prog. Polym. Sci.* **1999**, *24*, 221–274. [CrossRef]
17. Defoirdt, N.; Biswas, S.; De Vriese, L.; Tran, L.Q.N.; Van Acker, J.; Ahsan, Q.; Gorbatikh, L.; Van Vuure, A.; Verpoest, I. Assessment of the tensile properties of coir, bamboo and jute fibre. *Compos. Part A Appl. Sci. Manuf.* **2010**, *41*, 588–595. [CrossRef]
18. Levacher, D.; Saouti, L. Determination of Some Physical and Mechanical Properties of Mexican Natural Fibers, ANR-CONACYT: VAL-USES Project 2018–2020, Task 4: Characteristics, Future Uses and Valorization of Sediments 2020. Available online: https://imu.universite-lyon.fr/international/mexique/depot-du-projet-anr-conacyt-val-uses/ (accessed on 22 October 2021).
19. Khennache, M.; Mahieu, A.; Ragoubi, M.; Taibi, S.; Poilane, C.; Leblanc, N. Physicochemical and Mechanical Performances of Technical Flax Fibers and Biobased Composite Material: Effects of Flax Transformation Process. *J. Renew. Mater.* **2019**, *7*, 821–838. [CrossRef]
20. Fidelis, M.E.A.; Pereira, T.V.C.; Gomes, O.D.F.M.; Silva, F.D.A.; Filho, R.D.T. The effect of fiber morphology on the tensile strength of natural fibers. *J. Mater. Res. Technol.* **2013**, *2*, 149–157. [CrossRef]
21. Ramlee, N.A.; Jawaid, M.; Zainudin, E.S.; Yamani, S.A.K. Tensile, physical and morphological properties of oil palm empty fruit bunch/sugarcane bagasse fibre reinforced phenolic hybrid composites. *J. Mater. Res. Technol.* **2019**, *8*, 3466–3474. [CrossRef]
22. Barrera, I.; Allieri, M.A.; Estupiñan, L.; Martínez, T.; Aburto, J. Technical and economical evaluation of bioethanol production from lignocellulosic residues in Mexico: Case of sugarcane and blue agave bagasses. *Chem. Eng. Res. Des.* **2016**, *107*, 91–101. [CrossRef]
23. Bhatnagar, R.; Gupta, G.; Yadav, S. A review on composition and properties of banana fibers. *Int. J. Sci. Eng. Res.* **2015**, *6*, 49–52.
24. Chaib, H.; Kriker, A.; Mekhermeche, A. Thermal Study of Earth Bricks Reinforced by Date palm Fibers. *Energy Procedia* **2015**, *74*, 919–925. [CrossRef]
25. Rao, P.R.; Ramakrishna, G. Experimental Investigation on Mechanical Properties of Oil Palm Empty Fruit Bunch Fiber Reinforced Cement Mortar. *Mater. Today Proc.* **2021**, *46*, 471–477. [CrossRef]
26. Salih, M.M.; Imbabi, M.S.; Osofero, A.I. Mechanical properties of fibre-reinforced mud bricks. In Proceedings of the 2nd Conference Civil Engineering, Khartoum, Sudan, 3–5 December 2018. Available online: https://www.researchgate.net/publication/329786369 (accessed on 28 June 2021).
27. Bui, H.; Sebaibi, N.; Boutouil, M.; Levacher, D. Determination and Review of Physical and Mechanical Properties of Raw and Treated Coconut Fibers for Their Recycling in Construction Materials. *Fibers* **2020**, *8*, 37. [CrossRef]
28. Wei, J.; Meyer, C. Degradation mechanisms of natural fiber in the matrix of cement composites. *Cem. Concr. Res.* **2015**, *73*, 1–16. [CrossRef]
29. Subramanian, G.K.M.; Balasubramanian, M.; Kumar, A.A.J. A Review on the Mechanical Properties of Natural Fiber Reinforced Compressed Earth Blocks. *J. Nat. Fibers* **2021**, 1–15. [CrossRef]
30. Bui, H.; Boutouil, M.; Sebaibi, N.; Levacher, D. Effect of coconut fibres content on the mechanical properties of mortars. In Proceedings of the 3rd International Conference on Bio-Based Building Materials, Belfast, UK, 26–28 June 2019.
31. Salih, M.M.; Osofero, A.I.; Imbabi, M.S. Critical review of recent development in fiber reinforced adobe bricks for sustainable construction. *Front. Struct. Civ. Eng.* **2020**, *14*, 839–854. [CrossRef]
32. Mostafa, M.; Uddin, N. Effect of Banana Fibers on the Compressive and Flexural Strength of Compressed Earth Blocks. *Buildings* **2015**, *5*, 282–296. [CrossRef]
33. Bergström, S.G.; Gram, H.-E. Durability of alkali-sensitive fibres in concrete. *Int. J. Cem. Compos. Light. Concr.* **1984**, *6*, 75–80. [CrossRef]

34. Campos, A.; Neto, A.S.; Rodrigues, V.; Luchesi, B.; Mattoso, L.; Marconcini, J. Effect of raw and chemically treated oil palm mesocarp fibers on thermoplastic cassava starch properties. *Ind. Crop. Prod.* **2018**, *124*, 149–154. [CrossRef]
35. Then, Y.Y.; Ibrahim, N.A.; Zainuddin, N.; Hidayah, A.; Wan Yunus, W.M.Z.; Chieng, B.W. Static mechanical, interfacial, and water absorption behaviors of alkali treated oil palm mesocarp fiber reinforced poly (butylene succinate). *BioResources* **2015**, *10*, 123–136. [CrossRef]
36. USDA. United States Department of Agriculture, Foreign Agricultural Service 2021. Available online: https://ipad.fas.usda.gov/cropexplorer/cropview/commodityView.aspx?startrow=1&cropid=4243000&sel_year=2021&rankby=Production (accessed on 28 June 2021).
37. Van Soest, P.J.; Robertson, J.B. *Analysis of Forages and Fibrous Foods, a Laboratory Manual for Animal Science 1985*; Cornell University: Ithaca, NY, USA, 1985; 202p.
38. Sreekala, M.S.; Kumaran, M.G.; Thomas, S. Oil palm fibers: Morphology, chemical composition, surface modification, and mechanical properties. *J. Appl. Polym. Sci.* **1997**, *66*, 821–835. [CrossRef]
39. Xu, J.; Yao, J.; Zeng, C.; Zhang, L.; Xu, N. Preparation of binderless honeycomb silicalite-1 monolith by using bundled palm fibers as template. *J. Porous Mater.* **2009**, *17*, 329–334. [CrossRef]
40. ASTM C1557-03. *Standard Test Methods for Tensile Strength and Young's Modulus of Fibers*; American Society for Testing and Analysis: West Conshohocken, PA, USA, 2003.
41. Petroudy, S.D. Physical and mechanical properties of natural fibers. In *Advanced High Strength Natural Fibre Composites in Construction*; Elsevier BV: Amsterdam, The Netherlands, 2017; pp. 59–83.
42. Indran, S.; Raj, R.E. Characterization of new natural cellulosic fiber from Cissus quadrangularis stem. *Carbohydr. Polym.* **2015**, *117*, 392–399. [CrossRef] [PubMed]