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Citric acid treatment: A new approach to improving *Ampelodesma mauritanica* fibers-poly-lactic acid composites

Nadia Kharchi^a, Brahim Barka^b, Farid Rouabah^a, Abdel-Hamid I. Mourad^{c,d,*},
Deradji Dadache^e, Fois Magali^f, Haroun Houicha^g, Abdeslam Bencid^h

^a Université Ferhat Abbas Setif-1, Faculté de Technologie, Laboratoire de Physico-Chimie des Hauts Polymères (LPCHP), Setif 19000, Algeria

^b Université Ferhat Abbas Setif-1, Faculté de Technologie, Unité de Recherche des Matériaux Émergents, Setif 19000, Algeria

^c Department of Mechanical and Aerospace Engineering, College of Engineering, United Arab Emirates University, Al Ain 15551, United Arab Emirates

^d National Water and Energy Centre, United Arab Emirates University, Al Ain 15551, United Arab Emirates

^e Université Mohamed El Bachir El Ibrahimi de Bordj Bou Arreridj, Faculté des Science et de la Technologie, Bordj Bou Arreridj 34000, Algeria

^f Université Paris-Est Créteil Val de Marne, Créteil Cedex, Centre d'études et de recherche en thermique, environnement, et systèmes (CERTES), Créteil 94000, France

^g Université of Biskra, BP 145, Biskra, Laboratoire Génie Mécanique (LGM), Biskra 07000, Algeria

^h Research Center in Industrial Technologies CRTI, P.O. Box 64, Cheraga Algiers16014, Algeria

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ABSTRACT

This research investigated the impact of citric acid treatment on the adhesive properties of a composite material based on a poly-lactic acid matrix and *Ampelodesma mauritanica* fibers. Citric acid, a natural and biodegradable compound, was chosen as a more environmentally friendly alternative to the alkaline treatments traditionally used. Using a palatograph, biocomposite samples with a weight percentage of 20 % fiber were produced. Characterization was carried out using different methods such as Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), thermogravimetric analysis (TGA) and scanning electron microscopy (SEM) techniques, were used to examine the treated and untreated fiber samples. Citric acid treatment led to a reduction in the quantities of hemicellulose, lignin and wax. This observation is confirmed by the disappearance or attenuation of absorption bands at 1650 and 1731 cm⁻¹ in the spectra. In addition, the treatments improved crystallinity, thermal stability and surface roughness of Diss (*Ampelodesma mauritanica*) fibers. Furthermore, Poly-lactic acid composites reinforced with medium-length Diss fibers were subjected to mechanical tests (Izod impact strength, tensile strength and elongation at reapture), were used to characterize Poly-lactic acid composites reinforced with natural Diss (*Ampelodesma mauritanica*) fibers. In contrast with untreated composites, the mechanical properties of treated composites showed an increase in the modulus of elasticity, tensile strength, and elongation at reapture, with values of 4.680 GPa, 48.90 MPa, and 2.52 %, respectively. Scanning electron microscopy assessments of treated composites also confirmed these findings.

1. Introduction

The expression "biocomposites" denotes biopolymers reinforced with fibers of natural origin, also known as 'natural-fiber-based biocomposites' (Gholampour and Ozbakkaloglu, 2020).

These biocomposite materials are increasingly in demand for their environmental benefits and biodegradability (Siddiqui et al., 2024). This translates into a smaller environmental footprint than traditional materials, making them ideal for use in a variety of sectors (Zwawi, 2021).

PLA was chosen as the base material for this project, a type of

biodegradable plastic considered to be the most commercially viable option (Yussuf et al., 2010), is derived from sustainable resources such as corn starch or sugar cane and can be broken down by micro-organisms into water and carbon dioxide under industrial composting conditions (Trivedi et al., 2023).

Research is shifting from polymer composites based on synthetic fibers to biocomposites based on natural fibers (Challabi et al., 2019). Natural fibers are making significant inroads into composite reinforcement, replacing synthetic fibers (Werchefani et al., 2020). Natural fibers have many advantages, including being environmentally friendly,

* Corresponding author at: Department of Mechanical and Aerospace Engineering, College of Engineering, United Arab Emirates University, Al Ain 15551, United Arab Emirates.

E-mail address: ahmourad@uaeu.ac.ae (A.-H.I. Mourad).

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renewable, inexpensive, non-toxic and highly resistant (Christian, 2016). Composites based on *Ampelodesma mauritanica* have been improved by surface treatments that strengthen the interaction between *Ampelodesma mauritanica* fibers and poly-lactic acid matrix, resulting in composite materials that are stronger and perform better.

Recently, current research has focused on biodegradable polymer matrix composites reinforced with natural fibers: (Qian et al., 2013) conducted a study on the development of PLA matrix, while (Pujar and Mani, 2022) looked at the characterization of pigeon pea fibers reinforced with poly-lactic acid. of composites based on fibers of natural origin for applications. This work by researchers in composite materials illustrates the diversity of approaches being explored to develop sustainable, high-performance materials from renewable resources. Composites based on fibers of natural origin offer considerable potential for a wide range of applications, from the automotive industry to building materials and packaging, thus contributing to the development of sustainable and environmentally friendly products.

The enhancement of the adhesive properties of Diss fibers (*Ampelodesma mauritanica*) with citric acid treatment opens up new avenues for the application of these composites in a variety of industries, including automotive, packaging, and construction.

These bio-composites, with improved mechanical and thermal properties, offer a competitive alternative to traditional synthetic fiber composites, pushing forward the potential for sustainable materials in mass production.

Studies by of (Nouri et al., 2021) and (Touati et al., 2020) have highlighted that chemical treatments applied to *Ampelodesma mauritanica* fibers significantly improve their compatibility with polymers, promoting better adhesion to the matrix.

These studies mainly involved treatments with acetic acid, alkaline solutions and silane agents, while (Sarasini et al., 2018) treated Diss (*Ampelodesma mauritanica*) fibers using enzymes. In this study, we developed PLA matrix composites reinforced with *Ampelodesma mauritanica* fibers. To optimise the interaction between the fibers of *Ampelodesma mauritanica*, a herbaceous plant also known as “Diss” in Arabic and belonging to the Poaceae family, and the polymer matrix, we treated the fibers with a 5 % aqueous citric acid solution. This environmentally friendly treatment was chosen to replace sodium hydroxide, a more aggressive alkaline product. Our goal is to develop a new composite material based on Diss (*Ampelodesma mauritanica*)/PLA fibers with improved properties using a citric acid treatment to improve the interfacial compatibility between the two constituent phases of the composite using citric acid treatment to improve the interfacial compatibility between the two constituent phases of the composite. The citric acid treatment is a more sustainable alternative compared with traditional chemical treatments (e.g., alkali, silane). Being biodegradable and non-toxic, citric acid aligns with the overall goal of producing eco-friendly, green composites. This approach reduces environmental impact and aligns with the increasing global push towards using natural and biodegradable materials in industrial applications.

2. Materials

The biopolymer used in this study is a polylactic acid thermoplastic resin, 2003D. Its melting temperature is around 210°C, with a melt flow index (MFI) of 6 g/10 min at 210°C. Its density is 1.24 g/cm³.

The fibers of *Ampelodesma mauritanica*, a herbaceous plant also known in Arabic as “Diss” and belonging to the poaceae family, were collected in Bouandas, north of Sétif in Algeria.

The Diss stems were cleaned to remove traces of soil and other plant impurities, then rinsed thoroughly with water.

After drying in the open air, the stems were cut and then ground using a grinder. The crushed material was then sieved to obtain a particle size of between 150 and 250 µm.

2.1. Citric acid modification of *Ampelodesma mauritanica* fiber

Chemical treatment using acidification solutions was carried out to selectively remove lignin, hemicellulose and other residues from the fiber surface, thus improving adhesion with the PLA matrix. The *Ampelodesma mauritanica* fibers were treated with (5 %) of citric acid solution at room temperature for 2 h.

Furthermore, they were then carefully rinsed with distilled water to remove any acid residue. After complete drying in an oven at 60°C for 24 hours, the fibers were ready for use.

