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Elaboration and characterization of composite material based on epoxy resin and Cynara scolymus fibers: Weibull statistics analysis

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ABSTRACT

The research will fill the increasing demand of sustainable composite materials by designing and characterizing an epoxy based biocomposite that is reinforced with natural fibers that are derived through the Cynara scolumus (artichoke stem) agricultural waste. The study methodically examines how fiber reinforcement influences the performance of the composite under a controlled extraction of the fibers, and also the unidirectional laminates of the composite were produced with single, double, and triple ply. The fibers were methodologically described in physical properties with the help of water absorption kinetics based on the Peleg equation, and mechanical performance with tensile tests and impact tests according to the ASTM standards, including SEM and EDX tests. The most important findings include the fact that the mechanical properties are greatly increased with the number of fiber plies: single-ply composite reached the ultimate tensile strength of about 15 MPa, double-ply composite tensile strength was 32 MPa, and the triple-ply composite tensile strength was 60 MPa with better strain at break (about 2.2%). The resistance to impact also improved with the number of ply and the adhesion of fibers to the matrix was also confirmed by SEM with a small number of voids and EDX gave a fiber composition of 56.49% carbon and 34.05% oxygen. The paper presents the new application of the *Cynara scolymus* fibers, which are an untested agricultural waste, in epoxy composites, and is the first use of Weibull statistical analysis to describe the consistency and reliability of these fibers as a sustainable reinforcement. The paper concludes that

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Cynara scolymus fibers are an effective, renewable reinforcement, with a very good balance of low density, better specific strength, and acceptable moisture uptake, thus contributing to the valorization of agricultural residues to support the eco-friendly structural and semi-structural composites.

Nomenclatures

Symbol	Definition
L	Length of fiber (cm or m)
m	Mass or weight of fiber (g)
w	Weight of fiber (g)
w_1	Initial fiber weight before drying (g)
w_2	Fiber weight after drying (g)
M_R%	Moisture regain percentage (%)
M_C%	Moisture content percentage (%)
dex	Linear density of fiber (tex)
g(t)	Water sorption at time *tt*
w_0	Initial moisture content in Peleg model
k_1,k_2	Constants in Peleg model
t	Time (minutes)
UD	Unidirectional
SEM	Scanning Electron Microscopy
EDX	Energy Dispersive X-ray Spectroscopy
ASTM	American Society for Testing and Materials

1. Introduction

The creation of eco-friendly and sustainable composite materials has gained a rise in relevance in the contemporary materials engineering as a result of the increased environmental awareness and the need to promote decreased reliance on synthetic fibers. Natural fiber-reinforced polymer composites have been considered as promising substitutes of the traditional synthetic fiber composites with myriad benefits such as biodegradability, low costs, availability and environmental impact. The use of agricultural waste fibers in composite applications, among other natural fibers that are currently under research, offer rather promising prospects of value addition and reduction of waste. The agricultural residue of cynara scolymus, also referred to as artichoke, has the potential of being used as reinforcement material in polymer composites, both in waste management and the creation of sustainable materials [1].

There has been considerable interest in the natural fiber-reinforced composites because it can be used in many applications instead of glass and carbon fiber reinforced composite. Various factors affect the mechanical properties of these composites which include fiber-matrix interface, absorption of moisture, and the actual nature of the natural fibers. Absorption of water can be considered as one of the most difficult issues of natural fiber composites because it can have a significant influence on the mechanical and dimensional stability of the material. Zamri et al. [2] examined the influence of water absorption on unsaturated polyester hybrid composites with pultruded jute/glass fiber illustrating how water uptake may cause fiber swelling, plasticization of the matrix and degradation of fiber-matrix interface. They put emphasis on the need to learn about moisture behavior in natural fiber composites, especially in hybrid forms where natural fibers are used together with synthetic reinforcements to maximize their performance characteristics.

The hybrid composite formation that entails the use of natural fibers with synthetic reinforcements has posted encouraging outcomes in the realization of equal mechanical characteristics. Abd El-baky [3] also made an in-depth analysis of mechanical properties of the jute/glass/carbon fiber reinforced hybrid composition and noted that the strategic hybridization can positively influence the overall performance of the composite because of the complementary characteristics of various types of fibers. This would tackle some of the intrinsic shortcomings of the pure natural fiber composites whilst being sustainable. On this idea, Alshahrani et al. [4] studied the jute-basalt reinforced epoxy hybrid composite as a new kind of lightweight structural automotive, and it is possible to consider natural fiber hybrids in challenging engineering processes when weight loss and mechanical performance are key factors.

The range of application and potential of natural fiber-reinforced polymer composites cuts across many applications across various industries. Keya et al. [5] made an extensive discussion on natural fiber reinforced polymer composites, their historical background, their types, their benefits, and its broad range of application. Their review highlighted that natural fibers have great advantages in that they are renewable, low density, good specific mechanical properties, and less health hazards in the processing as compared with synthetic fibers. Of the numerous natural fibers that have been examined, the agricultural waste fibers including bagasse have gained a lot of attention because of their abundance and the possibility of being used in value-added purposes.

An example of the potential of agricultural waste fibers in composites can be bagasse, or fibers that are left by the extraction of sugar in sugarcane. Balaji et al. [6] conducted a review of bagasse fiber as a potential biocomposite material in the future with regard to its availability, low cost, and appropriate mechanical properties to be used as reinforcements. The use of these agricultural residues does not only offer the economic remedy of dealing with waste, but also helps in coming up with sustainable materials. The idea of using agricultural waste can be applied to other vegetable produces wastes, such as artichoke processing, which have the same properties and can be used as composites reinforcers.

The performance of natural fiber composite is highly dependent on the type of matrix material used. Epoxy resins are also one of the most popular thermoset polymers to be used as a composite component because of the high mechanical, chemical, and adhesive characteristics. New polymer systems and associated thermosetting polymers have been developed as a result of recent breakthroughs in polymer chemistry involving epoxy-based systems. Xu et al. [7] prepared fluoropolymer/glycidyl azide polymer (GAP) block copolyurethane as new energetic binders which proved innovations in synthesis of polymers which improve their mechanical properties and thermal performance. These advances in the polymer science offer the information about the method of optimization of the matrices that might be applied to the natural fibre composite systems.

Natural fibers have been widely researched so as to learn more about their reinforcement properties and their compatibility with other matrix systems. Kenaf fiber has been one of the most promising natural reinforcement and extensive researches have been conducted on knowing the nature and use of this reinforcement. Akil et al. [8] have conducted a comprehensive analysis of kenaf fiber reinforced composites and analyzed the properties of the fibers, processing mode, mechanical performance and their possible usage. Their research set up some background information regarding how natural fibers behave in polymer matrices and provided some of the important factors which influence composite performance. However, more recently, Owen et al. [9] surveyed the recent developments in the enhancement of mechanical and thermal characteristics of kenaf fibers/engineering thermoplastic composites by recently developed coating methods, and in this regard, the authors pointed out the significance of fiber surface modification in the improvement of fiber-matrix bonding and composite characteristics.

Agricultural waste fiber composites have been shown to have mechanical performance as evidenced by different research studies. Abd El-Baky et al. [10] measured mechanical characteristics of sugarcane bagasse-glass/polyester composites, which are beneficial to understanding the load carrying capacity and mode of failure in agricultural fiber-based products. Their results showed that fiber treatments and ideal fiber content are very important to attain good mechanical performance. This knowledge was developed by Gopalan et al. [11], who foretold the virtual fatigue parameters of fly ash, boron nitride, and sugarcane fiber-reinforced epoxy composite, indicating that it is an excellent characterization method to comprehend the long-term performance and durability of natural fiber composite when subjected to cyclic loading conditions.

Modern polymer chemistry studies have also been devoted to the creation of recyclable and re-processable thermoset systems which contain no end-of-life issues that are inherent to the conventional thermosets. This paper by Zhang et al. [12] presents innovations in the production of sustainable thermoset materials with enhanced processability and recyclability; the authors have created weldable, reprocessable, and water resistant polybenzoxazine vitrimer crosslinked by dynamic imine bonds. On the same note, Zhang et al. [13] researched on disulfide-mediated reversible polymerization of intrinsically dynamic smart materials and presented a new prospect of designing responsive and recyclable polymer systems. Such developments in dynamic polymer chemistry have provided possible avenues of developing more sustainable epoxy-based composite systems.

Thermoset composites now appear to be evaluated on the basis of their recyclability and environmental sustainability. Liu et al. [14] explored selective cleavage of ester linkages of the epoxy cured with anhydride through benign way and recycling the decomposed polymer in further preparation of epoxy and proved that it is also viable to recycle epoxy. Li et al. [15] have designed low-viscosity degradable benzyl cyclic acetal epoxy monomers, investigated synthesis, structure-property relationship, and the use in sustainable thermoset materials, which is another step forward in the context of sustainable carbon fiber composite.

Outside of mechanical uses, natural fiber-based materials have been found promising in energy storage and environmental uses. Mohamed et al. [16] made ready microporous carbon and carbon/metal composite materials based on bio-benzoxazine-linked precursor to capture and store CO2 and other gases as a source of energy, and this indicates the versatility of bio-based materials. Duan et al. [17] examined the structure regulation of hard carbon using enriched semi closed ultramicropores to achieve improved rapid sodium storage whereas Liu et al. [18] examined the recycling of the waste glass fiber reinforced epoxy resin by using electroless plating of magnetic particles and carbonizing of them to produce a microwave absorber. Shi et al. [19] analyzed heteroatoms co-doped porous carbons of amino acid based polybenzoxazine to achieve the best performances in electrochemical processes of CO2.

