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Physical, Chemical, and Mechanical Characterization of Coated Date Palm Leaf Fiber from the Middle Region of Iraq for Potential in Civil Engineering Application

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ABSTRACT

Date palm leaf fibers are suitable for engineering applications due to their availability, inexpensiveness, and eco-friendliness. However, there is a risk of biodegradation in the long term. This paper explores date palm leaf fiber (DPL) properties and the protection of the fibers from biodegradation to enhance their lifespan. To this end, two coating materials (bitumen and polyurethane) were used separately. Physical and mechanical tests were conducted to determine the most effective material to coat the DPL fibers. To comprehensively assess the performance of the coated fibers, their morphology was examined via microstructure analysis using scanning electron microscopy (SEM) and energy dispersive X-ray analyses (EDX) tests. This analysis encompasses their material properties, chemical composition, water absorption, and degradation, providing a thorough understanding of the protective coatings' impact on the DPL fibers. The tensile strength test results revealed that the maximum tensile strength of the bitumen coated date palm fiber (DPLB) is 7.4 MPa. The tensile strength is two times greater than the polyurethane coated date palm leaf fiber (DPLP) and untreated date palm leaf fiber (UDPL). The results of the degradation test revealed that the weight loss percentage is equal to 45.5 and 25 in the case of the UDPL and DPLP fibers, and no loss in weight in the case of the DPLB fiber. Out of all the test results, bitumen is considered the best due to its ability to resist the attack of chlorides and sulfate ionspresent in groundwater on top of being cheap, simple, and efficient.

Keywords: Bitumen; degradation; geo-natural materials; SEM/EDX; tensile strength

INTRODUCTION

Natural fibers such as coir, jute, and sisal have been used in geotechnical engineering for ground improvement and soil reinforcement (Chakravarthy et al. 2021; Nguyen & Indraratna 2023; Prasad et al. 2023; de Carvalho et al. 2023; A. Rahman et al. 2023) due to several advantageous factors including their affordability, abundance, biodegradability, favorable mechanical properties, low density, flexibility during processing, and ability to create environmentally-friendly. (Kurniawan et al. 2013; Zahari et al. 2015; Abdel-Hakim & Mourad 2020; Abd Rahim et al. 2022; Naz et al. 2023). This concept shift towards natural fiber materials (Zubir et al. 2024), rather than synthetic ones, has recently gained popularity in many civil engineering applications such as a soil-reinforcement strategy in sustainable geotechnics and road applications (Lal et al. 2017; Gowthaman et al. 2018; Panigrahi & Pradhan 2019; Ghosh et al. 2019; Kolathayar et al. 2020; Shirazi et al. 2020; Kolathayar et al. 2021; Abood & Shakir 2023; Bujang et al. 2023). Natural fibers are fundamentally different from synthetic fibers whereby physical-mechanical and biochemical features control the behavior of fiber. The shift to natural fibers reflects a growing awareness of sustainable practices and an emphasis on environmentally-friendly solutions in various engineering applications. When buried in the ground, these fibers are anticipated to have a lifespan of 1–2 years (Nguyen & Indraratna 2023; Kolathayar et al. 2019). However, to extend the life span of these materials for civil engineering applications, they should be treated with an appropriate preservative solution to safeguard against factors like moisture, fungi, and insects that can lead to biodegradation of the geo-natural materials; for long-term applications, an alternative to commercially-supplied geosynthetics is to use naturally-accessible, low-cost geo-natural materials to protect against natural predators (Kolathayar et al. 2019; Vela Silveira et al. 2022; Kaya et al. 2023). This dual emphasis on natural fiber composites and the preservation of geo-natural materials underscores the importance of sustainable and resilient solutions in civil engineering practices. Several studies have explored coating and treatment to improve the properties of natural fibers. (Bateni et al. 2011) studied the effect of treating oil palm empty fruit bunches (OPEFB) fibers with acrylonitrile butadiene styrene by evaluating the physical and mechanical properties of the coated fibers as well as their morphological characterization. The results showed that coating fibers with acrylonitrile butadiene styrene reduces the biodegradability of the fibers interacting with soil and protects the fibers from water absorption. Meanwhile, the elasticity modulus and tensile strength of the OPEFB fibers are enhanced with the treatment. Coating fibers increase the friction between the soil particles and fibers due to increased surface area. The shear strength parameter of the soils reinforced with fibers can be considerably enhanced. (Shanmugasundaram et al. 2018) studied a new portion of the areca palm leaf and stalk fibers, which were treated with various percentages of alkali (NaOH), and their chemical, physical, and mechanical properties. The chemical analysis revealed that the cellulose content increases while the levels of lignin, hemicellulose, ash, and wax decrease. According to the physical results, the diameter of the fibers reduces, and the density increases. Additionally, Kafodya & Okonta (2018) studied the effect of covering the surface of sisal fiber with natural-based water repellent to prevent water absorption. The mechanical properties of soil reinforced with fiber were investigated. The outcomes indicated that shear strength at the interface of the uncoated fibers is higher than that of the coated fibers at the soil-fiber composite's optimum moisture content. This coating can efficiently inhibit the water absorption of natural fibers and improve the long-term strength of the natural fiber-soil composite.