2.2. Bleaching agent

The bleaching as a complement to chemical treatment process improves whiteness, chemical reactivity and aesthetic appeal.

It also removes unwanted coloration from plant fibers, as well as removing microorganisms from the fibers. In this study, (5 %) of NaClO was used as the bleaching agent.

The experimental procedure involved immersing 1 g of *Ampelodesma mauritanica* fiber treated with 5 % citric acid in 50 ml of an aqueous sodium hypochlorite solution at 5 % concentration. The mixture was heated at 80°C for 2 hours with stirring (Safina Aridi et al., n.d). The fiber was then washed thoroughly with distilled water until the filtrate was neutral, and dried in an oven at 60°C for 24 hours.

2.3. Biocomposites preparation

To manufacture the biocomposites, polylactic acid (PLA) and *Ampelodesma mauritanica* fibers were completely dehydrated by vacuum drying for 24 hours at 60°C. Biocomposites were prepared by melting using a plastograph internal mixer at 190°C and 50 rpm for 8 minutes, following the method described by (Challabi et al., 2019). The melt blending can ensure even distribution of fibers in the PLA matrix after melt mixing, The fibers were then extracted from the counter-rotating screw and cooled before being ground. The compound was dried at 60°C for 24 hours to remove all traces of moisture. The biocomposite sheets were prepared using a hydraulic hot-press at 190°C.

The compression molding procedure involves preheating the compound material for 4 minutes, it was used to produce sheets with thicknesses ranging from 1 mm to 2 mm, depending on the desired application. The sheets were subjected to characterization after recovery.

Studies have shown that a fiber content of around (20 %) to (30 %) by weight provides the best balance between improved mechanical properties and ease of processing (Komal et al., 2020).

2.4. Characterization techniques

2.4.1. Fourier transform infrared spectroscopy

Fourier Transform Infrared (FTIR) spectra, acquired on a Spirit SHIMADZU IR spectrometer in the spectral region of 4000–400 cm⁻¹, were used to identify the characteristic functional groups of untreated UTDF and treated TDF fibers.

2.4.2. X-ray diffraction analysis

The EMPYREAN diffractometer was used to study the crystallinity of untreated and treated Diss fibers by X-ray diffraction, the analysis used X-rays obtained by Cu K α with a wavelength of 0.15406 nm. X-ray diffraction analysis was performed using X-rays generated at 40 kV.

Table 1
Chemical composition of *Ampelodesma mauritanica* fibers.

Samples	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Diss	45	26.7	16
(Bourahli and Osmani, 2013)	44.1	27	16.80

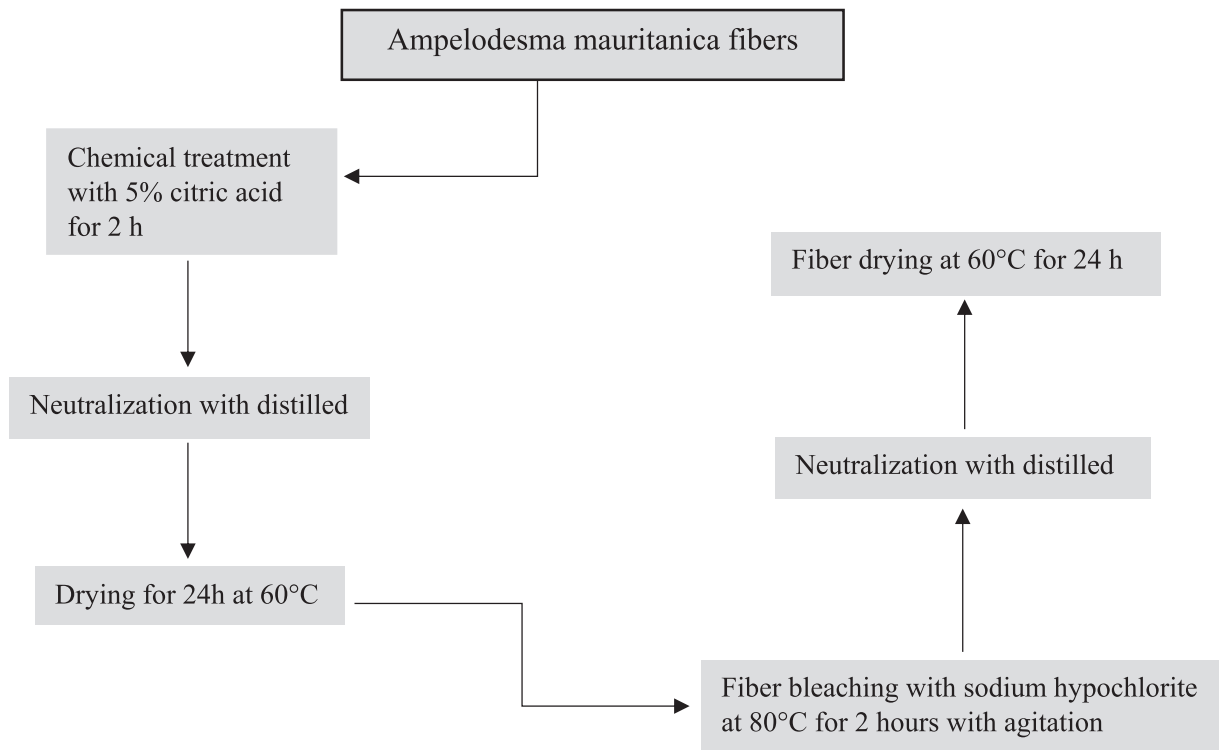


Fig. 1. flowchart of the Diss chemical treatment process.

Scans were measured over a range of angles 2θ .

Crystallinity Index (X_C) is often determined using the Segal method tailored to cellulose-based materials (Segal et al., 1952).

It is a quantitative measure of the relative amount of crystalline material in the fibers compared with the total material, including both crystalline and amorphous phases.

$$X_C(\%) = \frac{I_{002} + I_{am}}{I_{002}} \times 100 \quad (1)$$

Where:

I₀₀₂: The diffraction peak has its maximum intensity at an angle between 22° and 23° .

I_{am}: The scattering of the amorphous material was measured at a 2θ angle ranging from 18° to 19° (Bourahli and Osmani, 2013).

2.4.3. Thermogravimetric analysis

Untreated UTDF and treated TDF Diss (*Ampelodesma mauritanica*) fibers were analyzed by Thermogravimetric analysis (model Q600) to determine their thermal stability. Samples were heated from room temperature to 600°C under nitrogen at a constant rate of $10^\circ\text{C}/\text{min}$.

2.4.4. Differential scanning calorimetry

Differential scanning calorimetry analysis was conducted using a DSC model 2100 differential scanning calorimeter. A 10 mg sample was weighed, sealed, and subjected to all cycles at a heating and cooling rate of $10^\circ\text{C}/\text{min}$. Nitrogen was used as the purge gas at a flow rate of 50 ml/min. The degree of crystallinity was determined using Eq. (2).

$$X_C(\%) = \frac{\Delta H_m - \Delta H_{cc}}{\Delta H_m^\circ \times W_p} \times 100 \quad (2)$$

ΔH_m means enthalpy of fusion of the sample, ΔH_{cc} represents the heat of cold crystallization, ΔH_m° the fusion enthalpy for an ideal 100 % crystalline PLA, to be 93 J/g and W_p means the proportion of PLA by weight (Wang et al., 2018).

2.4.5. Scanning electron microscopy

Scanning electron micrographs of untreated fiber /PLA and treated /PLA composites samples were examined using a scanning electron microscope (SEM) of the type (Quanta 650-filament tungsten) with accelerating voltages of 10 kV.

2.4.6. Mechanical properties

2.4.6.1. Izod impact testing Pure PLA, UTDF/PLA and TDF/PLA materials were characterized by their impact resistance. Pure PLA, UTDF/PLA and TDF/PLA materials were evaluated using an unnotched Izod impact test. This test was performed using a JINGMI machine fitted with a 7.5 J pendulum.