Most recent trends in bio-based materials have also investigated the flex-electronics and energy storage applications. The paper by Pan et al. [20] introduced and produced completely biobased high-strength and high-toughness double cross-linked cellulose hydrogel of flexible electrolytes, which has seen the broadening of the application of natural materials in structural composites.

Techawinyutham et al. [21] examined natural fibers composites to the role of a sustainable alternative, but in most cases, the mechanical performance of the natural fibers composite is inferior to the synthetic one. Pineapple and hemp mat fabrics were used in creating hybrid natural/synthetic fiber composite with intermediate glass fiber mats to improve the performance properties. The composites were produced through the vacuum infusion process with a different arrangement of hemp and pineapple fibers stacked on the top of one another and to enhance mechanical strength and durability, the composites were hybridised by incorporating glass fibre mats. The mechanical findings showed that the pure hemp composite with intermediate layers of glass had the highest tensile and flexural test, with tensile strength of 58.24 Mpa and flexural strength of 98.35 Mpa. Viscoelastic test revealed that the pure pineapple fibre composite exhibited the highest storage modulus of 2875.51 Mpa which was in accordance with the trend in flexural modulus. Sharma et al. [22] used different machine learning (ML) models, which include Support Vector Regression (SVR), Decision Tree (DT), Random Forest (RF) and k-Nearest Neighbors (kNN) to predict mechanical properties of polymer nanocomposites prepared through a microwave process. The tensile strength, fracture toughness, and the Young modulus of Polypropylene/Carbon Nanotubes (PPNT), the High density Polyethylene/Carbon Nanotubes (HPNT), and Low density Polyethylene/Carbon Nanotubes (LPNT) composites were measured using a universal testing machine. The findings show that SVR is the best and most reliable model in the estimation of mechanical properties of all composites. It has been able to reduce inaccuracies and generalize and this has made it perform well even when dealing with limited datasets. The production of hybrid composites made with Innegra and hemp fibers into a synthetic epoxy matrix and their measurement of mechanical, thermal and viscoelastic properties was carried out by Yorseng et al. [23] under varied arrangement of stacking of the composites both prior to weathering and after weathering. Pure Innegra composites had a tensile strength of 106 Mpa and this dropped to 97.87 Mpa after weathering and hybrids comprising of hemp and Innegra with core layers showed tensile strength of 70.6 Mpa before weathering. Hybrids using Innegra as skin layers had the best flexural strength, but flexural characteristics were comparable. The mechanochemical study revealed that there were increased structural stability in hemp skin layer composites. A storage modulus (1131.34 Mpa) of the pure hemp composites declined extensively following weathering as indicated in dynamic mechanical analysis.

Sharath et al. [24] examined the production and properties of the luffa fibre reinforced epoxy composites with various stacking order and fibre treatment. The lay-up mix of composites was based on the manual lay-up technique combined with compression moulding. Three laminates were used, namely L1, L2 and L3; untreated luffa fibres, HCl-treated fibres and NaOH-treated fibres. Mechanical testing - Chemical tests showed that tensile, flexural strength, interlaminar shear strength, and impact strength all enhanced significantly when chemical treatments were applied in comparison to the untreated L1 laminate. The tensile strength of L3 treated with NaOH was the highest at 16.47 N/mm^2, flexural strength at 11.205 N/mm^2, interlaminar shear strength at 4.105 N/mm^2 and better impact energy absorption. The alkali treatment was also superior to the acid treatment in the enhancement of fibre-matrix adhesion through the removal of hemicellulose and lignin. Arpitha et al. [25] utilized the corncob waste available in the nearby agricultural fields alongside boron nitride particles as the reinforcement agent to make epoxy based composite. Composite material that has been fabricated has been defined in terms of tensile strength, flexural strength, the water up-take test as well as the fire resistance test. The hand layup method of fabricating nine laminates with varying ratios of corncob and epoxy are accomplished. It has been found out that 3.5/3.5 boron nitride and corncob ratio (wt./wt.) composition has the best flexural strength of approximately 36.7 N/mm^2 and no further characterization has been done to this composition composite.

The proposed research is highly novel and motive because the application of *Cynara scolymus* (artichoke stem) fibers as an untested form of agricultural wastes as the reinforcement component in the epoxy-based biocomposites is innovative. The necessity to make a breakthrough and find eco-friendly alternatives to synthetic fibers drives the research, as it is necessary to solve the issue of environmental concern and waste valorization. It has been methodologically presented as a systematic study of unidirectional laminates on single, two, and triple-ply designs with a combination of advanced characterization methods (Peleg model of water absorption, Weibull distribution of fiber consistency, SEM/EDX, and ASTM-standard mechanical tests) to analyze in detail the effect of ply count on performance. The results indicate that tensile strength (up to 60 Mpa) and impact resistance increases with the addition of more fiber plies, without reducing the fiber-matrix adhesion and void content, which makes the artichoke fibers an effective, renewable reinforcement in structural and semi-structural composite.

2. Experimental procedure and materials characterization

This section describes the different experimental steps implemented for the study of natural fiber-based biocomposite materials. It presents both the materials and equipment used, as well as the characterization techniques applied to evaluate the physical, mechanical, and morphological properties of the prepared samples.

The objective is to provide an overview of the experimental methods used and the methodology adopted to link the internal structure of the material to its overall behavior. The tests were carried out in accordance with international standards (ASTM) to ensure the reliability, comparability, and reproducibility of the results. The analyses performed allow for the evaluation of the biocomposite's performance and the quality of the fiber/matrix interface, a key parameter in the mechanical behavior and durability of the material [26–28].

Fig. 1 shows how *Cynara scolymus* fibers in stems of artichoke are extracted and retted to get the final product. This is the first preparation process that is vital in the production of high quality natural reinforcements. This is probably achieved by isolating the fibers of the plant by means of the organic matrix i.e. water or dew retting where microbial activity is used to degrade pectins and lignin and the fibers are then extracted using a mechanical force to obtain individual fibers. This regulated treatment is designed to purify the fibers, to promote the surface properties of the fibers and to make them more compatible with the epoxy matrix and therefore, guarantee the enhancement in the interfacial bonding and performance in the resultant biocomposite.

2.1. Water absorption

The moisture regain and content percentage of fibers were assessed using the ASTM 2654 method. A 0.264 g sample was tested in a controlled at room temperature of 25 °C and 62% relative humidity, placed in a 100 °C air oven. Weights were measured at 25-min intervals until a 0.1% mass decrease occurred. Moisture absorbency, regain, and content of the fibers were calculated by comparing weights before and after drying [29,30].

$$M_R\% = \frac{w_1 - w_2}{w_2} \times 100 \quad (1)$$

Fig. 1. Extracting and retting process.

$$M_C\% = \frac{w_1 - w_2}{w_1} \times 100 \tag{2}$$

The moisture regain percentage (MR%) refers to the moisture regain of fibers, while the moisture content percentage (MC%) indicates the moisture content.

Where: w_1 is the initial fiber weight before oven drying

w_2 is the weight after drying.

Table 1 display the measurement of 22 samples of fibers and measuring their length L (*Cm*) and weight m (g).

Using experimental results of gathered from examining the samples, the Peleg model was applied to predict the water sorption kinetics of Cynara Scolymus Fibers using the mathematical formula (Eq. (3)). where w_0, k_1, and k_2 and t is the time in minutes [31].

$$g(t) = w_0 + \frac{t}{k_1 + k_2 t} \tag{3}$$

Using following the ASTM E1750-01 standard, the linear density (d_{ex}) of Cynara Scolymus Fibers was calculated from ten fiber samples by measuring their length (*L*) in meters and weight (*w*) in grams [31].

$$d_{ex} = \frac{w}{L} \times 1000 \tag{4}$$

The mechanism of water absorption of the *Cynara scolymus* fibers is shown in Fig. 2 which represents the experimental apparatus in which the sample fibers are subjected to a controlled environment to test their hygroscopic property. It is done by putting already weighed fibers under a given temperature and humidity and monitoring their mass increase in a given time interval. The important parameters that can be calculated include moisture regain (M_r%) and moisture content (Mc%) that are important in comprehending whether the fibers are hydrophilic or not and predicting their performance and stability in terms of dimensions when incorporated in an epoxy matrix to use as composite materials.

3. Biocomposite fabrication

In this study, the main objective is to develop and characterize epoxy matrix biocomposites reinforced with unidirectional natural fibers extracted from Cynara Scolymus. This work is part of the current effort to replace synthetic fibers with plant-based reinforcements, which are lighter, biodegradable, and have a low environmental impact [32–34].

The approach adopted involves experimentally designing several lamination configurations to evaluate the influence of the number of fiber layers on the mechanical and physical properties of the final material. Three types of specimens were thus fabricated (as presented in Fig. 3).

This gradual design allows for the analysis of the effect of progressive reinforcement on the internal structure of the composite, the interlaminar cohesion, as well as on its overall behavior under tension, and moisture absorption. The entire manufacturing protocol (from the preparation of the matrix and fibers to the final shaping) was rigorously defined to ensure the reproducibility and quality of the resulting composites.

Table 1
Fibers and measuring their length and weight.