Kolathayar et al. (2019) and (Torio-Kaimo et al. 2020) conducted studies exploring the impact of coatings on natural fibers in distinct contexts. Kolathayar focused on increasing the durability of areca leaf sheaths by chemically modifying them with Copper Chromium Arsenic (CCA) composition. The study revealed that coating the areca leaf sheaths with CCA results in extended durability, reduced biodegradation of the sheaths, and an overall enhancement in lifespan. Similarly, Torio-Kaimo investigated the coating of coir fibers with kerosene and its effect on cohesive soil's strength and stress-strain response. The findings demonstrated that kerosene effectively reduces coir's moisture absorption by up to 170%. In a related investigation, (Mahdi et al. 2021) explored the use of Khalasa date palm leaf fibers (KDPLF) as a polymeric composite material reinforcement (epoxy as matrix and Khalasa date palm fibers as reinforcement). To that end, the morphology, water absorption, material properties, and chemical composition must all be examined. The morphology examination was done using scanning electron microscopy (SEM). The test results of the KDPLF cross-section revealed a significant hemicellulose content. The KDPLF showed many variances in mechanical characteristics and morphology, according to the findings. The characteristics of the KDPLF/epoxy composite are considerably affected by water absorption. As such, the stiffness and strength of the composite diminish when the moisture absorption of the KDPLF/epoxy increases.

Fattah et al. (2021) aimed to produce local materials using reclaimed rubber, subjecting them to high temperatures and pressure. The focus was on creating geogrids, despite being made of less durable and resilient polymer material than standard geogrids. Various ratios of old and fresh reclaimed rubber were tested to produce geogrids with rectangular apertures as well as those with honeycombed openings. Tensile testing was carried out on the product and the results showed that both forms of geogrids, honeycombed and rectangular, increase the foundation bearing capacity when compared to unreinforced soil in all proportions (20%, 40%, 60%, and 80%). It was determined that the insertion of geogrid of the same footing size at depth B beneath the footing provides the greatest improvement in bearing capacity. These studies collectively highlight the efficacy of coatings in enhancing the performance and longevity of natural fibers, offering valuable insights for various applications, from agricultural residues to soil reinforcement strategies in civil engineering.

The present work aims to produce a type of plant fiber from date palm leaf and enhance the life of geo-natural materials for geotechnical engineering applications. In this study, we used date palm tree fiber from the middle region of Iraq, a country with abundant palm trees. After the extraction, to enhance the fibers for geotechnical applications, the date palm leaf (DPL) fibers were coated with two types of coating materials (bitumen and polyurethane) separately for the purpose of comparison. Scanning electron micrograph (SEM) and energy dispersive X-ray analyses (EDX) were performed, the tensile strength properties were characterized, and the water absorption was measured along with the degradation.

METHODOLOGY

DEVELOPMENT OF DPL MATS USED FOR CIVIL ENGINEERING APPLICATIONS

The date palm leaf fibers were extracted from the date palm tree. The fibers were cut into equal strips of about 10 mm in width and soaked in water for about 10-15 minutes. These strips were then woven into mats by hand. Figure 1 shows the process of making a mat out of date palm leaf fibers.

