Environmental Science and Pollution Research (2023) 30:52124–52140
https://doi.org/10.1007/s11356-023-25946-1

RESEARCH ARTICLE

Effect of surface treatment on the technological properties of coconut fiber–reinforced plant polyurethane composites

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Received: 23 November 2022 / Accepted: 10 February 2023 / Published online: 24 February 2023

Abstract

Polymeric composites reinforced with plant fibers have numerous advantages, such as low cost, high raw material availability and good physical, mechanical and thermal properties. Thus, in recent years, they have been studied as thermal insulation substitutes for synthetic polymers in buildings. The aim of this study was to evaluate the technological properties of castor oil–based polyurethane composites reinforced with coconut fibers treated with hot water, alkaline solutions of NaOH and $Ca(OH)_2$ and corona discharge and without surface treatment as materials for the thermal insulation of buildings. The composites were produced by the *hand lay–up* method followed by compression; 10% by weight coconut fibers were used to replace the synthetic polymer. Specimens were produced, and physical, mechanical, thermal and microstructural tests were performed. The results showed that the polymer had a thermal conductivity of 0.016 W/(mK), while the composites produced with fibers treated with NaOH had a thermal conductivity of 0.028 W/(mK); therefore, these polymers are considered insulating materials ($k = 0.01$ to 1.0 W/(mK)). Thus, the composites produced with coconut fibers can be considered as lighter, less expensive and environmentally friendly alternatives to synthetic polymers.

Keywords Thermoset matrices · Biocomposites · *Cocos nucifera* · Thermal conductivity · Mechanical properties · Coir fiber composites

Introduction

Currently, organic and inorganic insulating materials that have negative side effects from the initial production phase until the end of their useful life are popular (Gaspar et al. 2020). In addition, some insulating materials are manufactured with substances that are harmful to health and the human body, e.g., reconstituted wood panels that use adhesives containing formaldehyde, a proven carcinogenic substance (International Agency for Research on Cancer—IARC; EPA 2016). In addition to the effects caused to human health, the use of petroleum-derived materials as insulation in buildings is not an ecological alternative. Moreover, the incorrect disposal of these materials in dumps and sanitary landfills after use causes environmental pollution, with an estimated volume of 12 billion metric tons of plastic waste disposed of irregularly by 2050 (Geyer et al. 2017).

During the rounds of Climate Negotiations' held during COP 27 aimed at setting goals and implementing urgent actions on the climate, it was announced that civil construction emitted 37% of CO_2 emissions in 2021 (United Nations 2022). Thus, composite materials produced with polymeric matrices reinforced with natural fibers emerge as a more sustainable alternative to replace materials of petroleum origin in applications aimed at thermal insulation of buildings, providing a reduction in CO_2 emission in the atmosphere, as well as the use of waste in products with high added-value. In this way, the use of lignocellulosic wastes as reinforcement in composites promotes an improvement in mechanical and thermal performance, and a reduction in weight and cost, in

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addition to valuing the waste in a product with greater added value (Wang et al. 2022).

Among the intrinsic properties of plant fibers that justify their use as a reinforcement material, their high specific strength, renewable nature, and the fact that they generate large volumes at a relatively low cost stand out (Yussof et al. 2016). Among the numerous sources of vegetable fibers, those from green coconut stand out in the production of composites, mainly due to their excellent mechanical properties, with emphasis on elongation at break, in addition to their wide local availability, renewability and excellent strength/weight ratio (Sudhakara et al. 2013). Brazil has a prominent place in coconut production worldwide, reaching the fifth position among the largest producers, with a production of 2349 thousand tons in 2019 (Food and Agricultural Organization of the United Nations – FAOSTAT 2022). Despite the advantages of using plant fibers in composites, some challenges due to the nature of lignocellulosic fibers, mainly related to their chemical constituents, result in poor wetting by the polymer, and consequently reduced mechanical properties. These disadvantages can be overcome by modifying the fiber surface using chemical treatment methods, such as alkalization, acetylation and benzoylation (Selvi et al. 2022), and physical treatment methods, such as the use of corona discharge, plasma and ultraviolet radiation (Sudhakara et al. 2013; Abidin et al. 2019).