The analyses were conducted at room temperature, utilizing five specimens with dimensions of $63.5\text{ mm} \times 12.7\text{ mm} \times 3\text{ mm}$.

2.4.6.2. Tensile testing. The specimen's tensile strength underwent testing following ASTM D638-1 standards, employing a Zwick Roell 7100 universal testing machine. Testing was conducted at a rate of 5 mm/min, with five specimens tested and their results averaged to guarantee result authenticity. The tensile tests were performed under quasi-static conditions at room temperature, around 20°C .

2.4.7. The water absorption

The water absorption test was carried out on PLA/UTDF and PLA/TDF samples of dimensions $(10 \times 10 \times 1)\text{ mm}^3$ in accordance with ASTM D570. After drying at 60°C for 24 hours and initial weighing (W_0), these samples were immersed in distilled water at room temperature 25°C for 24 hours. Following careful wiping, water absorption was calculated from the mass gained ($W_{24\text{ h}}$), using Eq. (3).

$$\text{Water absorption}(\%) = \left(\frac{W_{24} - W_0}{W_0} \right) \times 100 \quad (3)$$

3. Results

3.1. Characterization of UTDF and TDF

3.1.1. Fourier transform infrared spectroscopy

A comparison of the IR spectra of the fibers before and after the citric acid treatment is illustrated in Fig. 2. Fourier transform infrared spectroscopy (FTIR) was employed to analyze the functional groups present in both untreated and treated fibers.

The majority of observed peaks were attributed to various linkages within the cellulose structure, the principal component of Diss (*Ampelodesma mauritanica*) fiber. A prominent peak at 890 cm^{-1} in the spectrum indicated cellulose-like linkages between sugar molecules (Luzi et al., 2019). Additionally, a peak at 1030 cm^{-1} suggested the contribution of C-O stretching modes of hydroxyl groups and ether groups, while a peak at 1159 cm^{-1} corresponded to the C-O-C bond stretching vibration within cellulose glycosidic linkages (Bourahli and Osmani, 2013). The 1320 cm^{-1} peak was associated with alcoholic cellulose groups, and another band around 1420 cm^{-1} was linked to -CH₂-bending vibrations in cellulose.

A low absorbance peak at 1650 cm^{-1} in proximity to this region indicated the presence of lignin (Bourahli and Osmani, 2013). Furthermore, a peak at 1731 cm^{-1} signified the stretching vibrations of C=O bonds, potentially originating from various functional groups such as those found in lignin, hemicellulose, and pectin (Nouri et al., 2021).

Peaks within the $2857\text{--}2920\text{ cm}^{-1}$ range were associated with aliphatic saturated C-H stretching vibrations in cellulose.

In the 3334.72 cm^{-1} region, a band was observed primarily bound to O-H linkages from hydroxyl groups, while the $3609\text{--}3004\text{ cm}^{-1}$ band represented OH groups and hydrogen bonds of cellulose intramolecular and internetwork.

Significant changes were noted upon comparing the spectra of untreated and treated fibers. Firstly, the intensity of the 3334.72 cm^{-1} band decreased after citric acid treatment, indicating a reduction in hydroxyl groups (Céline et al., 2014). Secondly, treatment with a 5% citric acid solution decreased peak intensity at 2920 cm^{-1} and 2856 cm^{-1} , associated with the presence of lignin, pectin, and hemicellulose.

The reduction in intensity at 1650 cm^{-1} and 1731 cm^{-1} was also attributed to the degradation of lignin content after citric acid treatment. Citric acid, a natural, eco-friendly organic acid, acts as a coupling agent by introducing carboxyl groups (-COOH) on the fiber surface. These groups can form ester bonds with the hydroxyl (-OH) groups of the PLA matrix during the composite's processing. This chemical interaction between the fiber and matrix increases adhesion at the interface, which is often the weakest link in natural fiber composites.

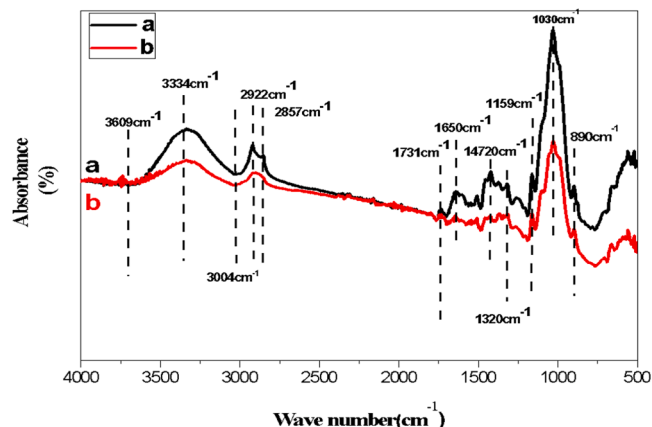


Fig. 2. FTIR spectra of Diss fibers:(a) untreated, (b) treated with C₆ H₈ O₂.

3.1.2. X-ray diffraction (XRD)

X-ray diffraction (XRD) analysis is a crucial method for evaluating the crystalline structure of both untreated and treated Diss fibers (*Ampelodesma mauritanica*), particularly following treatment with citric acid. This analysis provides valuable insights into changes in crystallinity and structural properties induced by the chemical treatment.

The results of X-ray crystallography analysis for untreated and treated Diss (*Ampelodesma mauritanica*) fibers are shown in Fig. 3.

The diffractogram of UTDF, TDF reveals three prominent peaks at 16.19° , 22.08° and 34.80° , indicating a native crystalline structure like that of cellulose I as observed by (Zergane et al., 2020). Associated with the lattice planes of peak (110), (200) and (004) respectively treated fibers often display sharper and more intense crystalline peaks compared with untreated fibers (Siró and Plackett, 2010).

Sharp peaks in the XRD pattern indicate well-defined crystalline structures, typically associated with cellulose, while, broad peaks or humps suggest disordered regions where cellulose chains are less organized or where non-cellulosic components like hemicellulose and lignin reside.

Table 2 shows the calculated fiber crystallinity values obtained from X-ray analysis, for untreated and treated samples. Diss (*Ampelodesma mauritanica*) fibers treated with citric acid show an improvement in crystal structure compared with untreated fibers. This results from the selective removal of non-cellulosic polysaccharides and the dissolution of less ordered (amorphous) components (Touati et al., 2020). Untreated fibers generally exhibit lower crystallinity due to the presence of natural impurities such as hemicellulose and lignin (Habibi et al., 2010).

In the other hand, citric acid acts as a chelating agent, selectively removing metal ions and potentially breaking down non-cellulosic components like hemicellulose and lignin (Bledzki and Gassan, 1999).

The treatment can enhance the relative crystallinity of the fibers by promoting the alignment and organization of cellulose chains (Wang et al., 2007). X-ray diffraction (XRD) results indicate that citric acid treatments applied to Diss fibers induce a substantial increase in their crystallinity. This phenomenon is due to the effective elimination of non-cellulosic amorphous components present in the fiber structure.

High fiber crystallinity, characterized by an ordered arrangement of molecular chains, gives fibers greater tensile strength. This property is transferred to the composite, improving its overall mechanical properties (Marwah et al., 2014).

3.1.3. Thermogravimetric analysis (TGA)

Thermogravimetric analysis was conducted on treated and untreated Diss fibers to assess their thermal behavior. The results of the analysis are presented in Fig. 4 and Table 3.

There are three major steps, the first step between 50 and 161°C is

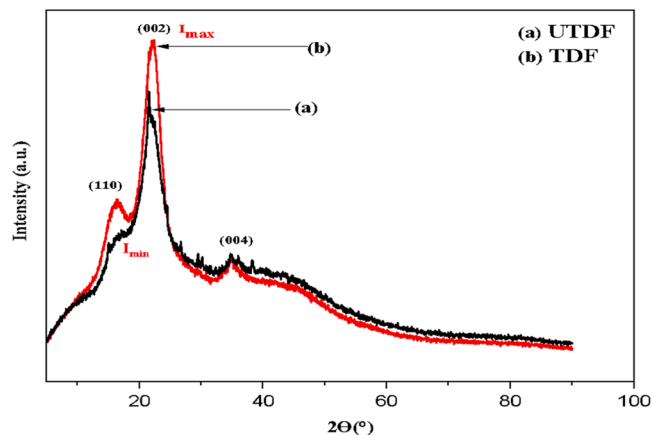


Fig. 3. The XRD spectra untreated and treated Diss (*Ampelodesma mauritanica*) fibers.