Length (cm)	Weight (g)	Length (cm)	Weight (g)	Length (cm)	Weight (g)
7,5	0,0180	6,2	0,0114	7,6	0,0204
7,5	0,0083	7	0,0050	7,4	0,0102
7,5	0,0094	7,4	0,0091	7	0,0041
7,5	0,0110	7,5	0,0103	7,4	0,0088
9	0,0096	7	0,0058	5	0,0069
8	0,0091	4,5	0,0035	7,7	0,0060
11	0,0088	7,4	0,0092	7,7	0,0063
11	0,0169	7	0,0057	5,1	0,0054
7,3	0,0083	7,4	0,0081	3	0,0048
7,6	0,0030	7,4	0,0193	5,9	0,0068
5,9	0,0156	5,4	0,0080		
5,9	0,0044	7,6	0,0063		

3.1. Preparation of the matrix

The matrix used for the development of the biocomposites is an epoxy resin combined with a peroxide-based hardener. This choice is justified by its good mechanical properties, its chemical compatibility with natural fibers, its moderate cost, and its wide commercial availability. Epoxy is commonly used in the manufacture of natural fiber composites due to its low viscosity, which promotes good fiber impregnation and effective interfacial adhesion [27,35].

The polymer preparation involves mixing the epoxy resin with 0.5% (by mass) of hardener, followed by manual mixing for 5 min to ensure homogeneity. The mixture is then poured into a mold that has been previously cleaned and coated with a thin layer of release wax, and then left to cure at room temperature for 24 h before demolding (Fig. 4).

3.2. Reinforcement preparation

The natural fibers are obtained from Cynara Scolymus, carefully cleaned, dried, and cut to an average length of 25 mm. These fibers are then manually aligned to form unidirectional (UD) reinforcements.

To maintain their orientation, the fibers were fixed with adhesive tape onto a sheet of paper, and then mechanically stitched to obtain stable and homogeneous fiber plies, thus ensuring good inter-ply cohesion and a uniform distribution of stresses within the composite (Fig. 5).

3.3. Fabrication of composite specimens

Fig. 6 shows the chronological process involved in the production of unidirectional laminated composite specimens by the hand lay-up process. It is started by the preparation of the epoxy resin matrix and the aligned *Cynara scolymus* fiber plies and then the fibers are carefully laid in to a pre-treated mold. After this, the laminate is pressed and cured at ambient temperature by impregnation of the fibers with the epoxy resin followed by wetting and adhesion. This results in the demolding, trimming and testing of the cured composite to provide a controlled and reproducible flow of fabrication in determining effects of various counts of ply on the properties of the material.

Fig. 7 shows the three unique laminate structures that have been made in tensile testing: (a) Single Ply composite, which is made of one layer of fiber in the epoxy and then (b) the two-layered composite, two layers of fiber and (c) the three-layered composite, three layers of fiber. This gradual design was applied with the purpose to research the impact of reinforcement level or the number of layers (i.e. reinforcement material and laminate thickness) on the mechanical performance of the composite in a systematic manner so that the effects of increment in level of reinforcement on the material properties like tensile strength, stiffness and load distribution could be analyzed.

4. Mechanical characterization of biocomposites

4.1. Tensile testing

Tensile tests were performed using a Zwick/Roell Z050 electronic universal testing machine with a capacity of 50 kN (Fig. 8), in accordance with ASTM D3039/D3039 M, specifically adapted for the characterization of polymer matrix composite materials. The force sensor (dynamometer) is connected to a data acquisition system enabling the simultaneous recording of time, displacement, load, and strain. The data obtained are stored and processed by computer using TestXpert V9.01 software [36].

This test aims to evaluate the mechanical behavior of natural fiber-epoxy composites subjected to tensile loading. The specimen is fixed between two clamps to ensure its stability before the application of an

Fig. 2. Water absorption process.

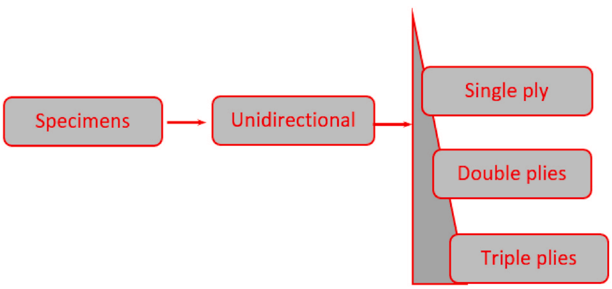


Fig. 3. Types of specimens.

Fig. 4. Epoxy resin and hardener used for the preparation of the matrix.

increasing load. The sample dimensions (cross-section and gauge length) are defined according to the standard. Force is applied gradually, while any change in length is recorded to determine the material's mechanical response, including its stiffness, maximum strength, and ductility [37].

4.2. *Impact resistance tests (Charpy/Izod)*

The impact tests were performed using a Zwick/Roell HIT pendulum

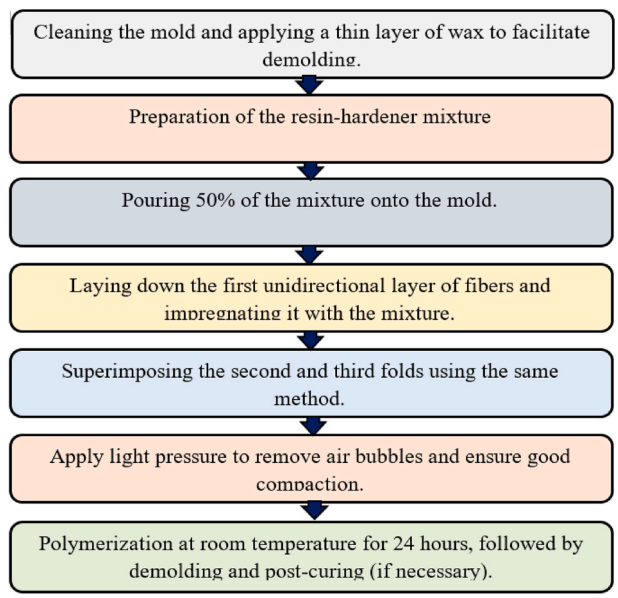


Fig. 6. Steps in the manufacturing of unidirectional laminated composite specimens.

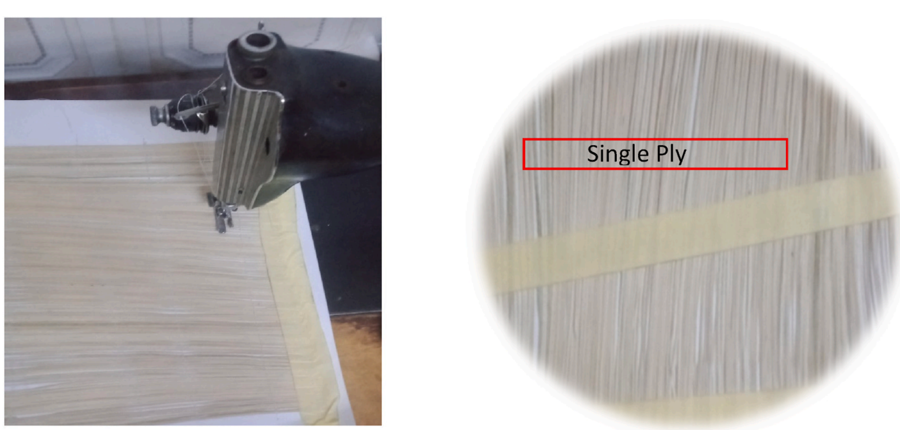


Fig. 5. Preparation and alignment of unidirectional fiber plies.

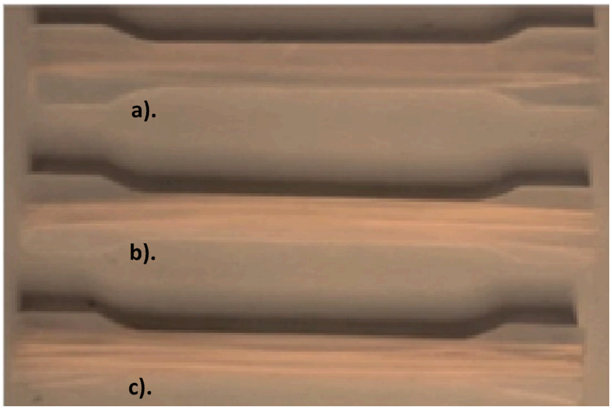


Fig. 7. Types of specimens for the tensile test. (a) Single Ply, (b) Double plies and (c) Triple plies.

Fig. 8. Zwick/Roell Z050 universal tensile testing machine.

impact tester (Fig. 9), in accordance with ASTM D 4814. The instrumented pendulum applies a controlled-energy impact, and the absorbed energy is calculated from the difference between the initial energy and the residual energy after fracture.

The recorded parameters (absorbed energy, impact velocity, maximum force, fracture mode) allow for the evaluation of the impact resistance of natural fiber-epoxy biocomposites. This type of test is widely used to characterize the toughness and dynamic behavior of bio-based composites [38].

For each natural fiber-epoxy composite formulation, three specimens were tested. This approach ensures the reproducibility of the results and allows for averaging the measured values, which reduces the effects of random variations and provides more reliable data on the material's resilience. The impact test according to ASTM D 4814 characterizes the composite's ability to withstand impacts and absorb energy in the event of a violent impact. The results of these tests are important for evaluating the material's resistance under real-world conditions, particularly in applications where the material may be subjected to shocks or dynamic loads [39,40].