Depending on their application, it is sometimes necessary to improve the mechanical properties of fibers through pretreatment. Pretreatment promotes improved compatibility between reinforcement and matrix by increasing interfacial adhesion between both, in addition to reducing fiber agglomeration in the matrix (Sanal and Verma 2019). Among the chemical treatments carried out on vegetable fibers, alkalization, or mercerization, stands out among the most used in the production of composites. The alkaline treatment acts by reducing chemical constituents that inhibit interfacial adhesion between polymer and vegetable fiber, such as extractives and lignin. Thus, there is disruption of hydrogen bonds and consequent increase in roughness, resulting in anchoring of the polymer in the grooves of the vegetable fibers and adequate stress transfer (Sanal and Verma 2019). In terms of physical methods, one of the most popular physical treatments is corona, which is used for surface oxidation activation. This is achieved by changing the surface energy of the cellulose fibers by means of an electrical discharge.

Several polymer matrices, especially those containing polyurethane, which is considered one of the best thermal insulators, have been used in the production of thermal insulating materials for buildings. Polyurethane is a polymer composed of polyol and prepolymer, which are usually of petroleum origin. The development of polyurethane derived from castor oil was first proposed in studies in the 1940s as a more sustainable alternative produced with a low-cost and renewable source material (Vilar 1993). Several authors have evaluated the thermal conductivity of composites produced with polyurethanes obtained with castor oil-based polyol reinforced with plant fibers (Carriço et al. 2017; Zanini et al. 2021; Carvalho et al. 2022) and found their behavior to be as good as thermal insulation materials. Faria et al. (2020b) evaluated the physical and mechanical properties of polymeric composites produced with coconut fibers and polyurethane matrix based on castor oil, in which they observed improvement in mechanical strength as higher fiber contents were inserted in the composites. A similar study was carried out by Carvalho et al. (2022) in which the authors evaluated the replacement of the polyurethane matrix based on castor oil by piassava fibers for application as floors, and observed satisfactory resistance to abrasion for long periods without wear loss. However, there are no studies in the literature using coconut fibers treated under different methods in vegetable polyurethane matrix composites aiming their application as thermal insulation in buildings.

Based on the above studies, the aim of this study was to evaluate the use of coconut fibers treated with hot water, alkaline solutions of NaOH and $Ca(OH)_2$ and corona discharge and without surface treatment in castor oil–based polyurethane composites as materials for thermal insulation in buildings, promoting the valuation of coconut crop residues in products with higher added value and obtaining an alternative environmentally friendly composite.

Materials and methods

Materials

Coconut fibers purchased from Nutriplan® (Cascavel, Paraná, Brazil) were used in this study. The density of the coconut fibers was 1200 kg/m^3. NaOH (Dinâmica® Química Contemporary Ltd.) with a purity greater than 98%, $Ca(OH)_2$ (Êxodo Científico Ltda.) with a purity greater than 98% were purchased from CS Científica (Lavras, Minas Gerais, Brazil). The castor oil–based plant polyurethane (AGT 1315) was supplied by IMPERVEG® (Aguaí, São Paulo, Brazil). The physicochemical properties of the plant polyurethane resin are shown in Table 1.

Surface treatment of coconut fibers

Before each treatment, the coconut fibers were ground using a hammer mill, sieved through a 4.76 mm (4 mesh) sieved and retained in a 2.38 mm (8 mesh) sieve. After the fibers were sieved, they were dried in an oven at 105 ± 3 °C until a moisture content of 3% was obtained.

Table 1 Physicochemical parameters of the plant polyurethane polymer

Parameter	Polyurethane plant
Apparent viscosity (25 °C)	0.43063 Pa·s
Solid content	79.43%
pH	7.0
Density	1125 (kg/m^3)

Coconut fibers were treated with hot water, NaOH, $Ca(OH)_2$, and corona discharge. Hot water treatment was used because it is less aggressive to the fiber, solvent chemical free (such as alkaline solution) as well as minimized weight loss of lignin and cellulose. Whose objective is only the removal of superficial extractive (waxes) (Moura et al. 2019). The thermochemical treatment was performed according to the methodology of Leão et al. (2015), using a beaker containing the fibers with 1 L of deionized water, in which the mixture was heated at 80 °C for 2 h, then the fibers were dried in an oven at a temperature of 105 ± 3 °C for 24 h.