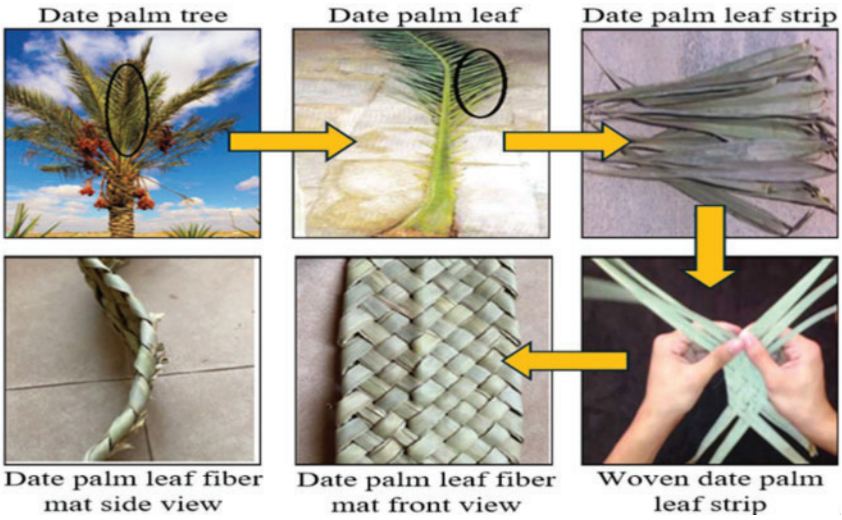


FIGURE 1. The process of making a date palm leaf fiber mat

COATING MATERIALS AND COATING METHOD

Natural fibers can be made more durable by chemically altering the hydroxyl groups in the cellulose polymer or applying various coatings to the fiber's surface. Due to their biodegradability, cellulose-based materials such as natural fibers must be protected from corrosive agents (Al-Hassnawi et al. 2024). A particular condition may be necessary for protection because of water absorption, soil minerals, and organisms. Since lignin and cellulose contain hydroxyl groups, natural fibers can be altered. Additionally, the chemical substance coating of the fibers decreases water absorption while protecting them from bacterial and fungal attacks. In previous studies, various materials used for coating plant fibers were identified. Therefore, the selection of coating material is subject to quality and cost standards. In the current study, two types of coated materials were chosen for the purpose of comparison, one being low-cost (bitumen) and the other high-cost (polyurethane). Several tests were conducted, and the results were compared to determine the optimal coating material. The performance of these coating materials is mentioned in the results.

BITUMEN

Bitumen, a viscous substance that must be extracted from an oil reservoir, is a low-grade crude oil with a complex, heavy hydrocarbon composition (Rhasbudin Shah et al. 2023). Bitumen is difficult to extract from the ground. However, it can naturally rise to the earth's surface in petroleum seeps, where fossil fuels and petroleum products leak out of the ground rather than being contained underground. Moreover, bitumen is oil sand's most

abundant fossil fuel component (Raymond & Leffler 2005). The bitumen utilized in this study is known as bituproof, which has a viscosity of 40 g/ltr. Bituproof 40S is a black/brown emulsified cold-sprayed bitumen liquid that acts as a damp-proof and vapor barrier for concrete and masonry and a general-purpose primer for bituminous membranes. After weaving the date palm leaves as previously mentioned, the painting process using bitumen material was carried out after ensuring the thorough homogenization of the bitumen material through careful stirring with a wooden stick. Subsequently, the painting was done using a brush, covering all surfaces and edges thoroughly. Then, it was left to dry in a well-ventilated area for a minimum of 24 hours.

POLYURETHANE

Diisocyanates (TDI and MDI) and polyols are used to create polyurethanes, which are polymeric polymers. Polyurethanes come in many forms, and each is manufactured somewhat differently. Platine clear polyurethane gloss-057ME is an extremely durable two-component polyurethane varnish with a high gloss that was employed in this investigation. In this study, the polyurethane material was diluted by 10 percent, by mixing the diluted substance with the polyurethane material. The mixture was thoroughly stirred using a glass rod and then applied with a brush, ensuring complete distribution on all sides of the palm leaves. It was then left to dry for a minimum of 24 hours.

THE SOIL USED FOR DEGRADABILITY TEST

The clay soil was taken from north Iraq for use in the degradability investigation reported in this paper. The soils' geotechnical properties were evaluated per ASTM standards, and the results are shown in Table 1. The following qualities of the experimental soil were evaluated:

1. Optimum moisture content (OMC) and maximum dry density (MDD) from Standard Proctor Test
2. Grain size distribution.
3. Soil classification according to the Unified Soil Classification System (USCS), including Atterberg's limits.

The pH of the soil was also determined using the method given in the associated literature (Kalra et al. 1995).

TABLE 1. The physical properties of the soil

Soil property	Value
Liquid limit %	42
Plastic limit %	22
Plasticity index %	20
Soil classification according to the USCS	CL
OMC%	23.90
MDD (g/cm^3)	1.680

SEM AND EDX TESTS

Using an S50 scanning electron microscope, the fiber surfaces and cross-sections of the coated and uncoated date palm leaf fibers were examined. The morphologies of the coated and uncoated date palm leaf fibers were observed. Energy dispersive X-ray analysis (EDX) was used to analyze the chemical reactions for the coated and uncoated fiber.