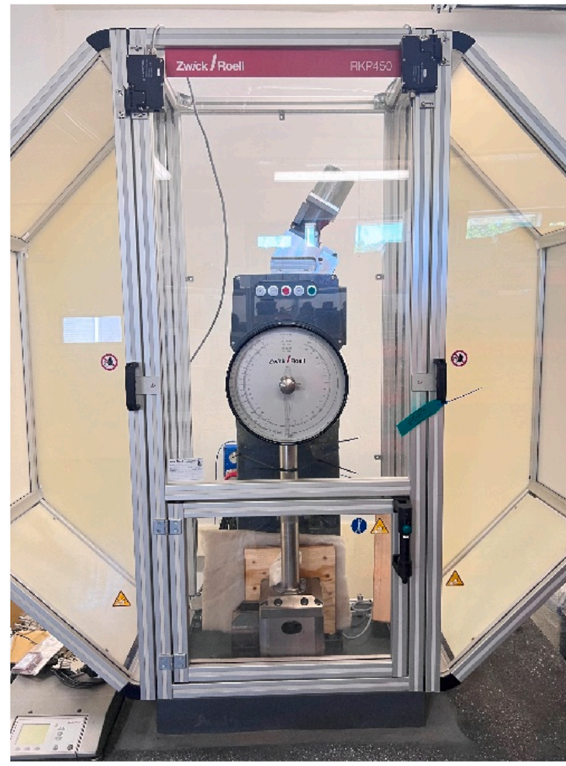


Fig. 9. Mouton pendulum impact tester, Zwick/Roell model, used for the impact test.

5. Results and discussion

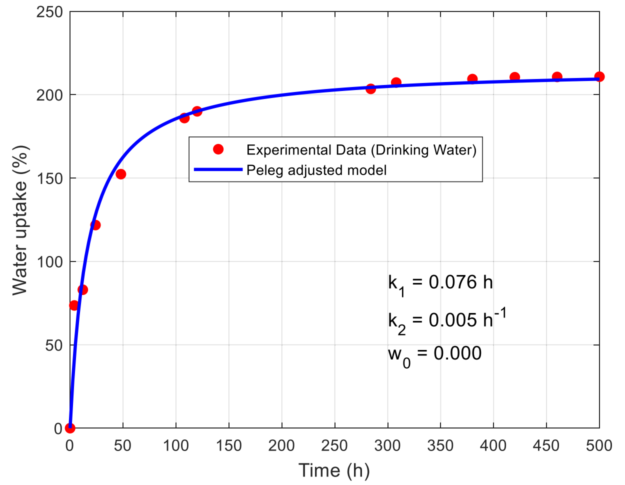
5.1. Water absorption results

Fig. 10 compares the experimental kinetic absorption of water using cynara scolymus fibers with the Peleg model prediction of water of three water types; drinking water, sea water and mineral water. The Peleg model which is expressed as in equation (3) is a close match with the experimental results showing its dependability in explaining the time-related absorption behaviour of moisture in these natural fibers. The findings show that there is a common trend in water uptake by water type, whereby there is an initial steep uptake phase, that is gradual to the approaching equilibrium phase which is typical of lignocellulosic fibers. This good consistency justifies the application of Peleg model in foretelling the hygroscopic behavior of the artichoke based fibers in diverse environmental factors which is significant in determining whether they can be used in composite applications where water resistance is an issue.

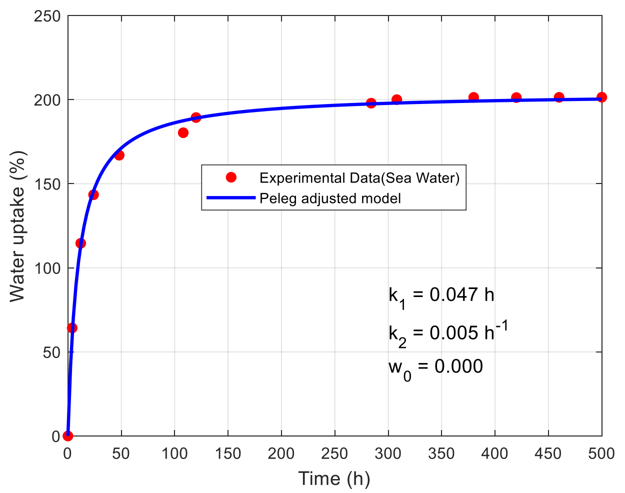
A graphical representation of the behavior of water absorption in *Cynara scolymus* fibers is given in Fig. 11, showing the way moisture uptake varies with time. A typical plot is that of an extremely fast absorption phase, which is then slowly followed by a steady approach to saturation, which is attributed to the hydrophilic nature of lignocellulosic fibers because of their composition of cellulose and hemicellulose. This graphical representation can determine the affinity of the fibers to the water substance which is a vital element when utilizing composite materials since moisture which is in excess amounts deteriorates the interface of the fibre-matrix, and compromises the mechanical characteristics. The data emphasize the need of fiber treatment or composite design to reduce the sensitivity to water in the structure use.

5.2. Weibull distribution

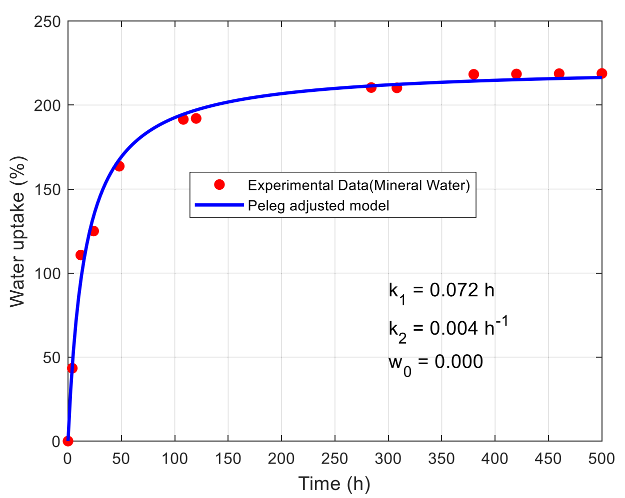
A graph that illustrates the Weibull distribution of the *Cynara*



a. Drinking Water



b. Sea Water



c. Mineral Water

Fig. 10. Comparison of experimental water absorption results and those obtained using the Peleg model.

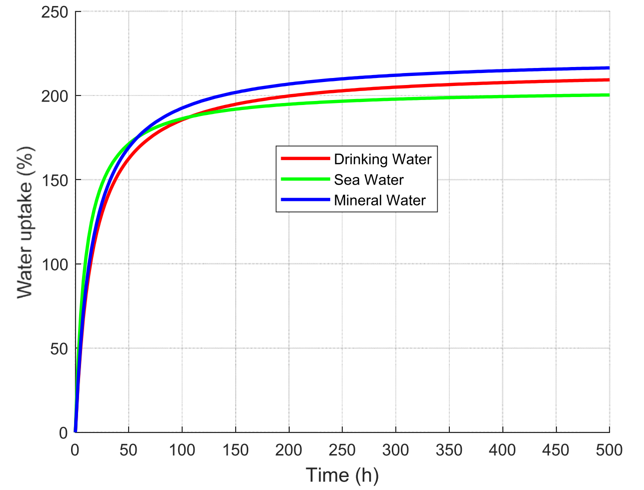


Fig. 11. Graphical representation of water absorption by *Cynara scolymus* fibers.

scolymus fibers length was done (Fig. 12) in which the lengths were analyzed to describe the variability and dependability of the lengths. The plot indicates that the length of fibers is distributed by Weibull distribution, where the shape parameter (β) is approximately equal to 2.5, and the scale parameter (η) is approximately equal to 7.5 cm, showing the medium distribution and characteristic length of about the mean. The cumulative probability curve increases progressively meaning that a significant proportion of fibers are within the range of 5 cm to 10 cm and the probability of short fibers (less than 3 cm) and long fibers (more than 12 cm) is low. The presence of this distribution validates the fact that the extracted fibers are relatively consistent in terms of length, which is critical in terms of uniform reinforcement of the composite materials.

The length of *Cynara scolymus* fibers was analyzed by the Weibull distribution as shown in Fig. 13 that displays the survival probability plots. The plot depicts the likelihood of each fiber to be longer than a certain length demonstrating an initial dramatic drop in the probability of survival to the shorter fibers and a more gradual fall to longer fibers. The curve shows that a very small percentage of the fibers (perhaps less than 20-30 percent) survive at the higher length values (e.g. greater than 9-10 cm), which is due not only to the natural variability, but also to the fact that shorter lengths of fibers are more common in the batch. This statistical characterization is a positive indication that the fiber population is not homogeneous and shorter length predominates, which is also extremely important in terms of reinforcement efficiency and composite processing.

Fig. 14 shows Weibull distribution curves of the mass of *Cynara scolymus* fibers, which shows the statistical data dispersion of fiber mass. The plot reveals that the fiber weights are normally distributed by the Weibull distribution with most of the values falling below the lower range (around 0.004-0.020 g), as seen by the sharp inclination and later flattening off of the cumulative distribution function. The natural heterogeneity of the fibers is proven by the shape parameter (β) and scale parameter (η) obtained on the basis of the curve, which are the measures of the dispersion and characteristic weight, respectively. This distribution is critical in the determination of the uniformity of the reinforcement material and in estimating the effect it has in the composite properties.