The procedure for the alkaline treatment of the fibers was similar to the process described by Suardana et al. (2011). After drying, the fibers were placed in glass beakers with a capacity of 2.5 L. Pre-weighed solutions of NaOH and $Ca(OH)_2$ with a concentration of 5% by weight were inserted into the containers, separately, then the beakers were placed in heaters at 95 °C for 1 h. Subsequently, the fibers were washed with deionized water until the dripping water of the fibers had a pH of approximately 7.0. After being washed, the fibers were dried in an oven at 105 ± 3 °C for 24 h. The mass losses of the dry fibers were determined to confirm the efficiency of the treatments.

Corona discharge treatment (Plasma-Tech, model P-1, Corona Brazil Ltda.) was performed for 5 min, using a potential of 12 kV, current of 60 mA, and frequency of 60 Hz. The discharge was carried out in air (25 ± 3 °C, 70 ± 5% relative humidity) at an average distance of 2 cm between the sample and the electrical source.

Characterization of coconut fibers

The aspect ratio of the coconut fibers was determined using ImageJ software. Thirty measurements were performed, and the length and diameter of the fibers were obtained for subsequent calculation of the aspect ratio using Eq. (1).

$$AR = \frac{L}{D} \quad (1)$$

where AR is the aspect ratio, L is the average length of the coconut fibers (mm) and D is the average diameter of the coconut fibers (mm).

To determine the chemical constituents, the coconut fibers were ground in a hammer mill and then sieved through 0.420 mm (40 mesh) and 0.250 mm (60 mesh) sieves using those retained in the 60 mesh sieve.

The total extractive content was determined according to the NBR 14,853 standard (Brazilian Association of Technical Standards—ABNT 2010); the insoluble lignin content was determined according to the NBR 7989 standard (ABNT 2010); the ash content was determined according to the NBR 13,999 standard (ABNT 2017); and the holocellulose content was determined according to the procedure described by Browning (1963).

Before carrying out the tensile test, the coconut fibers were stored in a climate-controlled room (20 °C and 65% relative humidity). Tensile strength, Young's modulus and elongation at break properties were determined based on standard D3379-75 (American Society for Testing and Materials—ASTM 1989). A more detailed description of the test can be found in Faria et al. (2020a).

Production of composites

The resin was obtained after mixing the prepolymer with the polyol in a ratio of 1:1.5 after being mixed at 150 rpm in a beaker for 5 min under vacuum. Then, the mixture was poured into a metal mold, left for 2 h, and subjected to compression molding (4.0 MPa, 22 °C, 24 h).

The production of the composites was based on the work by Merlini et al. (2011) and Faria et al. (2020b), who mixed the fibers with the prepolymer under magnetic stirring in a beaker at 150 rpm for 5 min under vacuum (Fig. 1). Then, polyol was added. Subsequently, the mixture was poured into a 200 mm × 200 mm × 3 mm metal mold. The composites were prepared by manual lamination, followed by compression molding at room temperature with a pressure of 4.0 MPa. The curing time of the composites was 12 h at room temperature (approximately 22 °C). The experimental design is shown in Table 2.

After removal from the press, the specimens were taken to a climate-controlled room with a constant temperature of 20 ± 2 °C and a relative humidity of 65 ± 5% for 7 days, ending the solidification period of the plant polyurethane resin.

Characterization of composites

Apparent density

The apparent density of the resin and composites was determined by dividing the mass (measured on an analytical balance with an accuracy of 0.01 g) of the specimens by the calculated volume of the specimens (base area multiplied by height, measured with a caliper). To determine the density