Table 2
Crystallinity of untreated and treated Diss (*Ampelodesma mauritanica*) fibers.

Samples	Crystallinity ($X_c\%$)
Untreated Diss fibers	44.2
Treated Diss fibers	52.39

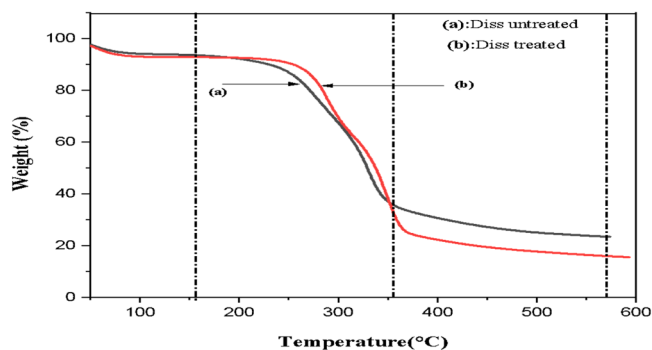


Fig. 4. TGA thermograms of untreated and treated Diss fibers.

Table 3
Thermogravimetric analysis results for both of untreated and treated Diss (*Ampelodesma mauritanica*) fibers and their biocomposites.

Samples	T_{onest} (°C)	T_{max} (°C)
UTDF	172.20	349.49
TDF	225.88	365.50
Neat PLA	302.86	386.37
UTDF/PLA	254.49	352.51
TDF/PLA	285.93	363.32

associated with the evaporation of moisture, with a weight loss of around 6%. Interestingly, Z. Touati (Touati et al., 2020) reported a similar mass loss of about (5%) when employing an alkaline treatment.

The second step between 200 and 356°C contributes to the decomposition of hemicellulose and cellulose (Sarasini et al., 2018).

Hemicellulose degrades easily between 200 and 315°C, while the main temperature range for cellulose pyrolysis is between 315 and 400°C (Sarasini et al., 2019; Covino et al., 2020; Zhang et al., 2015). The

third step is linked to the degradation of lignin and occurs in the range of 400 and 572°C (Touati et al., 2020).

After chemical treatment with (5%) citric acid, the thermal stability of the fibers was improved, as shown by the increase in the onset of degradation temperature (T_d) from 172.20 to 225.88°C and the increase in the maximum degradation temperature (T_{max}) from 349.45 to 365.50°C. These observations suggest that treated fibers exhibit greater resistance to thermal degradation compared to untreated fibers.

3.1.4. Morphological analysis

The surface morphology of untreated and treated Diss (*Ampelodesma mauritanica*) fibers was examined at various magnifications using a scanning electron microscope.

Fig. 5 shows the SEM micrograph of the untreated Diss (*Ampelodesma mauritanica*) fibers, indicating an irregular surface covered with impurities.

SEM micrographs of untreated Diss (*Ampelodesma mauritanica*) fibers typically show a heterogeneous surface with irregularities and natural impurities. Fiber surfaces may exhibit rough textures as a result of the presence of hemicellulose, lignin, and other non-cellulosic components. Additionally, some lignin may be present on the surface according (Zergane et al., 2020). Fig. 6 illustrates SEM micrographs of Diss (*Ampelodesma mauritanica*) fibers modified with (5%) citric acid, the fiber surface becomes cleaner and more uniform, that can be attributed to the removal of wax components, hemicellulose, lignin and other impurities. Smoother and more uniform fiber surfaces are observed post-treatment, indicating reduced surface roughness and improved fiber-matrix adhesion. This improvement in surface condition improves adhesion at the interface between composite materials.

3.2. Biocomposites characterization

3.2.1. Thermogravimetric analysis

Thermogravimetric analysis (TGA) was used to study the thermal stability of neat PLA and filled PLA composites. Fig. 7 shows the TGA curves for neat PLA, PLA/UDF and PLA/TDF. The introduction of Diss (*Ampelodesma mauritanica*) fibers, with or without prior treatment, modified the thermal stability of the composites, as shown in the TG curves, compared with PLA (Gozdecki et al., 2023). The reported initial and maximum degradation temperatures for neat PLA are 302.86°C and 386.37°C, respectively.

For composites, the presence of untreated and treated Diss (*Ampelodesma mauritanica*) fibers in PLA reduces their thermal stability compared with PLA neat, A. A. Yussuf et al. found that incorporating natural fibers like Kenaf and Rice husk into PLA resulted in a decrease in its thermal stability (Yussuf et al., 2010).

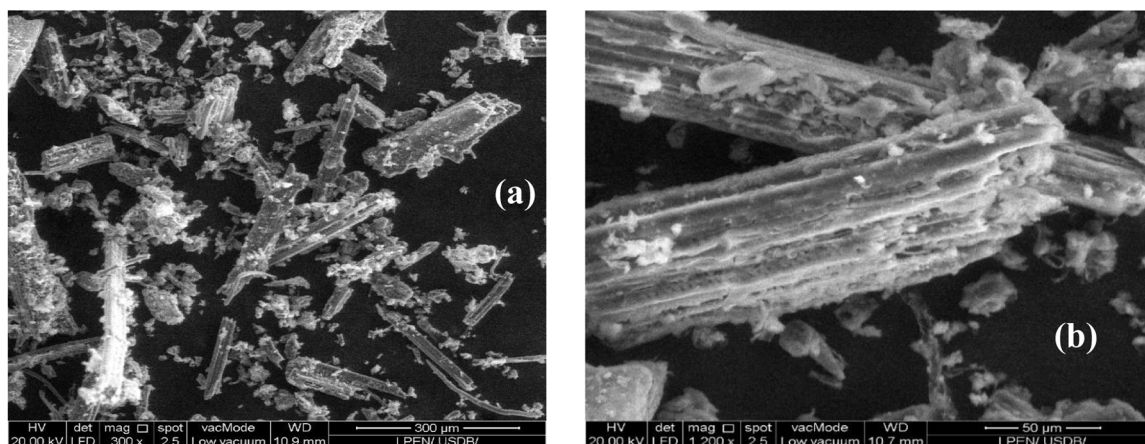


Fig. 5. Scanning electron microscope (SEM) micrographs of untreated Diss (*Ampelodesma mauritanica*) fibers at (a) Magnification 300× and (b) magnification 1200×.