Fig. 15 shows the survival plots of the weight distribution of the *Cynara scolymus* fibers as obtained by Weibull analysis. The plot is used to depict the likelihood of a fiber to be heavier than a certain value, essentially the percentage of the fibers that remained intact after a certain weight was reached. Based on the curve, it may be concluded that the most frequent fibers will be relatively light, and the probability

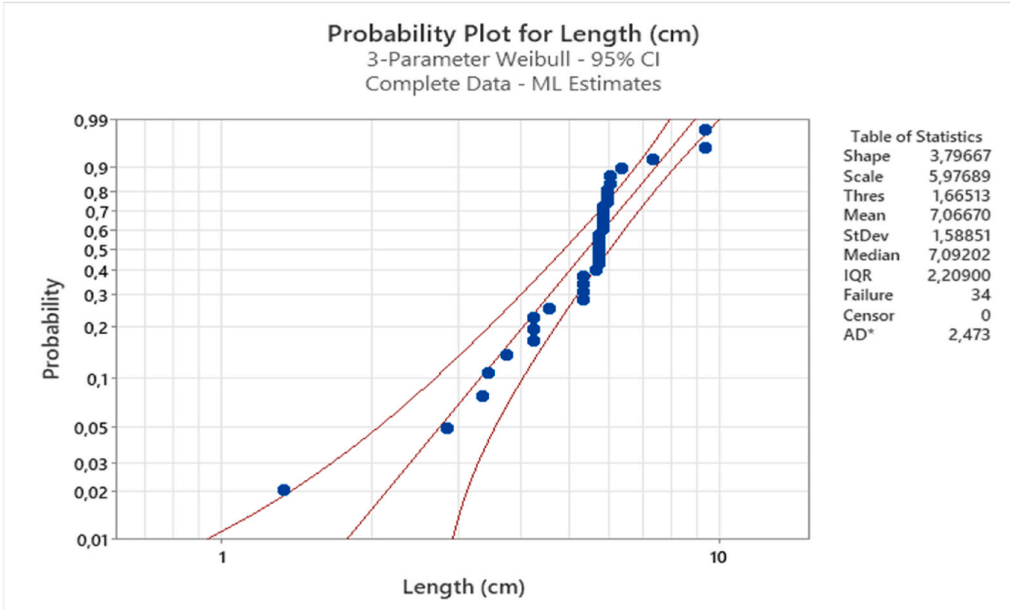


Fig. 12. Weibull distribution curves of length for *Cynara scolymus* fibers.

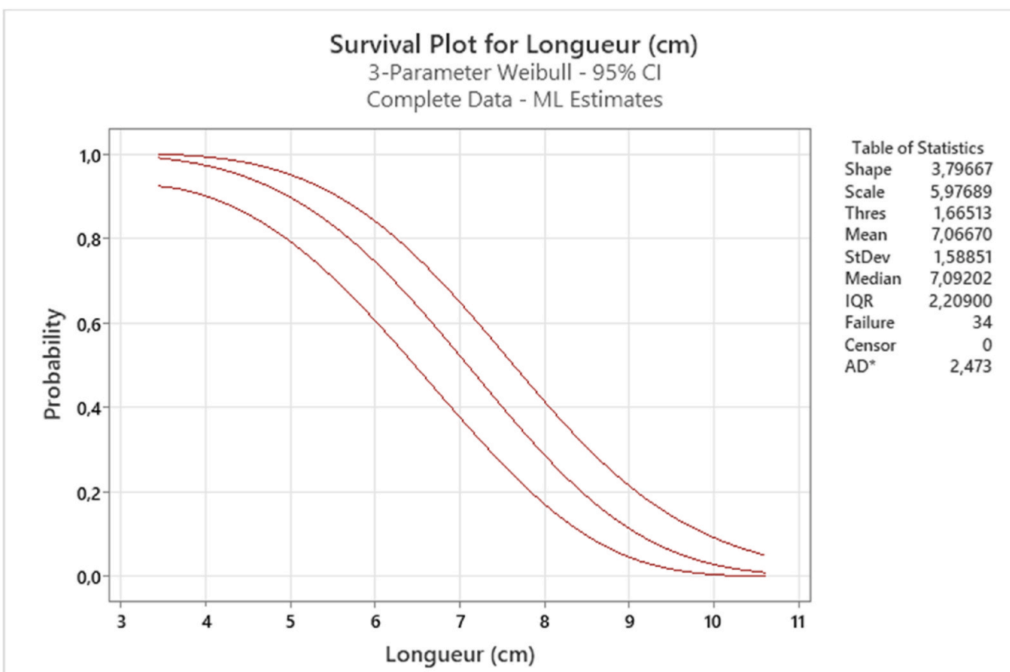


Fig. 13. Survival plots of length for *Cynara scolymus* fibers.

of survival will decrease widely with an increase in weight. An example would be that the plot with a very low weight (e.g. less than 0.005 g) would have a high probability of survival (near 100%), which would decrease dramatically as the weight goes closer to a value of 0.02 g, and only a minor fraction of the fibers would be heavier. This is a natural behavior of the extracted artichoke fibers as they are naturally variable and lightweight in nature which is similar to their application as low-density reinforcements in biocomposites.

Fig. 16 indicates the curves of Weibull distribution of the *Cynara scolymus* fiber weight, which reflects the statistical variability and reliability of the weight of the fibers. This is a characteristic curve whose scale parameter (which is probably the mean fiber weight) and the shape parameter expounding the variability of weights. As example, when the scale parameter is close to the mean weight (say, that is, 0.01 g), the distribution implies that the majority of fiber weights are centered about that value, and the shape parameter (assuming it is greater than 1) implies that the distribution is skewed towards heavier values, indicating natural variation of fiber morphology. This discussion validates the fact that the weight of the fibers are Weibull distributed which is applicable in the prediction of mechanical performance as well as consistency in fabricating composites.

The survival plots in Fig. 17 show the likelihood of a fiber of *Cynara scolymus* to have a weight greater than a specified value. The plot demonstrates that the probability to survive decreases gradually with

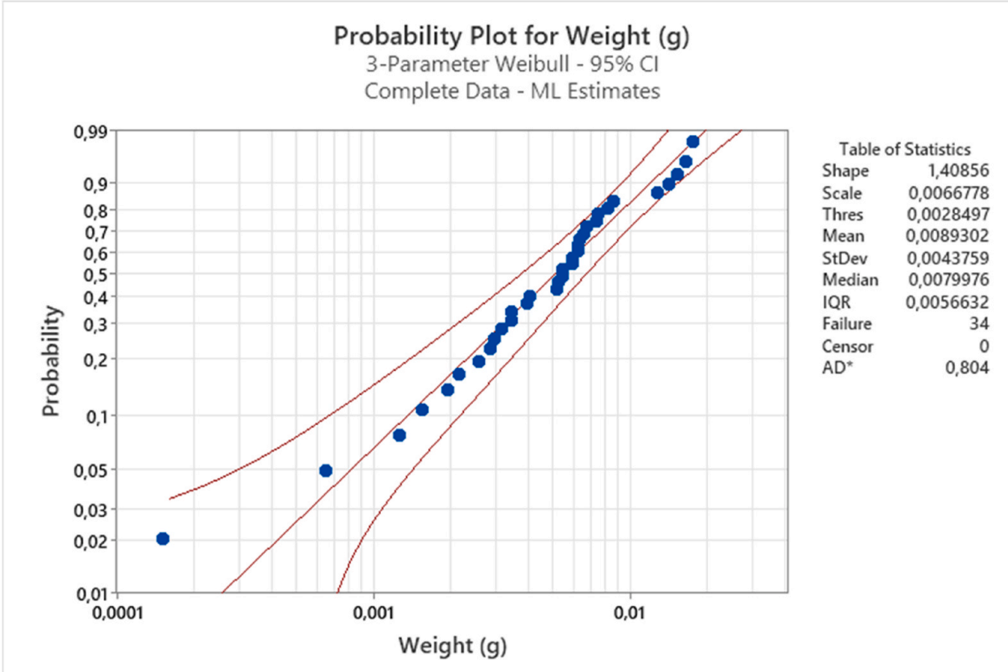


Fig. 14. Weibull distribution curves of Weight for *Cynara scolymus* fibers.