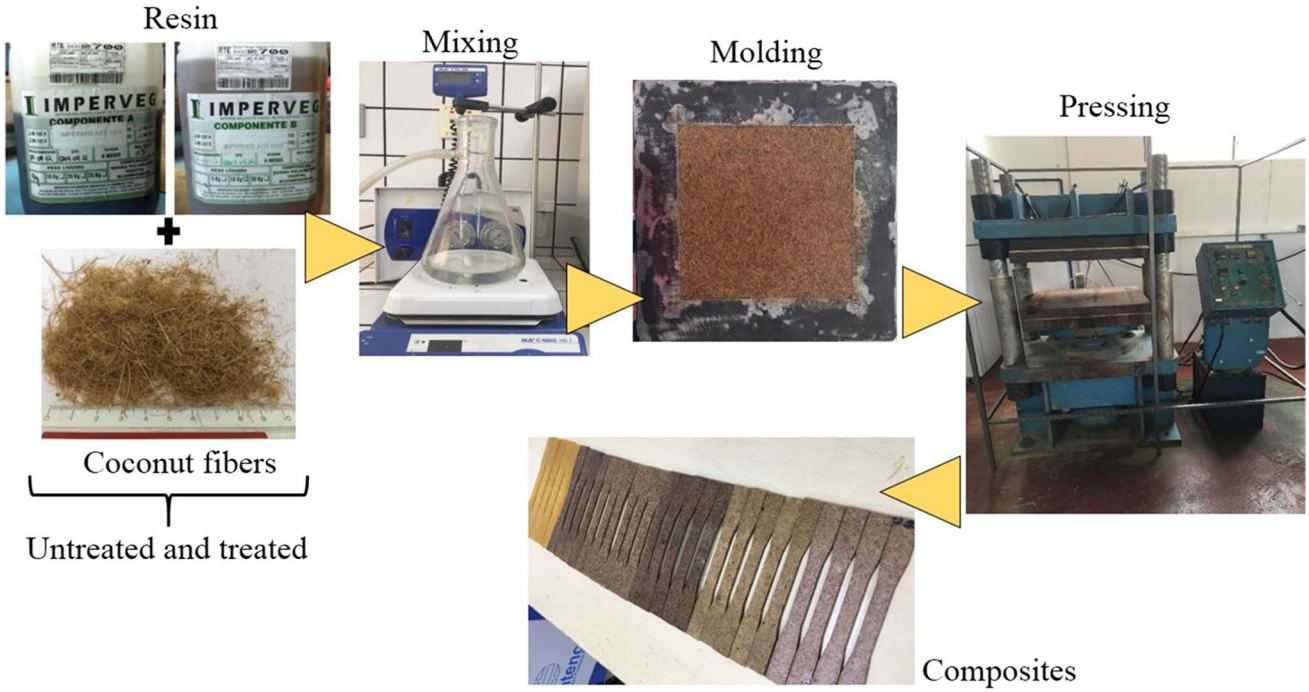

Fig. 1 Composite molding process. Adapted from Faria et al. (2020b)

Table 2 Experimental design of the composites produced

Composition	Volumetric fraction (mass%)*	Number of samples
Resin	- -	3
Without treatment	10	3
Hot water	10	3
Alkaline treatment (NaOH)	10	3
Alkaline treatment ($Ca(OH)_2$)	10	3
Corona discharge	10	3

*Volumetric fractions based on the work by Moura et al. (2019)

of the resin and the composites, five specimens of each volumetric fraction were analyzed.

Void content

The void content was calculated based on the theoretical and experimental densities of the resin and the composites produced, according to Eq. (2) (D2734-16 ASTM 2016).

$$\text{Void content} = \left(\frac{\rho theorical - \rho expiremental}{\rho expiremental}\right) \times 100 \quad (2)$$

The experimental density used was calculated in the "Apparent density" section, while the theoretical density was measured using Eq. (3).

$$\rho \text{ theoritical} = \frac{1}{\frac{wc}{\rho c} + \frac{wp}{\rho p}} \quad (3)$$

where wc and wp are the volumetric fractions of the coconut and resin fibers, respectively, and ρc and ρp are the densities of the fibers and resin, respectively.

Water absorption

The water absorption of the resin and the composites after 24 h of immersion was determined according to the procedure in standard D570 (ASTM 2018). To perform the test, the specimens were modified to form a rectangular shape with dimensions of 30.0 mm × 12.0 mm × 3.0 mm (length, width and thickness, respectively) for sampling. A total of 30 specimens were used to determine the water absorption of the resin and composites, with five specimens of each composition.