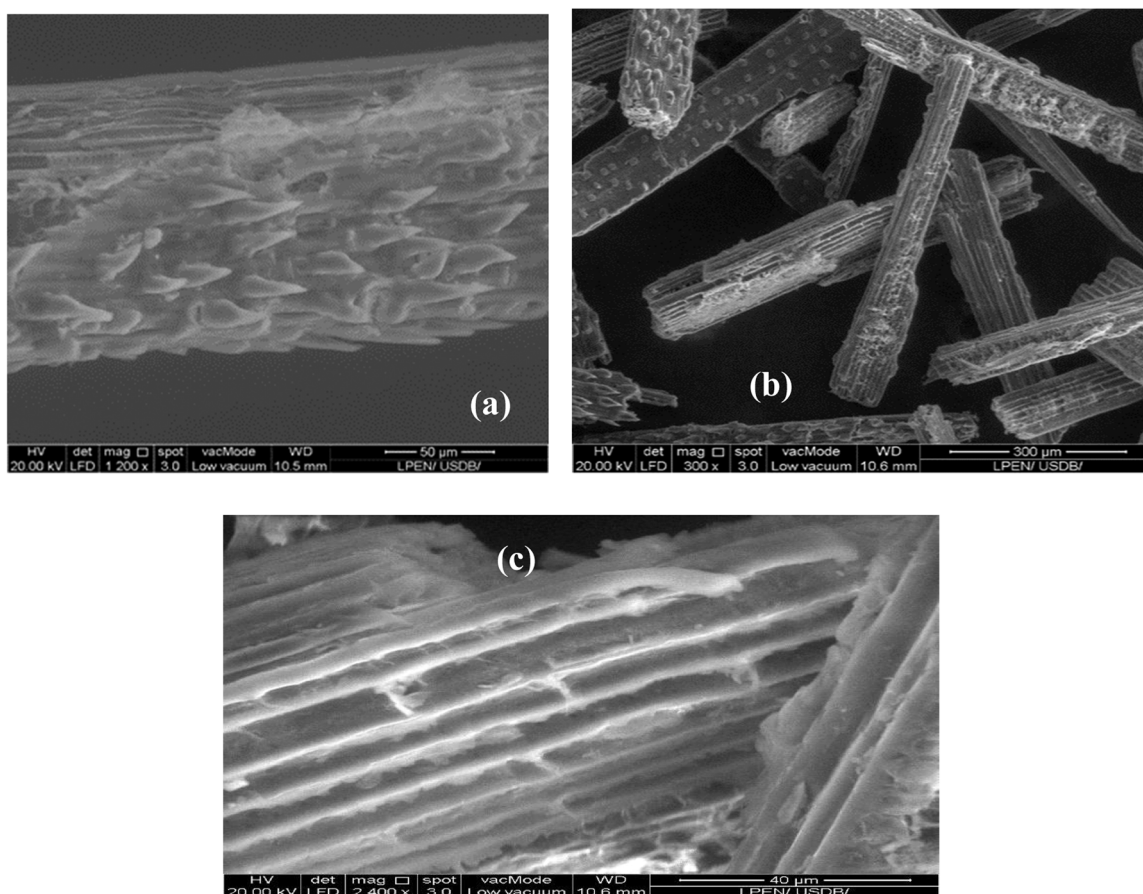


Fig. 6. Scanning electron microscope (SEM) micrographs of treated Diss (*Ampelodesma mauritanica*) fibers at different magnifications (a) 300×, (b) 1200× and (c) 2400×.

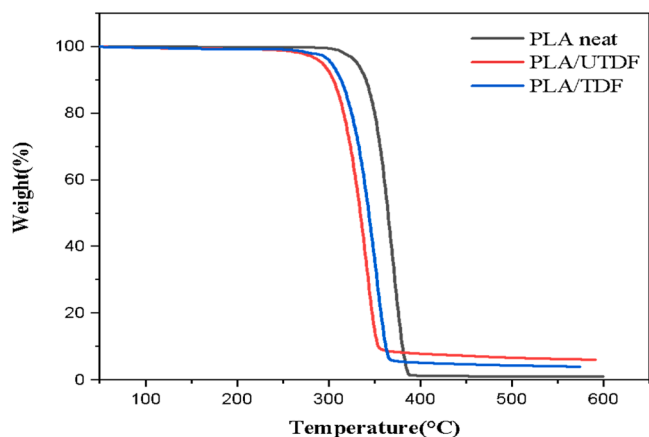


Fig. 7. TGA thermograms of pure PLA, PLA/UTDF and PLA/TDF.

In contrast, composites containing treated Diss (*Ampelodesma mauritanica*) fibers exhibit increased thermal stability compared with those with untreated Diss (*Ampelodesma mauritanica*) fibers, and the corresponding data were shown in Table 4. Removing impurities is a major factor in increased thermal stability. Impurities can act as weak points and degrade at lower temperatures, compromising the composite (Tablit et al., 2024). In conclusion the thermal stability of PLA/TDF can be improved by chemical treatment of fibers by citric acid. Treated *Ampelodesma mauritanica* fibers can enhance the thermal resistance of PLA composites, making them suitable for applications requiring thermal stability. Citric acid treatment has been shown to improve the

Table 4

Thermal Analysis Results of PLA and Its Composites via DSC.

Samples	T _g (°C)	T _c (°C)	T _m (°C)	ΔH _c (J g ⁻¹)	ΔH _m (J g ⁻¹)	X _c (%)
Neat PLA	66.3	137.04	168.43	2.49	65.55	67.80
Untreated Diss fibers (UTDF)	72.40	148.93	166.46	4.59	62.36	70.93
Treated Diss fibers (TDF)	74.50	145.82	164.19	4.13	58.98	73.72

Table 5

Izod impact strength analysis of neat PLA, PLA/UTDF and PLA/TDF composites.

Samples	Izod impact strength (J/m)
Neat PLA	24.15 ± 4.49
PLA/UTDF	10.42 ± 3.42
PLA/TDF	15.06 ± 6.01

thermal stability of natural fibers by modifying the fiber surface. This makes the fibers more compatible with the thermal processing conditions of PLA, ensuring better processing conditions and a more homogeneous composite.

The improved interface and fiber dispersion reduce the chances of thermal degradation of the fibers during manufacturing.

3.2.2. Differential scanning calorimetry

The thermal properties of pure PLA and its composites (PLA/UTDF and PLA/TDF) were characterized by differential scanning calorimetry (DSC) as shown in Fig. 8 and Table 4. Poly(lactic acid) (PLA) neat

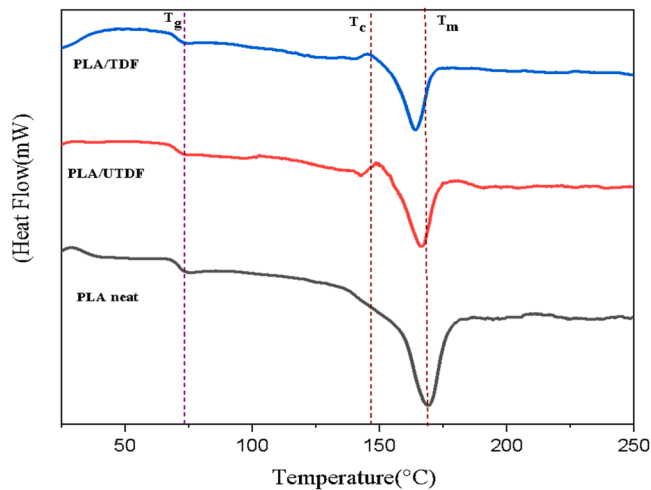


Fig. 8. DSC Analysis of Neat PLA, PLA/UTDF, and PLA/TDF.

underwent three clear transitions. The glass transition occurred at 66.3°C, followed by cold crystallisation at 136.62°C and finally melting at 168.43°C. Indeed, PLA's glass transition temperature (T_g) marks the change from a hard, glassy state to a soft, rubbery state. (Farah et al., 2016). T_c indicates the temperature at which PLA crystallizes upon cooling and is influenced by the cooling rate and the presence of nucleating agents (Di Lorenzo, 2006).

Finally, T_m is the temperature at which the crystalline regions of PLA melt, indicating the polymer's thermal stability (Gupta and Kumar, 2007).

The adding of untreated and treated Diss (*Ampelodesma mauritanica*) fibers into PLA resulted in an increase in T_g to 72.40°C and 74.50°C, respectively.

Interactions between PLA and Diss (*Ampelodesma mauritanica*) fibers reduce free volume, leading to an increase in viscosity through reduced chain mobility (Finnerty et al., 2023). Research by (Qian et al., 2013) evaluated the impact of alkaline surface treatments on the thermal properties of PLA/bamboo composites using DSC. The addition of 30 % untreated fibers resulted in an increase in glass transition temperature from 56.4°C to 59.1°C. Moreover, studies carried out by (Orue et al., 2018) highlighted the importance of surface treatment in modulating the thermal properties of PLA composites reinforced with sisal fibers, as shown by DSC analysis. They observed an increase in T_g from 60.3°C to 62.4°C after the incorporation of 30 % by weight of treated fibers. On the other hand, the cold crystallisation temperature increased by about 12.31°C and 9.2°C for PLA composites containing untreated and treated Diss (*Ampelodesma mauritanica*) fibers, respectively, relative to neat PLA.