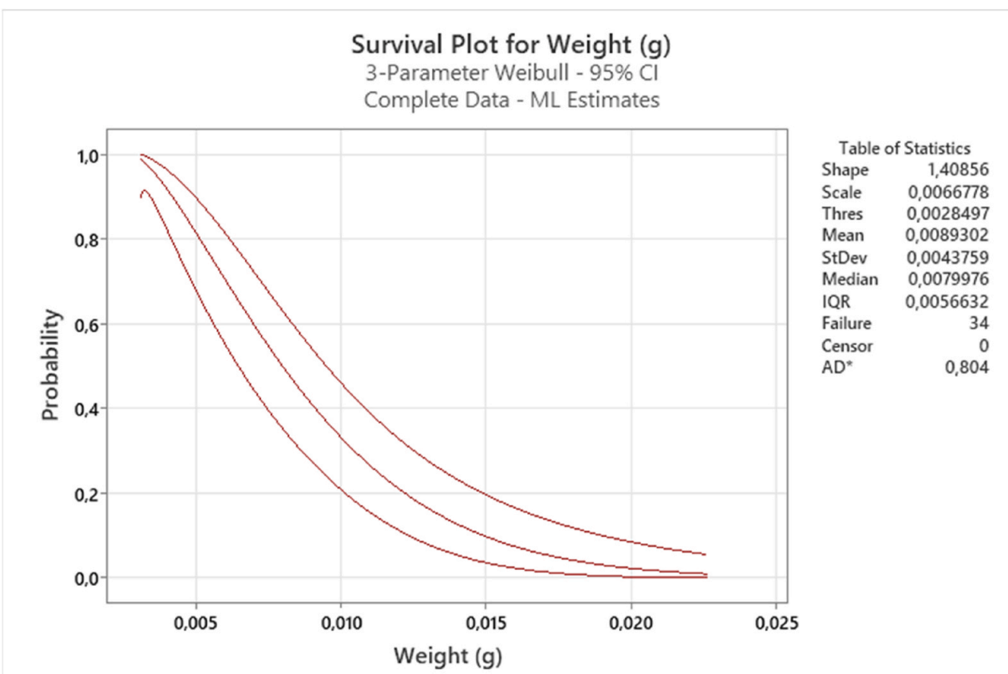


Fig. 15. Survival plots of weight for *Cynara scolymus* fibers.

the increase in the fiber weight, which means that the number of lighter fibers in the sample is higher. As an example, at very low weights (e.g., below 0.005 g) the survival probability will be approximately 100 percent, but then will drop rapidly, with only a finite percentage of the fibers surviving at weights around 0.015-0.020 g. This distribution is characteristic of the natural distribution of the mass of fibers, and a majority of them are relatively light (approximately 0.005-0.010 g) with occurrences of a few heavier fibers, which is also compatible with the heterogeneous distribution of natural fibers produced by agriculture.

5.3. Tensile testing

Fig. 18 depicts tensile stress-strain characteristics of unidirectional biocomposites with the reinforcement of *Cynara scolymus* fibers in single, double, and triple ply configurations. The curves show that the higher the number of fiber plies, the higher the mechanical performance of the composite: the single-ply composite has the lowest ultimate tensile strength (around 750 Mpa) and strain on break (around 2.2 %) and, respectively, the triple-ply composite is characterized by higher mechanical performance (around 1600 Mpa and 3000 Mpa) and a slightly

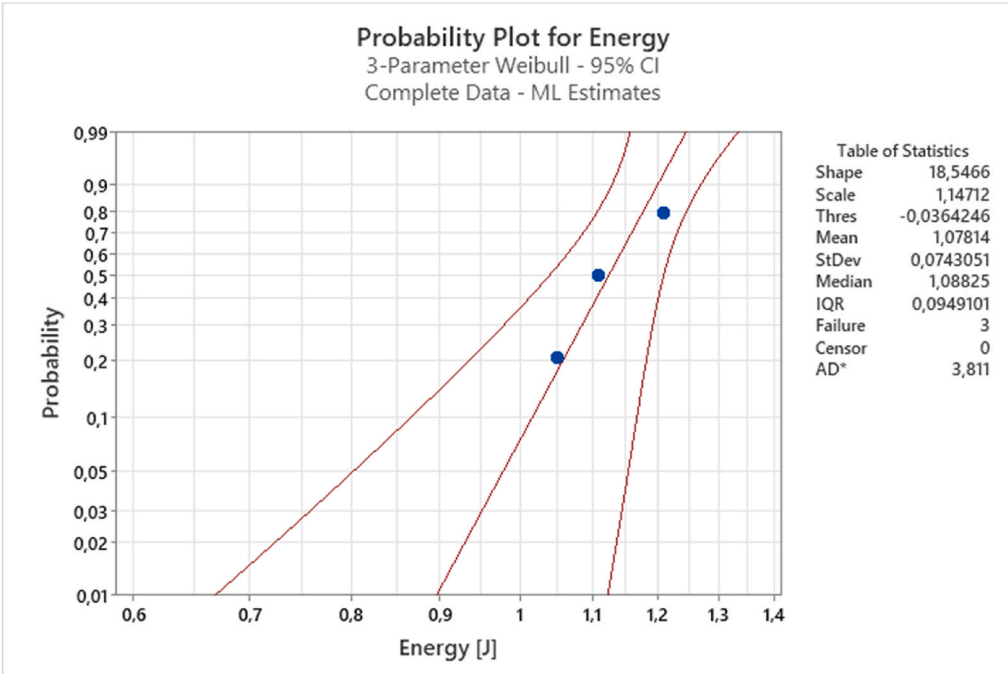


Fig. 16. Weibull distribution curves of Weight for *Cynara scolymus* fibers.

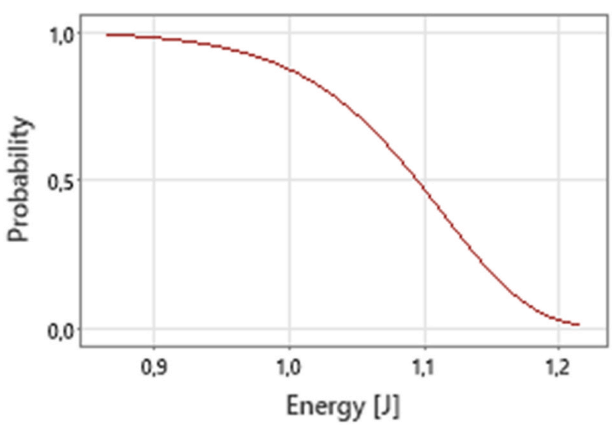


Fig. 17. Survival plots of Weight for *Cynara scolymus* fibers.

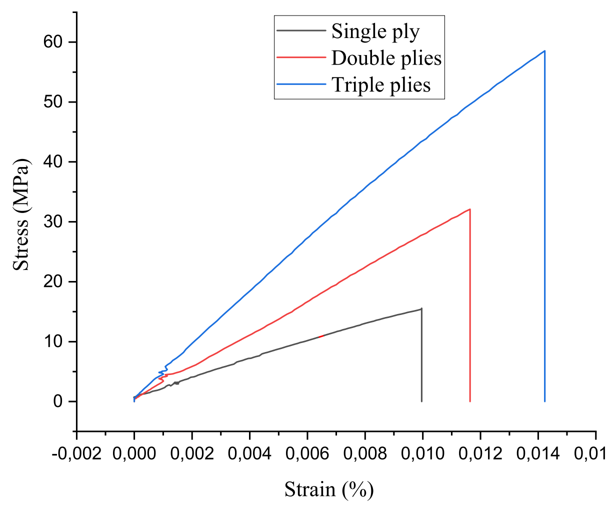


Fig. 18. Stress-strain curves obtained during tests performed on the three unidirectional (UD) biocomposite samples with Cynara Scolymus fibers.

higher ductility (around 2 % and 2.2 %). This tendency proves the increase in the number of fiber layers can enhance both stiffness and load-carrying capacity probably because of the improvement in the stress distribution and the augmentation of the fiber-matrix interaction, although the triple-ply system also reflects a slight increase in brittle behavior as the curve slopes steeper to breaking point.

5.4. Impact resistance tests (Charpy/Izod)

Fig. 19 shows the stressstrain graphs of three samples of unidirectional (UD) biocomposites reinforced by *Cynara scolymus* fibers on the influence of ply number on the mechanical behavior. The tensile strength of the single-ply composite is the lowest (about 25-30 Mpa) and the failure strain is comparatively low (about 2%). The two-ply design has significant improvement where tensile strength increases to approximately 40 Mpa and strain to failure is approximately 3 percent suggesting increased load bearing performance and ductility. The triple-ply composite exhibits the highest strength (approximately 50 Mpa) and the highest strain at failure (approximately 4 %), which proves that the number of fiber plies is the most effective way to enhance the stiffness and toughness of the composite, probably because of the enhanced stress distribution and superior adhesion of the fibers to the matrix.

5.5. SEM analysis

In the single-ply biocomposite, the SEM images (Fig. 20) show the main micro-architecture characteristic of the quality of the fiber-matrix interface. They present adhesive behavior overall of the *Cynara scolymus* fibers and epoxy resin which is primarily good, having few visible voids or debonding, indicating effective impregnation. The fibers seem to be properly dispersed and embedded in the matrix, but some small trace fibers tend to pull-out or pull-out surfaces are likely to be observed, which is typical of natural fiber composites. The no-large-scale defects are helping the noted mechanical performance in which the single-ply composite showed better stiffness and strength in comparison to pure epoxy in terms of tensile and impact test results.

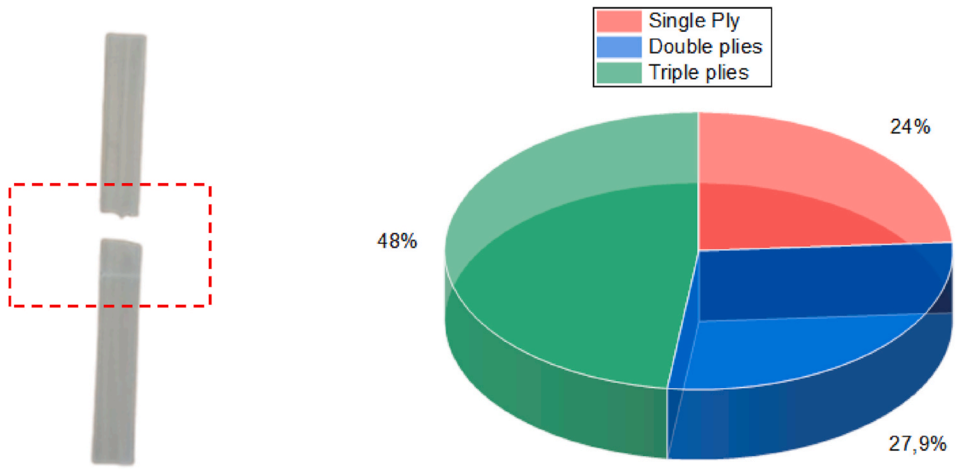


Fig. 19. Stress-strain curves obtained during tests performed on the three unidirectional (UD) biocomposite samples with Cynara Scolymus fibers.