Tensile strength

The resin and composites were evaluated by tensile testing according to the methodology described in standard

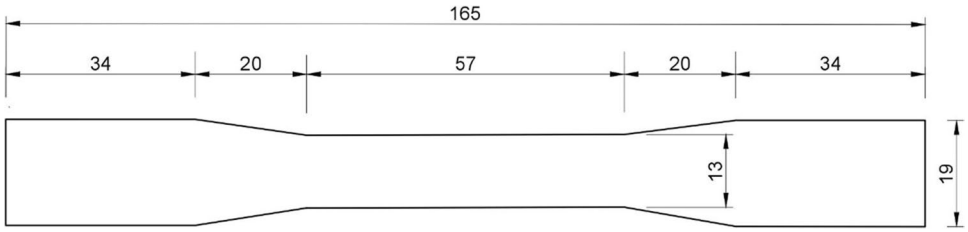

Fig. 2 Type 1 tensile specimens (D638-14, 2014). Units are reported in millimeters

D638-14 (ASTM 2014). The type 1 specimens (Fig. 2) were broken in an AROTEC universal testing machine (SP, Brazil), with a 20 kN load cell and a test speed of 2 mm/min. To determine the tensile strength, Young's modulus and elongation at break of the resin and the composites, six specimens were analyzed.

Wettability

For the analysis of wettability, the contact angle measurements on the longitudinal surface of the resin and the composites were performed using the sessile drop method and a Krüss DSA25 instrument with the DSA3 software module (Hamburg, Germany). In this test, distilled water was used.

The drop deposited on the material had a volume of 10 µL, and the measurements were performed every 1 s for 60 s. The initial contact angle (θA) was recorded after 5 s, and the final contact angle (θA) was recorded after 60 s. For each composition, 25 θA measurements were made, totaling 150 measurements.

Thermogravimetric analysis (TGA)

The thermal degradation of the resin and composites was determined using a DTG Shimadzu 60 thermogravimetric analyzer (Kyoto, Japan). Approximately 10 mg of powder sample was placed in the analyzer and heated to 900 °C at a heating rate of 10 °C/min in a nitrogen atmosphere (flow rate = 60 mL/min).

Surface morphology

The surface of the fibers and fractured composites were analyzed by scanning electron microscopy (SEM). For this, the specimens were covered with a layer of gold in an evaporator and analyzed in a scanning electron microscope LEO EVO 40 XPV operated at 20 kV.

Thermal conductivity

The thermal conductivity of the resin and composites was determined using two overlapping chambers made of expanded polystyrene with a thickness of 2.5 cm each. To avoid any interference with the external environment, 2.0 cm thick expanded polystyrene plates coated with aluminum foil were used internally. At the bottom of the lower chamber, a 25 W incandescent lamp was inserted and connected to a temperature controller module (Thermostat W1209), which maintained the incident temperature of the sample at 60 °C. Thermocouples were placed near the upper and lower sides of the sample. To ensure the stability of external climatic conditions, the apparatus was placed inside a climatic chamber (20 ± 3 °C; relative humidity 65 ± 5%). The test began after the upper chamber remained at a stable temperature for a period of approximately 1 h. The experimental apparatus can be seen in Fig. 3.

The thermal conductivity was calculated based on the Fourier law using Eq. (4) (Bergman et al. 2018). The heat flux emitted by the lamp was obtained using a solar radiation meter with three readings.

$$K = \frac{qx'' \times t}{DT} \tag{4}$$

where K is the thermal conductivity of the sample (W/(mK)); qx " is the heat flow (200 W/m^2); t is the thickness of the specimen; and DT is the temperature variation on the upper and lower sides of the specimen (K).

Statistical analysis

The data were analyzed considering completely randomized designs to evaluate the properties of the fibers and composites. The results were subjected to analysis of variance (ANOVA) and Tukey's test at 5% significance to evaluate the effect of the surface treatment of the fibers and their insertion in the composites. The data were processed using Sisvar 5.6.