TENSILE STRENGTH AND RESIDUAL TENSILE STRENGTH

The mechanical properties of the coated and untreated date palm leaf fibers were evaluated using a tensile machine. To compare the effect of coating on the tensile strength of the fibers, the tensile strength and residual tensile strength were measured after aging for varying amounts of time. The tensile testing machine was used at a 5 mm/min rate. Tensile strength testing was carried out on the treated and untreated DPL fiber in accordance with ASTM D6637-01. A sample of treated and untreated woven DPL fiber at 40 cm length, 5 cm width, and 2 mm thickness was tested to determine the maximum tensile strength. Similar samples were placed in an airtight box filled with moist soil with optimum moisture content for three (3) months to determine the residual or loss in fiber tensile strength. DPL fibers were placed 10 cm from the surface of the soil. This method was mentioned by (Carvalho et al. 2015) and (Ghosh et al. 2019).

WATER ABSORPTION

The water absorption test procedure was performed as per ASTM D 570-98 (2018). Samples with dimensions of (20x20x1 mm) were dried at room temperature for 48 hours and weighed (wd), and then immersed in distilled water for 80 days, with the weight of swollen samples (ws) determined at various time (t) intervals. The percentage of water absorption at various time intervals was calculated.

DEGRADATION TEST

The degradation of the treated natural fibers was measured following (Dalev et al. 2001) and (Abdel-Hakim & Mourad 2020). The same clay soil used in the experiment was poured into a plastic container in a 4 cm layer. Weighed samples with dimensions of (20x10x1 mm) were buried in a layer of 1 cm thick soil. The thick paper was used to cover the plastic cup. The cover fabric was consistently wetted with distilled water in order to keep the moisture in it. The weight of the studied samples was measured weekly for nearly three months, during which time the samples were taken out, cleaned with distilled water from the soil, and dried for two hours at room temperature.

RESULTS AND DISCUSSION

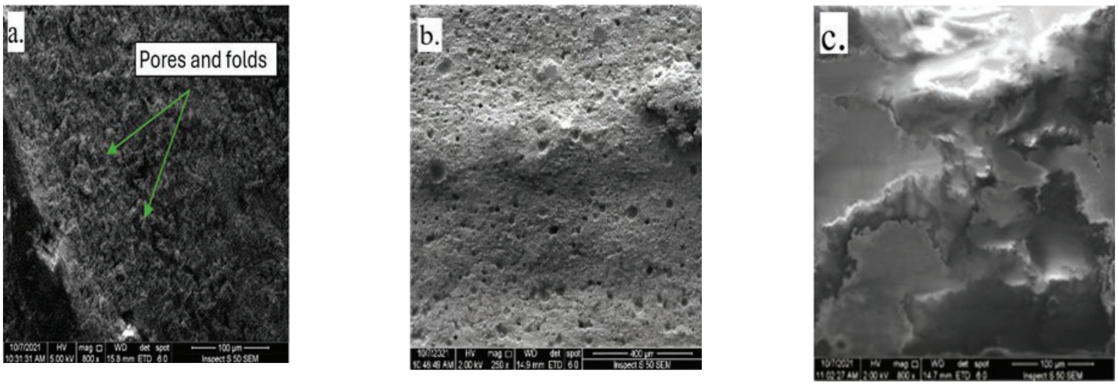
FIBER SURFACE TOPOLOGY SEM

Scanning electron micrographs (SEM) and energy dispersive X-ray analyses (EDX) were performed to evaluate the treatment's performance on the treated and untreated date palm leaf fiber materials.

SEM image of the surface structure of the untreated DPL fiber is presented in Figure 2a, whilst the images for the treated DPL fiber with bitumen and polyurethane are presented in Figures 2b and 2c, respectively. DPL fiber, like natural fibers derived from plants, consists mainly of cellulose, hemicellulose, pectin, lignin, and wax. The lignin and wax content of the DPL were visible prior to treatment and not visible after treatment. This result is consistent with that of previous studies (Saadaoui et al. 2013; Swain et al. 2018; Asim et al. 2021). As stated in the literature, the voids are present in DPL fiber, indicating a certain degree of porosity.

In contrast, the porous surface was helpful for better mechanical fiber interlock with the coating materials. It is well-known that porosity promotes higher moisture absorption. Uniform covering and full coating of the fiber surface are important in protecting the fiber from degradation, water, and physical damage. The comparison between the topology of the untreated and treated DPL fibers showed a coating layer on the surface of all the fiber surface pores and folds. The surface of the coated fiber appears smoother than the surface of the uncoated fiber and potentially increases the interface area between the soil particles and fiber. These results agree with those of (Mahdi et al. 2021) and (Bateni et al. 2011).