This can be explained by the existence of interactions between Diss (*Ampelodesma mauritanica*) fibers and polylactic acid (PLA) matrix. The melting temperatures of the composites were all below that of neat PLA, although these variations can occur due to fiber-matrix interactions (Qin et al., 2011).

The T_m of PLA/UTDF and PLA/TDF composites generally remains similar to that of pure PLA, though the presence of fibers can lead to decreases due to the different thermal conductivities of the components, this improvement could be attributed to a better distribution of fibers within the matrix, as well as stronger bonds between Diss fibers and the matrix.

PLA/TDF composites have a greater degree of crystallinity than pure PLA or PLA/UTDF composites. The improved interfacial adhesion between TDF and PLA facilitates more uniform nucleation and crystallite growth.

In conclusion, the results obtained show that the thermal properties of pure PLA, PLA/UTDF and PLA/TDF differ due to the presence and

treatment of Diss (*Ampelodesma mauritanica*) fibers. DSC analysis reveals these differences, highlighting the impact of fiber incorporation and treatment on T_g , T_m , T_c and X_c of the composites.

3.2.3. Morphology of fracture surface

Fig. 9 and Fig. 10 provide a visual comparison of the structural morphology of PLA-based composites reinforced by Diss (*Ampelodesma mauritanica*) fibers. Fig. 9 shows a composite in which the fibers have not undergone any surface treatment. Fig. 10, on the other hand, shows the significant surface modification applied to the Diss (*Ampelodesma mauritanica*) fibers and its effect on the morphology of the resulting composite.

Scanning electron microscope (SEM) micrographs of the PLA/UTDF composite reveal the presence of defects such as voids and interfacial cavities, attributable to poor wettability of the untreated fibers by the PLA matrix, or imperfect wetting of the fibers during composite preparation, resulting in local separation of the interfaces. In comparison, SEM images of the PLA/TDF composite show no such defects, suggesting better adhesion and more homogeneous dispersion of the processed Diss (*Ampelodesma mauritanica*) fibers in the composite material (Asaithambi et al., 2017). SEM and FTIR analysis revealed a significant increase in fibre surface roughness after citric acid treatment. Direct consequence of this surface modification is improved fiber-polymer interfacial adhesion.

3.2.4. Mechanical properties

3.2.4.1. Izod impact strength. Untreated natural fibers are inherently hydrophilic, which creates poor compatibility with the hydrophobic PLA matrix, resulting in weak interfacial bonding. Citric acid treatment reduces the hydrophilicity of the fibers by reacting with surface hydroxyl groups.

This not only enhances fiber dispersion within the PLA matrix but also reduces moisture absorption, thereby improving the composite's mechanical properties over time, such as strength and stiffness.

According to (Siddiqui et al., 2024) and (Ferrandez-Garcia et al., 2020), the rate of moisture absorption decreases when the number of small fiber particles is high, which can lead to better adhesion between matrix and filler, thus increasing the density of the composite and reducing the porosity rate and therefore moisture absorption.

The incorporation of *Ampelodesma mauritanica* fibers (20 %) into PLA has resulted in a very compact surface structure, limiting crack propagation and improving the composite's impact resistance (Pujar and Mani, 2022)

The Izod impact test is a standardized method used to measure the impact resistance of materials. It is particularly useful for evaluating the toughness of composite materials. For composites untreated PLA/UTDF and PLA/TDF, we found respectively 10.42 and 15.06 J/m compared with 24.15 J/m for pure PLA.

Improved adhesion between the Diss fibers and the PLA matrix results in an increase in the impact resistance of the biocomposite. Moreover, the roughness of the fiber surface improves interfacial adhesion, thereby optimising the mechanical properties of the PLA/TDF biocomposite. For example, PHBV(Poly(3-hydroxybutyrate-co-3-hydroxyvalerate)/wood flour composites (70/30 and 60/40) showed a lower impact resistance than pure PHBV, dropping to 30 J/m and 24.4 J/m respectively compared with 34 J/m. This decrease is attributable to a ductile-to-brittle transition induced by the addition of wood flour (Wang et al., 2019; Ashori and Nourbakhsh, 2010). It can be concluded that impact resistance is determined by a combination of physical and geometric properties of the wood fiber (crystallinity, % cellulose, elongation, geometry and length) and the polymer matrix (Callister, 2018).

Fig. 11 shows the results of the Izod test, which assesses the impact resistance of PLA and its biocomposites. The PLA/UTDF, PLA/TDF, and

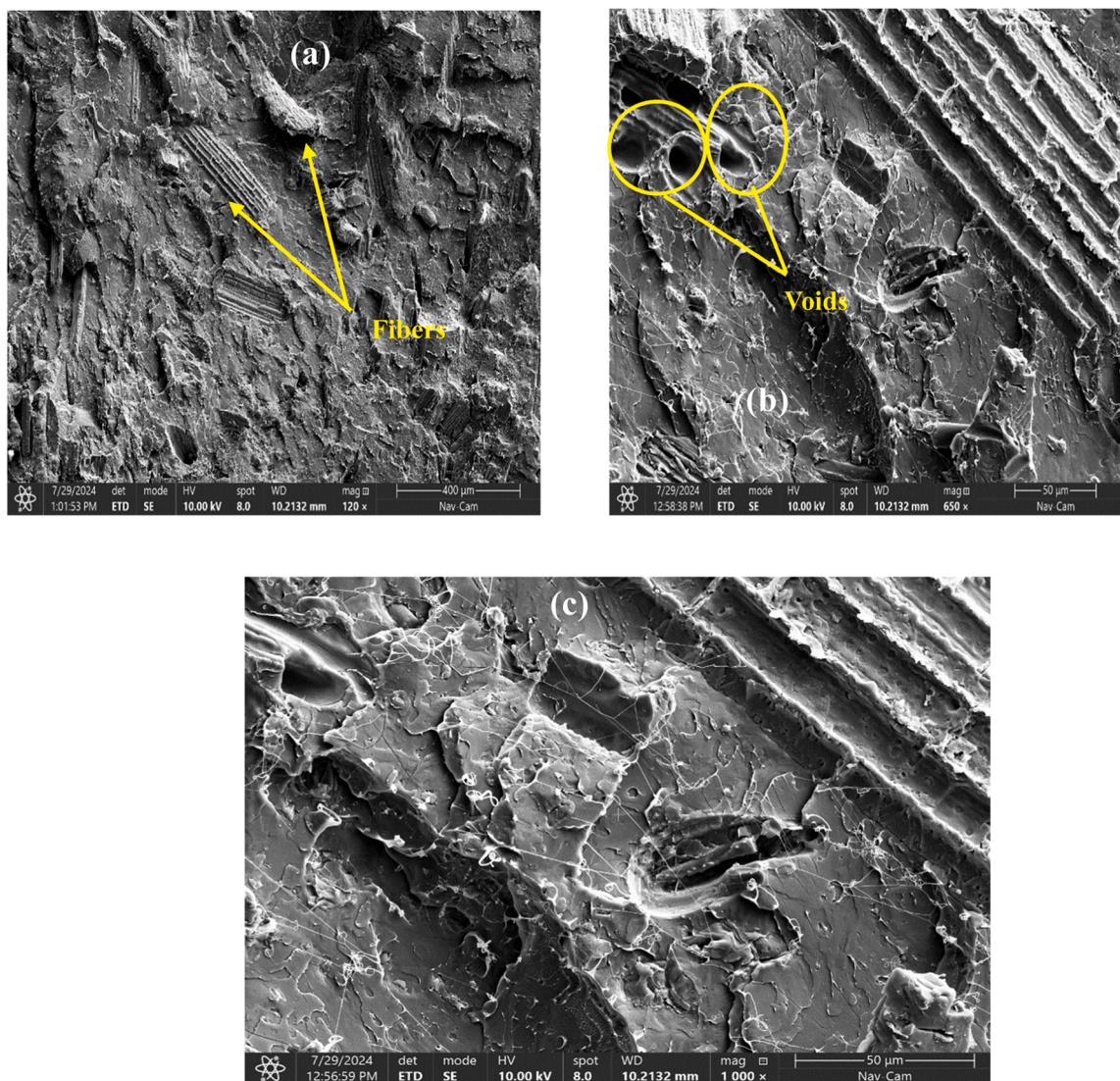


Fig. 9. Scanning electron micrographs showing PLA/UTDF composites at (a) magnification 120 \times , (b) magnification 650 \times and (c) magnification 1000 \times .

neat PLA biocomposites represent some of the materials investigated. The pristine PLA sample was used as a reference for comparison, showing an Izod impact strength of 24.15 kJ/m. Since there was probably insufficient interaction among the Diss (*Ampelodesma mauritanica*) fiber particles and the polymer matrix, the composite PLA/UTDF demonstrated a reduced impact strength of 10.42 J/m. By contrast, the PLA/TDF biocomposite exhibited a high impact strength of 15.06 J/m, but PLA/UTDF could not enhanced interfacial adhesion between the PLA matrix and the Diss fiber particles. This lack of enhancement is attributed to the inadequate interfacial adhesion, which also limits the compatibility between PLA and Diss fibers.