Results and discussion

Characterization of coconut fibers

The mechanical properties of the composites depend on several factors, such as the length of the fibers, the way in which they are loaded and their orientation in the matrix. According to Faria et al. (2020b), the mechanical properties of polymer composites are directly influenced by the aspect ratio of the

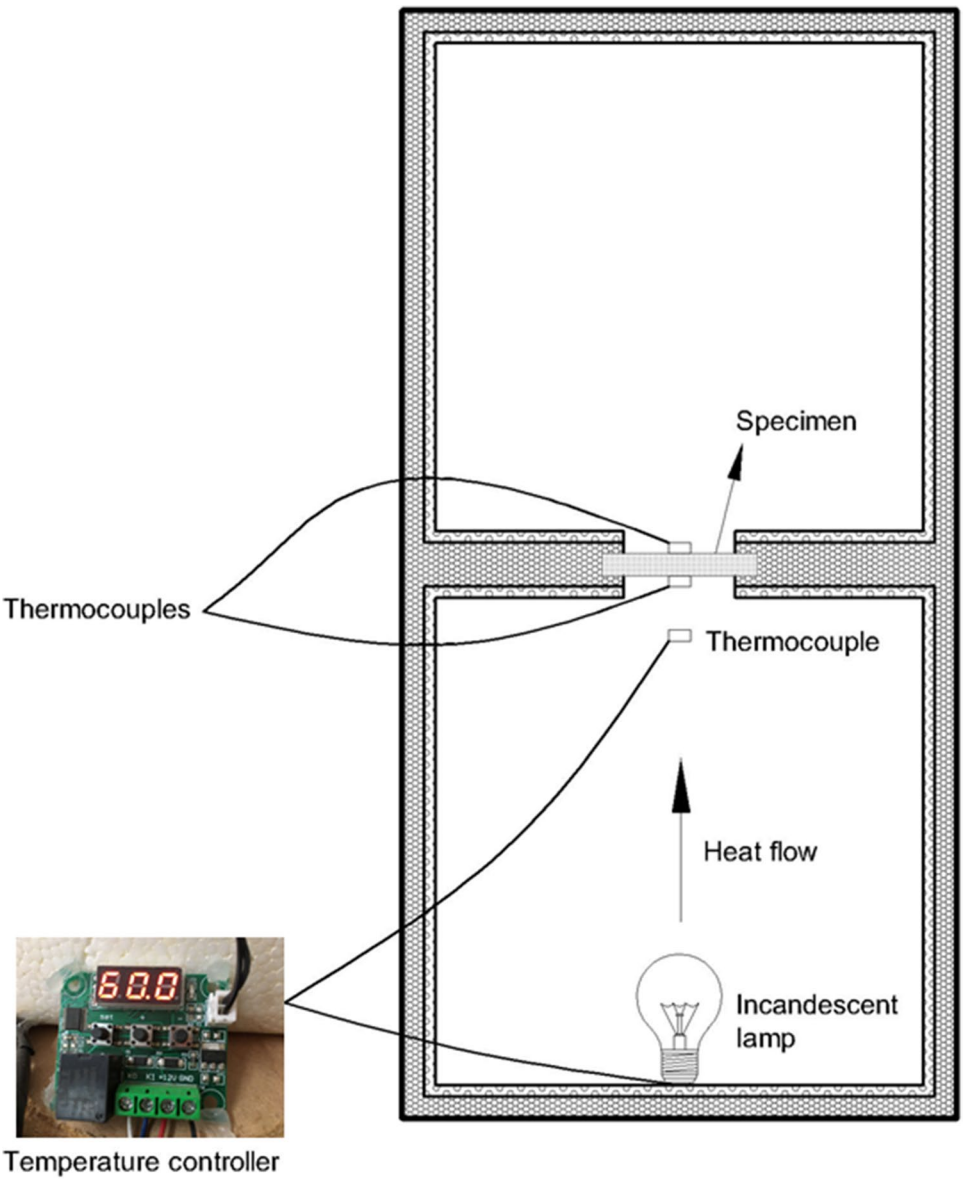

Fig. 3 Experimental apparatus used to determine the thermal conductivity

fibers. Thus, characteristics such as aspect ratio, direction and orientation of fibers and interfacial adhesion between fiber and matrix play an important role in load transfer (Kabir et al. 2012). The coconut fibers without surface treatment were 2.50 mm long and 0.16 mm in diameter and had an aspect ratio of 15.91. An aspect ratio greater than 10 is interesting because the load transfer per unit volume of the fiber is improved by the greater surface area in contact with the polymer matrix (Levy Neto and Pardini 2016). In this way, the mechanical behavior of the composites depends on the aspect ratio of the fibers, in which the fiber dimension determines its use as reinforcement or filler (Khlif et al. 2022).