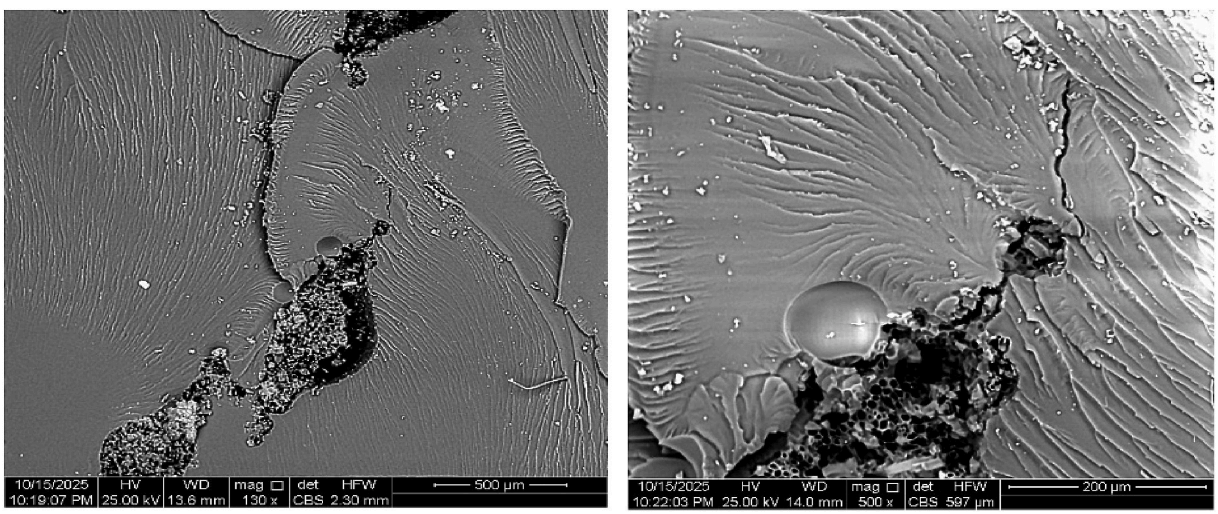


Fig. 20. Images obtained by scanning electron microscopy (SEM) of the single-ply biocomposite.

Fig. 21 displays SEM images of the double-ply biocomposite, where the fiber dispersion is not as irregular as in the single-ply one and interfacial adhesion is better than the single one. The pictures show reduced voids and improved bonding between the fibers and the matrix that probably led to the increased mechanical performance in the case of increased tensile strength and impact resistance as witnessed in the

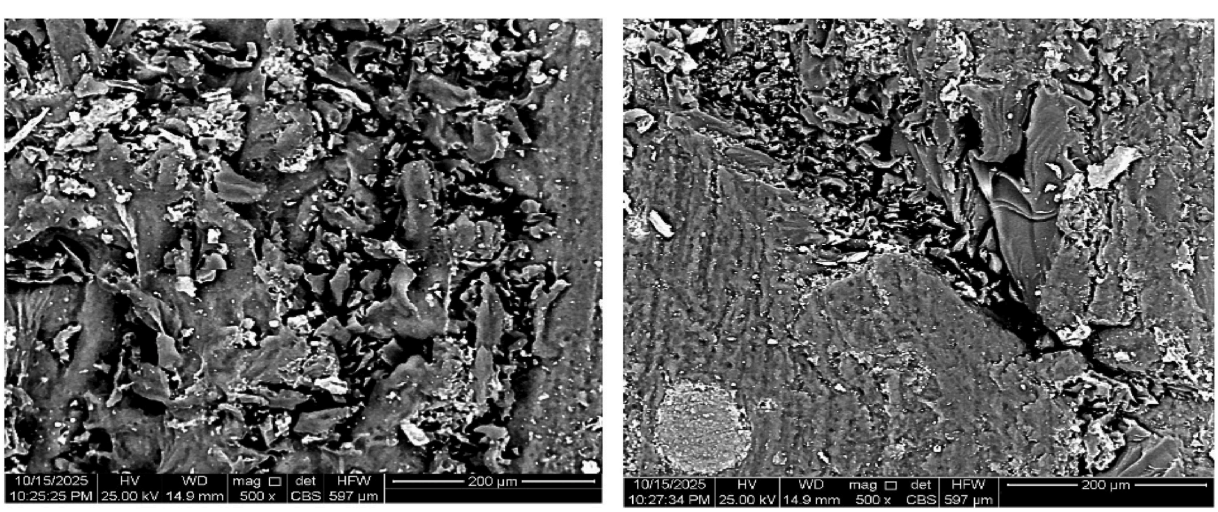


Fig. 21. Images obtained by scanning electron microscopy (SEM) of the double plies biocomposte

experimental data. The fibers can be seen to be well distributed in the epoxy matrix, which implies that the reinforcement and the epoxy transfer stress efficiently, which is a major consideration in the enhanced load carrying capacity and structural stability of the composite.

In the case of triple-ply biocomposite (Fig. 22), the SEM images indicate that the structure is densely packed and well impregnated with enhanced fiber-matrix adhesion than the lesser plies. The micrographs reveal a reduced number of visible gaps or holes at the intersection, meaning that the extra layer of *Cynara scolymus* fibers is wetted and placed into the epoxy matrix successfully. This increased integration is probably a part of the mechanical improvements, including increased stiffness and strength, through improved transfer of stress between the matrix and the reinforcing fibers and minimized the occurrence of defect sites, which might form the basis of failure.

5.6. EDX analysis

EMEB-EDX analysis of the outer surface of the fibers shows a predominance of carbon (56.49% by mass) and oxygen (34.05%), as well as other elements in small quantities, such as sodium (1.10%), aluminum (1.96), chlorine (0.34), potassium (0.26), and others.

This composition is typical of lignocellulosic plant fibers, mainly composed of cellulose, hemicellulose, and lignin, rich in hydroxyl groups (–OH) responsible for their hydrophilic character [41,42].

The high oxygen content reflects the presence of polar groups that promote hydrogen bonding with the epoxy matrix, but can also increase sensitivity to moisture.

In general, the surfaces of different lignocellulosic fibers are compared by their theoretical atomic ratio between oxygen and carbon (O/C ratio), which is 0.83 for pure cellulose, 0.33 for lignin, and the lowest ratio is 0.1 for extracts [43].

The EDX analysis of the *Cynara scolymus* fiber surface (Fig. 23) shows that it has a composition typical of lignocellulosic fibers in which carbon (56.49% by mass) and oxygen (34.05%), are predominant. The cellulose, hemicellulose and lignin content is consistent with the presence of these elements and the high proportion of oxygen groups are evidence of the numerous amounts of hydroxyl (–OH) groups that are present, which makes the fiber hydrophilic in nature. Minor elements observed are sodium (1.10%), aluminum (1.96%), chlorine (0.34) and potassium (0.26) elements. Although the oxygen-to-carbon (O/C), atomic ratio is not specifically determined in this case, based on these values one can infer oxygen-carbon ratios as well and compare them to the reference materials (e.g., 0.83 in the case of pure cellulose), which can help to understand the potential of interfacial bonding of the fiber with the epoxy matrix and its sensitivity to moisture.

For the epoxy resin alone, the elemental composition is dominated by carbon (82.51%) and oxygen (17.25%), consistent with the chemical structure of bisphenol A diglycidyl ether (DGEBA) and the curing agent. The low proportion of oxygen indicates a dense cross-linked structure after polymerization, giving the matrix good cohesion and low water permeability [44,45].

When the EDX analysis of the neat epoxy matrix (Fig. 24) is taken, it shows that the composition is largely carbon-oxygen, with carbon and oxygen occupying 82.51 and 17.25 % of the mass, respectively. Such elemental profile remains in line with the cured framework of a bisphenol A diglycidyl ether (DGEBA)-type epoxy resin, a very cross-linked polymer structure having minimal oxygen content compared to carbon. The resultant heavy, water-free system enhances the good cohesion as well as low permeability of the moisture to the matrix, which facilitates its function as a stable and protective phase in the composite system.

6. Conclusions

This work was also intended to design and define sustainable epoxy composite reinforcers using *Cynara scolymus* fibers, where Weibull statistical analysis was used to determine fiber consistency and systematic mechanical testing was used to determine the influence of fiber ply count. In direct correlation to this objective, the following are the conclusions made.

1 The retting and controlled extraction process were effective to produce *Cynara scolymus* fibers with the right surface features to be reinforced in the composite. It was shown that the fibers had a medium and fairly homogeneous length distribution with a Weibull scale of length being about 7.5 cm and characteristic weight of about 0.01 g, which is consistent over the whole reinforcement potential.

2 Peleg equation was successfully used to model the kinetics of water absorption of the fibers, which showed that the fibers are hydrophilic, as it is characteristic of lignocellulosic materials. The values were measured as moisture regain (M_r%) and moisture content ((Mc%) and the absorption tendencies were analyzed as stability at high initial rates and then equilibrium - that is of critical importance in estimating environmental durability in composite applications.