The improvement in Izod impact resistance can be attributed to enhanced compatibility and dispersion of the Diss fibers, which effectively reduce stress concentrations and facilitate better energy dissipation during impact.

The homogeneous dispersion of Diss fibers (*Ampelodesma mauritanica*) in the composite material, observed in SEM after chemical treatment with citric acid, contributes to a significant improvement in the mechanical properties of PLA biocomposites.

3.2.4.2. Tensile properties. Fig. 12 compares the modulus and tensile strength of pure PLA and PLA/Diss composites, with and without citric acid treatment. Five samples were tested for each material.

Each material type was tested with five samples. The mechanical properties of pure PLA and its composites reinforced with UTDF and TDF are summarised in Table 6. This shows the effect of reinforcements on tensile modulus, tensile strength and elongation at reapture.

The modulus and strength tensile of the PLA composite reinforced with untreated Diss fibers were measured at 4.044 GPa and 46.37 MPa, respectively. After treating the Diss (*Ampelodesma mauritanica*) fibers with (5 %) citric acid, these values increased to 4.680 GPa for the modulus and 48.90 MPa for the tensile strength. The improvement in mechanical properties can be explained by a better interface between the fibers and the PLA.

This combination results in composites with improved tensile strength, and modulus (John and Thomas, 2008). In conclusion, these natural fibers enhance the tensile strength and modulus of PLA, leading to composites that are stronger and more rigid compared with pure PLA.

Pure PLA usually shows significant elongation at reapture, Fig. 13, showing that it may stretch before breaking. When compared with other polymers, PLA is known for being brittle, this leads to relatively little elongation at reapture values. Whenever compared with neat PLA, the elongation at reapture tends to decrease while untreated Diss (*Ampelodesma mauritanica*) fibers are incorporated into the PLA matrix.

Stress concentration detects and reduced ductility are a consequence of weak interfacial interaction between the hydrophilic fibers and the

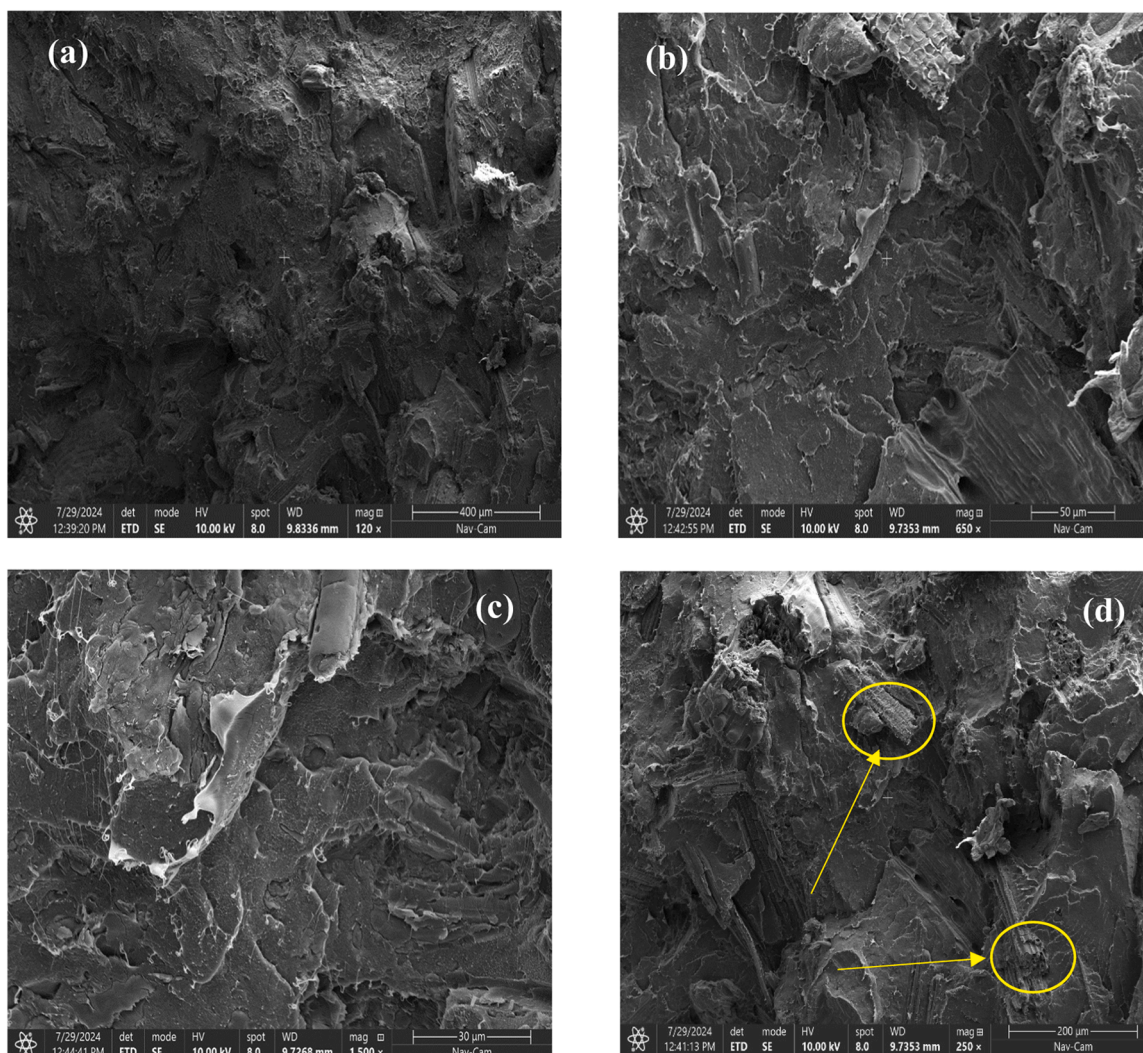


Fig. 10. Scanning electron micrographs showing PLA/TDF composites at (a) magnification 120×, (b) magnification 650×, (c) magnification 1500× and (d) magnification 250×.

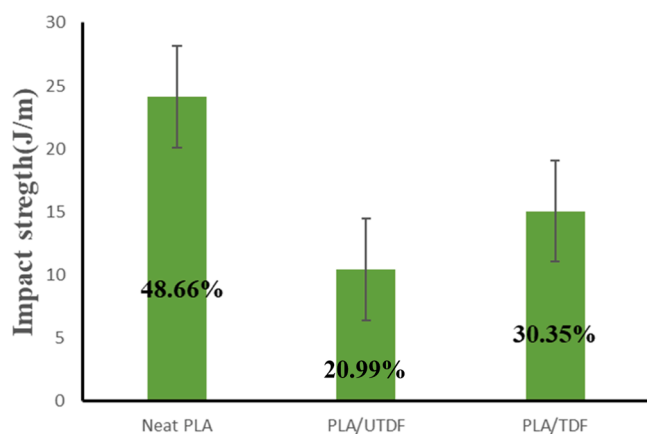


Fig. 11. Impact of chemical treatment of Diss fibers on the impact resistance of neat PLA, PLA/UTDF and PLA/TDF composites.

hydrophobic PLA matrix. Diss (*Ampelodesma mauritanica*) fibers have the potential to act as composite imperfections causing the material to fail prematurely whereas under tensile stress. An increase in elongation at rupture of the citric acid treatment can be attributed to better

interfacial bonding.