The length of the fibers plays an important role in the final strength of the composites because longer fibers promote greater fiber adhesion in the polymer matrix due to the greater friction or coiling of the fiber in the matrix. In addition, a longer fiber length leads to the formation of longer bridges between the cracks when the matrix fails and is less likely to be anchored in a part of the matrix that separates from the rest of the composite (Danso et al. 2015). Capela et al. (2017) observed that the stiffness of composites produced with an epoxy matrix increased by 25% when the length of the fibers increased from 2 to 4 mm but decreased when the length of the fibers was 6 mm. Thus, the lengths of the coconut fibers used in the present study are within the range verified by the authors, avoiding low fiber dispersion and disorder in the polymer matrix.

The surface treatment removes extractives, hemicelluloses and lignin from the surface of the fibers. This leads to a reduction in the diameter and an increase in the aspect ratio of the fibers, improving their mechanical properties. In this way, surface treatments are excellent alternatives aimed at

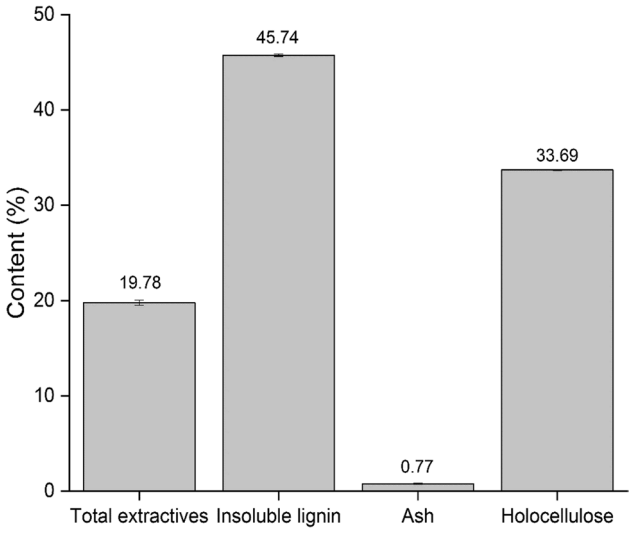

Fig. 4 Chemical constituents of the untreated coconut fibers

producing fibers with a higher aspect ratio, directly and positively influencing the response of composites to mechanical stress (Khlif et al. 2022).

In addition to the geometry of the fibers, the chemical constituents present in the plant fibers play an important role in the properties of the composites. This is because the behavior of a composite depends on the properties of the phases that constitute it as well as on the interfacial adhesion between them (Callister and Rethwisch 2019). Thus, high levels of some chemical constituents, such as extractives and lignin, may hinder the success of the reinforcement interaction with the matrix, inhibiting the adhesion mechanisms (physical, chemical and mechanical) related to the polymerization process of the polymer (Pizzi 1994). Figure 4 shows the mean values of the chemical constituents of the untreated coconut fibers.

As observed in Fig. 4, coconut fibers without surface treatment had a high total extractive content (19.78%). Some extractives present in lignocellulosic materials, such as fatty acids, terpenes, phenols, waxes and greases, can lead to increased plant fiber surface energy, promoting surface inactivation (Kumar and Pizzi 2019). In composite materials, where it is desired that polymer matrix stresses are distributed to the coconut fibers (reinforcement material), the presence of high levels of extractives can lead to reduced interfacial adhesion, resulting in a decrease in the mechanical properties of the composites. Cabral et al. (2016) observed a total extractive content of 17.75% in coconut fibers without surface treatment, while those treated with 5% NaOH alkaline solution had a content of 6.23%. Anuchi et al. (2022), in a study on the efficiency of the pretreatment of coconut fibers using protic ionic liquid, found total extractive contents of 3.40%. Given the natural origin of these fibers, the species, soil type, use and climatic conditions may be the cause of deviations in their chemical composition (Faria et al. 2020a).