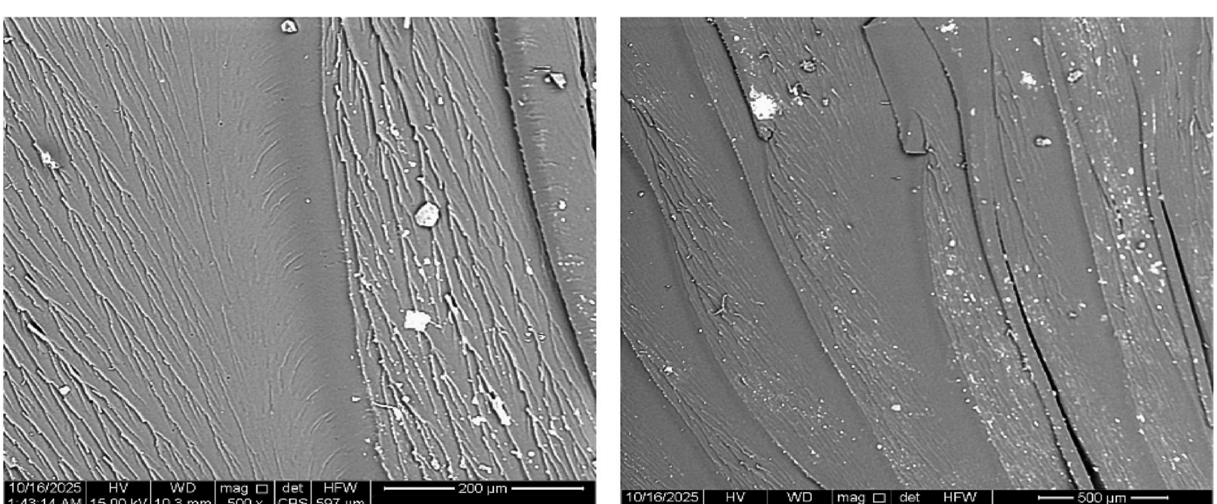


Fig. 22. Images obtained by scanning electron microscopy (SEM) of the triple plies biocomposite.

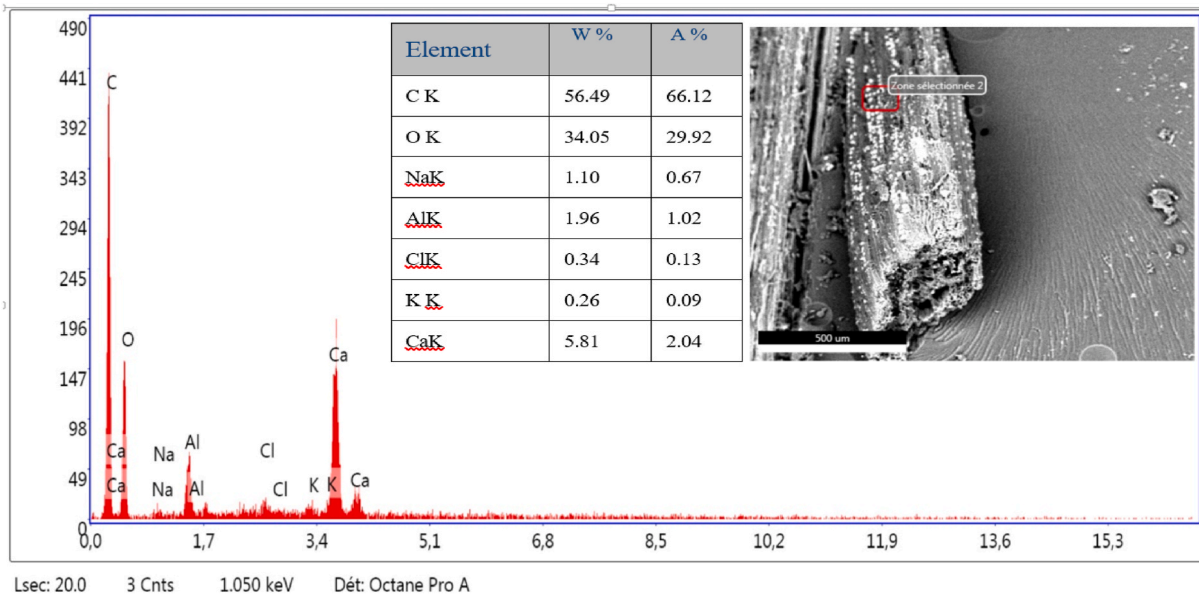


Fig. 23. EDX analysis for the fiber.

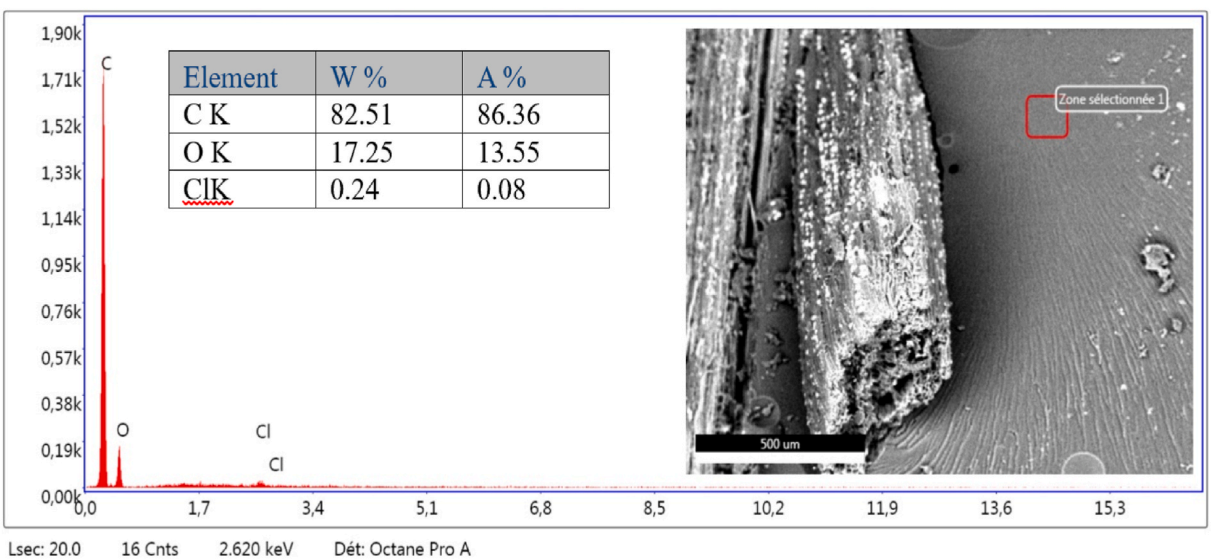


Fig. 24. EDX analysis for the matrix.

3 Tensile: There was an apparent positive relationship between mechanical performance and the number of fiber plies:
  - Single-ply composites were able to reach a final tensile strength of about 15 MPa and strain at break of about 1.5%.
  - The increase in the strength (~32 Mpa) and strain (~2 %) was found in the case of double-ply composites.
  - Triple-ply composites had the best strength (approximately 60 Mpa) and ductility (approximately 2.2%), which showed good load transfer and interfaces.

4 Impact tests (Charpy/Izod) showed that there was an increase in energy absorption and toughness with an increase in fiber content. The triple-ply design showed the highest impact resistance which could be explained by the more significant bonding of the fibers with the matrix and the distribution of stress in favor of the composite which could be applied in the case of dynamic or semi-structural.

5 SEM analysis found that there was good interfacial adhesion between the *Cynara scolymus* fibers and the epoxy matrix with little voids or debonding. The composite with triple-ply exhibited the least variation in the dispersion of the fibers and impregnation, which is associated with better mechanical features.

6 The analysis of EDX has shown that the fibers were primarily composed of carbon (56.49%) and oxygen (34.05%), which is typical of natural fibers rich in cellulose. The epoxy was a carbon and oxygen (82.51% and 17.25%, respectively) cross-linked, hydrophobic structure. The miscellaneous profiles above endorse the mechanical compatibility and water sensitivity that is observed.

7 Weibull distribution analysis confirmed that the length of fibers and weight had predictable statistical patterns, the shape parameters showed a moderate variation. Such reliability is justification of reproducibility of composite fabrication and performance prediction.

8 The composites developed had a good balance of low density, enhanced specific strength and reasonable moisture uptake. The application of artichoke stem fibers, which is a waste product of

agriculture, contributes positively to the ecological conditions and enhances the creation of renewable, lightweight, and environmentally friendly composite substances to be used in structures and semi-structural purposes.

9. The main issues were controlling the natural variability of the properties of natural fibers, ensuring uniform adhesion of the fibers to the matrix and controlling moisture absorption to retain dimensional and mechanical stability. The management of the void formations during hand lay-up and the dispersion of the fibers in several plies was also very challenging.
10. Later applications will involve chemical modification of fibers to give them greater hydrophobicity and interfacial adhesion. They will be long-term aging of the environment and the fatigue behavior as well as hybrid composites that combine synthetic/natural fibers. The processes of manufacturing processes and life-cycle assessments also demand scaling up and require commercial application and sustainability validation.

This research does not only show that the concept of *Cynara scolymus* fibers as a sustainable reinforcement is viable, but it also forms a research baseline on how to continue investigations into hybrid bio-composites, scalable production methods, and lifecycle analysis, which will further encourage the development of environmentally friendly structural materials.

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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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