Figure 2d presents an SEM of the cross-section of an uncoated DPL fiber, which shows the thickness of the uncoated DPL fiber. Figures 2e and 2f are the SEM images of the cross-section of the coated (DPL) fiber with bitumen and polyurethane, respectively. The thickness of the coated layer can be seen, and the fiber's cross-section in the coated state is less than in the uncoated state. This indicates that the coating layer penetrated the fiber structure and filled the grooves and folds, showing strong interfacial bonding between the fiber and the coating materials. These results agree with those of (Swain et al. 2018). Figures 2e and 2f indicate that the coating materials increased the coated fiber's section area, as compared to the uncoated DPL fiber cross-section, where the average thickness of the uncoated DPL equals 165 µm as mentioned in Figure 2d, while the average thickness of the DPLB and DPLP are 379.62 µm and 446.95 µm, respectively.



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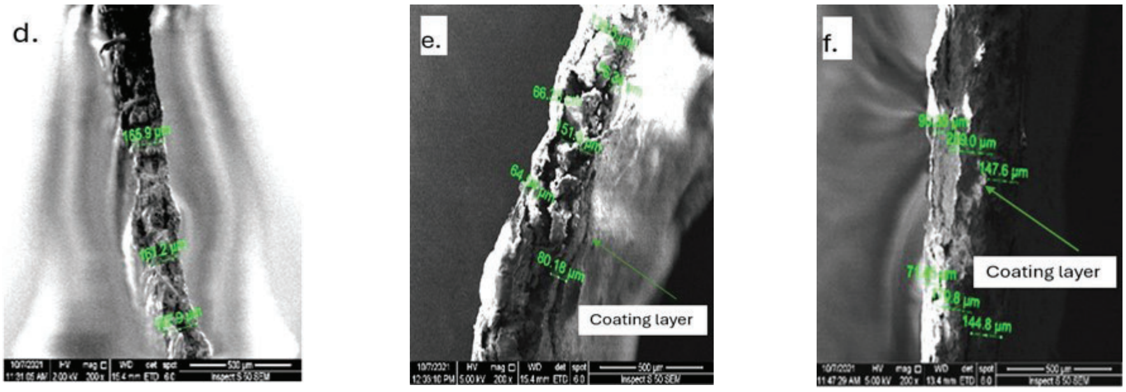
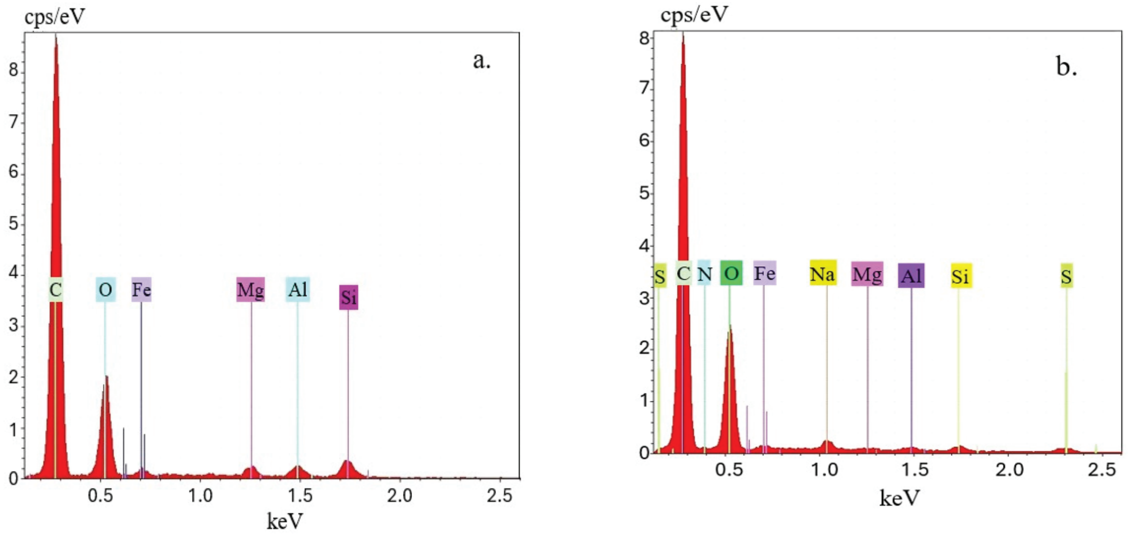


FIGURE 2. SEM surface structure of a) UDPL fiber, b) DPLB fiber, c) DPLP fiber, and SEM cross-section of d) UDPL fiber, e) DPLB fiber, f) DPLP fiber.

EDX

The chemical reactions during fiber coating were characterized using energy dispersive X-ray analysis (EDX) on the untreated and treated DPL fibers. The fibers consist of carbon (C), oxygen (O), and other materials in small amounts, such as sodium (Na), aluminum (AL), silica (Si), potassium (K), sulfur (S), calcium (Ca), and chlorine (Cl). The behavior of the different lignocellulosic fibers is compared to the theoretical atomic ratio between oxygen to carbon ratio (O/C ratio), which is 0.83 for pure cellulose, 0.33 for lignin, and 0.1 for extractive.