By improving the fiber-matrix adhesion, the treated fibers can better transfer load from the PLA matrix, leading to a more efficient reinforcement. This translates into enhanced mechanical properties such as tensile strength, modulus of elasticity, and impact resistance. Citric acid treatment helps fibers bond more strongly to the PLA matrix, reducing the chances of fiber pull-out during mechanical loading, which contributes to improved durability and performance.

3.2.5. The water absorption

Fig. 14 shows that composites reinforced with *Ampelodesma mauritanica* fibers absorb more water than pure PLA after 24 hours of immersion. PLA/UTDF and PLA/TDF composites show absorption rates of 2.28 % and 1.53 % respectively, compared with 0.65 % for pure PLA. Interestingly, citric acid treatment of *Ampelodesma mauritanica* fibers significantly reduces the water absorption of the corresponding composite. This result suggests that the treatment improves the interface between the PLA matrix and the fibers (Petchwattana et al., 2019).

4. Conclusion

In the present work, in order to evaluate the effectiveness of chemical treatment with citric acid as a surface modification method of *Ampelodesma mauritanica* fibers, was chosen. This treatment increases the

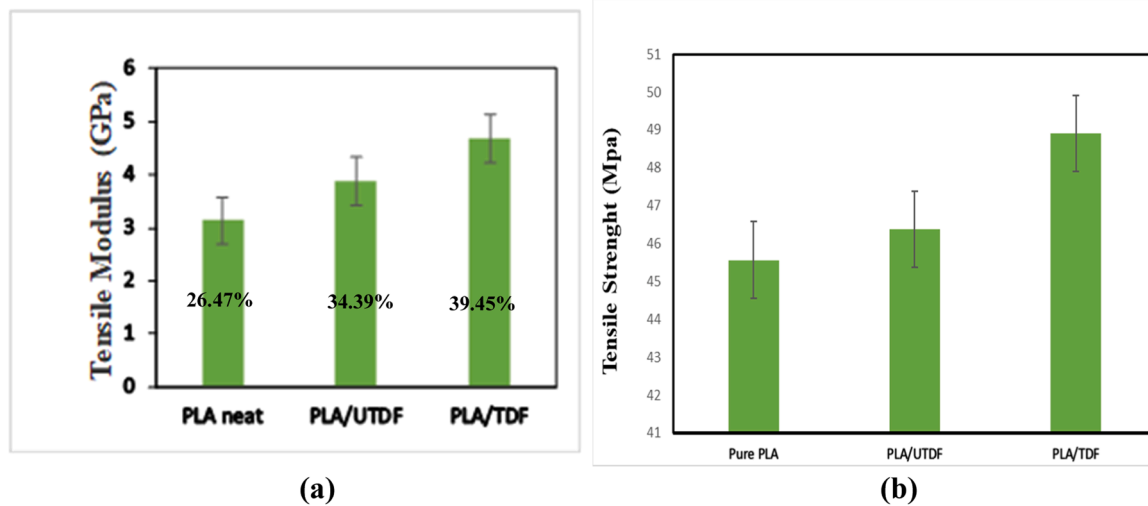


Fig. 12. Mechanicals properties of composites: (a) tensile modulus and (b) tensile strength.

Table 6
Analysis of Mechanical Properties in PLA/UTDF and PLA/TDF Composites.

Samples	E(GPa)	Tensile Strength (MPa)	Elongation at repture(%)
PLA	3.140 ± 0.41	45.56 ± 3.65	3.1±0.70
PLA/UTDF	4.044 ± 0.35	46.37 ± 4.00	2.34±0.64
PLA/TDF	4.680 ± 0.81	48.90 ± 3.85	2.52±0.34

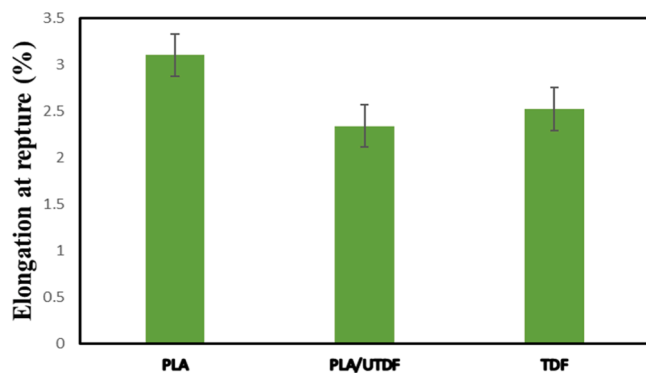


Fig. 13. Elongation at repture of polylactic acid and its composites.

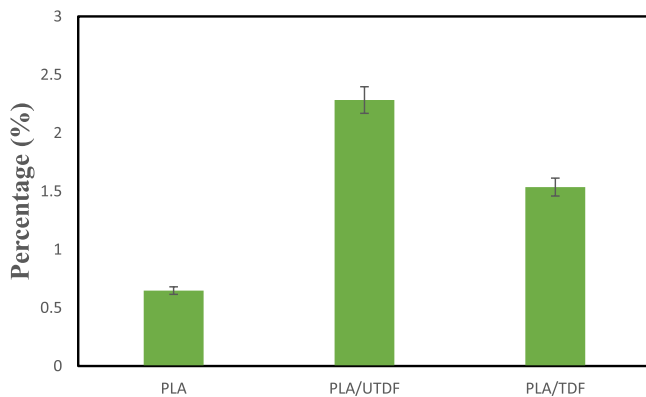


Fig. 14. Water absorption of purPLA, its biocomposites PLA/UTDF and PLA/TDF.

surface roughness by removing non-cellulosic compounds

(hemicellulose, lignin, wax). The proposed morphologies (SEM) and thermal (DRX) on an improved surface modification of *Ampelodesma mauritanica* fibers. The surface of *Ampelodesma mauritanica* fibers contributes to generate and increase the number of functional groups displayed on the surface and which interact with the PLA matrix.

A better interfacial adhesion within the biocomposite between polymer and fibers guarantees a large charge transfer and consequently a composite material with more efficient properties.

The interfacial adhesion of the biocomposite (PLA/TDF) was analyzed using a scanning electron microscope (SEM), this device allowed to show a clear increase in this adhesion. However, the citric acid treatment contributed to the improvement of the thermal stabilization of the biocomposite as illustrated by the TGA curves. However, tensile properties showed an optimal result.

It was observed that some mechanical properties such as elongation at break, tensile strength and Young's modulus were improved and their values reached 2.52 %, 48.90 MPa and 4.68 GPa respectively.

PLA reinforced *Ampelodesma mauritanica* fiber composite offers a range of benefits including improved tensile properties, biodegradability, durability and economic advantages that make these composites versatile and more suitable for a multitude of applications in the emergence of new biodegradable materials.

In conclusion, the novelty of the citric acid treatment of *Ampelodesma mauritanica* fibers lies in its ability to enhance the adhesive properties between natural fibers and a PLA matrix through an eco-friendly process. The impact of this treatment is reflected in improved mechanical and thermal properties of the composite, leading to a stronger, more durable, and sustainable material that has potential applications in various industries.

CRedit authorship contribution statement

Farid Rouabah: Writing – review & editing, Methodology, Formal analysis, Data curation. **Brahim Barka:** Writing – review & editing, Validation, Investigation, Formal analysis, Data curation. **Nadia Kharchi:** Writing – original draft, Validation, Methodology, Investigation, Conceptualization. **Haroun Houicha:** Writing – review & editing, Resources, Formal analysis, Data curation. **Fois Magali:** Writing – review & editing, Investigation, Formal analysis, Data curation. **Deradji. Dadache:** Writing – review & editing, Resources, Investigation, Formal analysis, Data curation. **Abdel-Hamid Ismail Mourad:** Writing – review & editing, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization. **Abdeslam Bencid:** Writing – review & editing, Resources, Methodology, Formal analysis.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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