Figure 3 and Table 2 summarize the results of the EDX test. The O/C ratio measured for untreated is 0.215, and treated fibers with bitumen and polyurethane is 0.802 and 0.03, respectively. From these results, it is possible to indicate an increase in the O/C ratio between the untreated and bitumen treatment, which proves the effectiveness of the bitumen treatment in removing impurities. In contrast, the O/C ratio decreases between the untreated and polyurethane treatment, indicating the ineffectiveness of using polyurethane treatment on date palm leaf fibers. From the test findings, it is easy to see that the treated fiber's silica concentration decreases when compared to the untreated fiber. This decline is caused by the elimination of lignin from the fiber's surface, which reduces the silica bodies' ability to attach to the fiber's holes (silica cavities). The results reveal that the durability treatment procedure used is very effective for this type of material, which agrees with Bezazi et al. (2022) and Daud et al. (2013).



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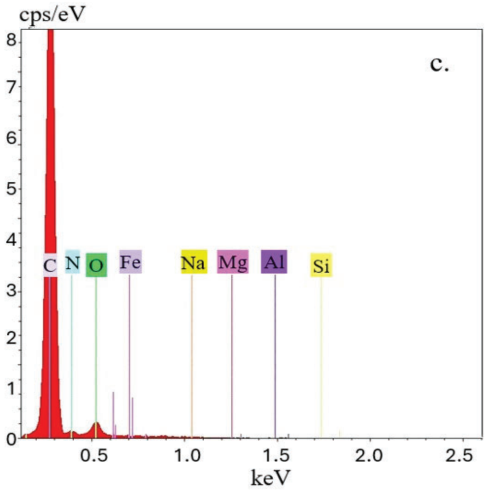


FIGURE 3. EDX results for a) untreated DPL fiber, b) DPL fiber treated with bitumen, c) DPL fiber treated with polyurethane.

TABLE 2. Chemical composition of treated and untreated DPL fiber

Element	Atom. C (wt.%)		
	Untreated DPL fiber	DPL fiber treated with bitumen	DPL fiber treated with polyurethane
Oxygen	16.73	8.88	0.16
Carbon	77.77	11.07	5.32
Silica	3.04	0.73	-
Iron	1.02	0.44	-
Aluminum	0.80	0.24	-
Magnesium	0.64	0.14	-
Hydrogen	-	77.46	94.43
Sodium	-	0.45	-
Nitrogen	-	0.50	0.09
Sulphur	-	0.09	-

TENSILE STRENGTH OF FIBERS

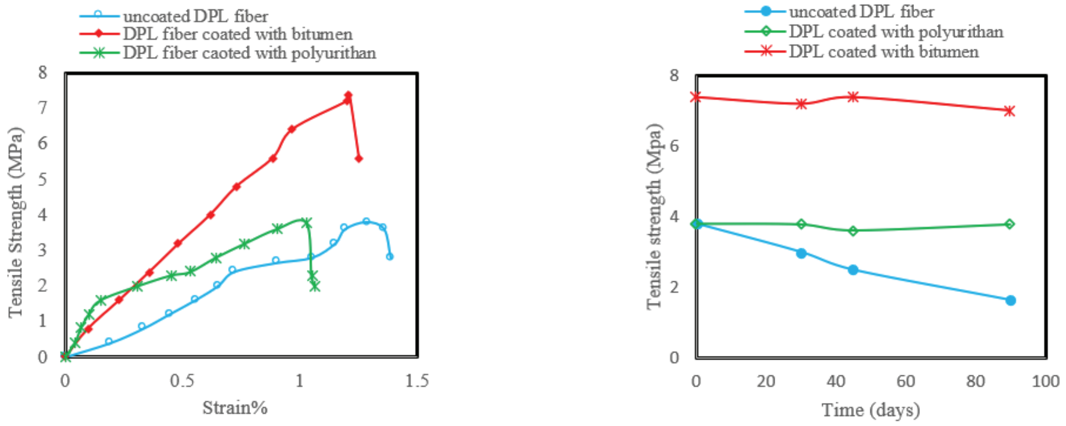
Figure 4a shows the tensile stress-strain behavior of each untreated and treated DPL fiber before aging (before degradation). The test results indicate that the bitumen-coated DPL fiber exhibits a maximum tensile strength of 7.4 MPa, which is twice as high as the tensile strength of both the polyurethane-coated DPL fiber and the untreated DPL fiber, both of which have a tensile strength of 3.8 MPa. It is worth noting that there is no significant improvement in tensile strength when comparing the untreated and polyurethane-coated DPL fiber.

The coated DPL fiber's strain and tensile strength were improved by the bitumen-filled date palm fibers acting as a unit fiber. The coated materials which cover the fibers were broken before the failure of the DPL fiber. This behavior was explained by a decrease in strain value in the case of the coated fiber compared to the uncoated fiber. The coating breaks clearly show the difference in strain between the DPL fiber and coated materials. This weakening has little effect on the strength of the fiber-reinforced soil due to the slight stress in the soil sample. These results are consistent with that of Bateni et al. (2011) and Biswas et al. (2014).

Figure 4b shows the results from the tensile strength test performed on the untreated and treated DPL fibers measured at different intervals after exposure to degraded soil. The results show a loss in the tensile strength value of the untreated DPL fibers. On the other hand, no loss in tensile strength was observed for the DPL fibers treated with bitumen or polyurethane over the time intervals for degradation. This can be explained by the ability of the coated materials in protecting the fibers from the surrounding circumstances.

WATER ABSORPTION

The water absorption of the treated and untreated DPL fibers is shown in Figure 5. The results clearly show that the water absorbed by the DPL fiber increased to 1.8% as the time interval increased to 40 days. This might be attributed to the DPL fiber's propensity to absorb water. After 40 days, it was noted that the percentage of water absorbed decreased to 1.2%, which could be explained by the growth of bacteria and fungi that feed on the DPL fiber thus leading to weight loss (Crawford et al., 2017). The results show that the water absorption of the treated fiber is lower than that of the untreated fiber. The bitumen coating method reduces the water absorption more than the polyurethane coating. This could be explained by the ability of the coating layer to reduce the amount of water absorbed by the DPL fiber. In the case of the DPLP, the percentage of water absorption is 0.4%, whilst the DPLB shows a 0.19% water absorption.



a.

b.

FIGURE 4. a) Tensile stress vs. strain behavior, b) Tensile strength loss over time aging for untreated, treated DPL fibers

DEGRADATION

After three (3) months of aging in the chosen soil and exposure to moisture and fungi, the degradable characteristics of the treated and untreated DPL fiber were evaluated. Figure 6 demonstrates the effect of fungus on fiber degradation; the fungus only affected the dark section of the fiber. In comparison, the treated fiber with polyurethane is less affected by fungus. Otherwise, no fungi effect is observed in the treated fiber with bitumen.

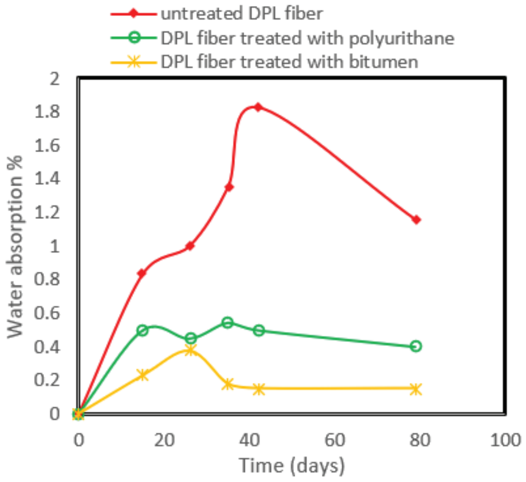


FIGURE 5. Percentage of water absorption for treated and untreated DPL fiber in distilled water with time

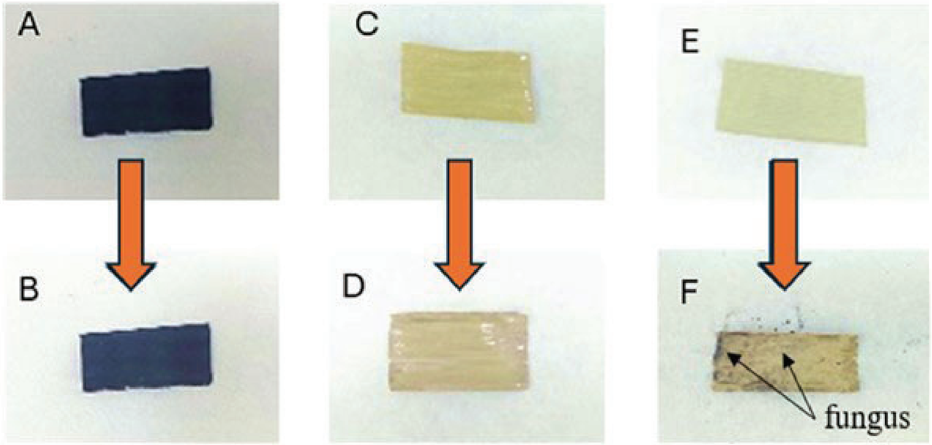


FIGURE 6. Date palm leaf fiber a) coated with bitumen before aging, b) coated with bitumen after 3 months of aging, c) coated with polyurethane before aging, d) coated with polyurethane after 3 months of aging, e) uncoated fiber before aging, f) uncoated fiber after 3 months of aging.

The results of the weight loss are graphically plotted in Figure 7. The percentage of weight loss is equal to 45.5 and 25 in the case of the UDPL and DPLP fiber, while there is no weight loss in the case of the DPLB fiber. The results show that the degradation of the date palm leaf fiber decreases in all conditions treated, with the percentage of weight loss decreasing by approximately 20% in the case of the DPLP and approximately 45% in the case of the DPLD. The weight loss results show that the coating affects the DPL fiber's protection from degradation.

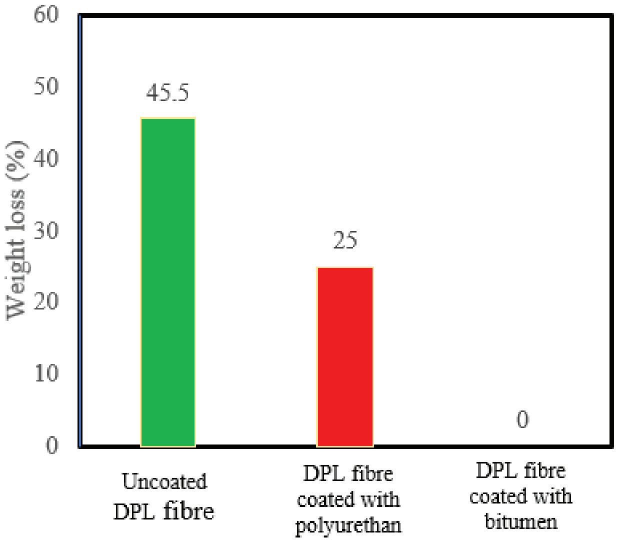


FIGURE 7. Degradation of the uncoated DPL fibers, DPL coated with polyurethane, and DPL coated with bitumen after 90 days.

CONCLUSION

This paper introduces date palm leaf fiber (DPL) as a geo-natural material. From the test results, the following conclusions could be obtained:

1. From the scanning electron micrograph (SEM), the comparison between the topology of the untreated and treated DPL fibers clearly revealed that there was a coating layer on the surface of the fiber covering all the surface pores and folds. The surface of the coated fiber appears smoother than the surface of the uncoated fiber. The DPLB appears to have some impermeable gaps, which increases the interface area between the soil particles and fiber. From the SEM results, the cross-section of the coated DPL fiber is

less than the uncoated DPL fiber, indicating that the coating layer's penetration into the fiber structure filled the groove and folds.

2. The material elements before and after fiber coating were characterized using energy dispersive X-ray analysis (EDX) on both the UDPL and DPL fibers. The EDX test results revealed that the durability treatment procedure is very effective for this type of material. Treated DPL fiber helps eliminate lignin from the fiber's surface, reducing the silica bodies' ability to attach the fiber holes. The bitumen-coated fiber (DPLB) was more durable than the polyurethane coating fiber (DPLP).
3. The tensile strength test results revealed that the maximum tensile strength of the DPLB fiber is 7.8 MPa which is two times greater than that of the DPLP fiber and UDPL fiber.
4. The findings of the water absorption test demonstrated that the treated fiber has lower water absorption than the untreated fiber. The bitumen coating approach was discovered to limit water absorption more than the polyurethane coating method.
5. After three (3) months of aging in the specified soil and exposure to moisture and fungi, the degradable properties of the treated and untreated DPL fiber were evaluated. The percentage of weight loss is equal to 45.5 and 25 in the case of the UDPL and DPLP fibers, respectively, while there is no weight loss in the case of the DPLB fiber. In comparison, the polyurethane-treated fiber was less impacted by fungus. Otherwise, no fungal effect was found in the bitumen-treated fiber.

ACKNOWLEDGEMENT

We would like to thank everyone who has contributed to this work.

DECLARATION OF COMPETING INTEREST

None.

DATA AVAILABILTY STATEMENT

The submitted article includes all data and models developed or used during the study.

Notation

In this paper, the following symbols are used:

DPL = date palm leaf

DPLB = bitumen coating date palm

DPLP = polyurethane coating date palm leaf fiber

UDPL = untreated date palm leaf

Ws = weight of swollen samples

Wd = weight of dried samples

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