For the insoluble lignin content (45.74%), similar values were observed by Anuchi et al. (2022) and Mohammed et al. (2015); these authors found lignin contents of 41.3% and between 40 and 45%, respectively. Lignin is a macromolecule constituted by an aromatic system composed of phenylpropane units, presenting hydrophobic behavior and is the reinforcing agent that unites the cells of the individual fibers (Patel and Parsania 2018). Thus, higher levels of insoluble lignin positively affect the dimensional stability of the composites, resulting in lower levels of water absorption. In addition, because it is a natural adhesive, higher levels of this chemical component are desirable to improve the fiber-matrix adhesion, consequently increasing the mechanical properties of the composites.

Holocellulose refers to the fraction of material composed of cellulose and hemicelluloses; these two chemical constituents are present in plant fibers with hydrophilic behavior. Cellulose is a linear biopolymer that constitutes about 40 to 50% of almost all plants, composed of microfibrils that have crystalline (highly ordered) and amorphous (less ordered) regions (Klock and Andrade 2013). Microfibrils build fibrils and these build cellulosic fibers. Thus, the fibrous structure of cellulose promotes high tensile strength and structural stability to the lignocellulosic fiber (Patel and Parsania 2018). In turn, hemicellulose is responsible for a considerable amount of moisture absorption, biodegradation and thermal degradation of the fiber due to its lower strength. Because it is the most hydrophilic component of the cell wall of plants, higher levels are undesirable. The results observed in the present study are slightly lower than those observed by several authors, who reported holocellulose values of 39.28% (Rojas-Valencia et al. 2018), 44% (Hasan et al. 2021) and 43.69% (Irawan et al. 2018).

Ash is composed of organic matter from the soil, which, despite the low content of this component found in the coconut fibers in the current study, can influence the quality of fiber-matrix adhesion and promotes the wear of the cutting tools during the processing of composites.

Thus, the final properties of the composites are related to the intrinsic properties of the matrix and the reinforcement as well as the adhesion between them (Verma et al. 2013). Thus, the chemical constituents directly affect the quality of the adhesive bond. The final properties of the composites are also influenced by the mechanical properties of the coconut fibers, where higher values of these properties provide effective stress transfer from the matrix to the reinforcement, intercepting the propagation of cracks and consequently avoiding catastrophic failures. Table 3 shows the mechanical properties of the coconut fibers obtained in the tensile test.

As shown in Table 3, no significant differences were observed for the mechanical properties of coconut fibers as a function of the surface treatments performed. Although the surface treatments reduced the diameter of the fibers

Table 3 Average tensile properties of coconut fibers

	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)
Without treatment	258.66	2.43	31.78
Hot water	239.16	2.51	31.72
Alkaline treatment (NaOH)	264.58	2.83	39.99
Alkaline treatment $Ca(OH)_2$	279.16	2.86	35.62
Corona discharge	252.89	2.21	25.57

due to the removal of the extractives and part of the lignin, the properties of the fibers were not affected. Surface treatments with hot water and alkaline solutions remove certain amounts of wax, lignin, oil and other impurities, increasing the surface roughness and consequently improving the wettability of the fiber surfaces, resulting in better physical and mechanical properties of the composites (Zhang and Hu 2014). Similar results were observed by Tran et al. (2013) for coconut fibers without surface treatment; the authors noted a tensile strength of 250 MPa, Young's modulus between 4 and 5 GPa and an elongation at break between 20 and 40%. Arsyad et al. (2019) observed a tensile strength of 186.4 MPa and an elongation at break of 28.3%, both of which were lower than those in the present study. This difference is attributed to the source of the coconut plant from which the fibers were extracted or the extraction method used (Adeniyi et al. 2019).

While the surface treatment with hot water did not cause changes in the mechanical properties of the coconut fibers, the treatments with alkaline solutions of NaOH and $Ca(OH)_2$ promoted an increase in the elongation values at break, which was also observed by Karthikeyan et al. (2014), who found a 23% increase in the elongation at break for fibers treated with NaOH compared to those without surface treatment. This increase is explained by the consequent reduction in fiber stiffness caused by the removal of part of the lignin present in the fibers treated with an alkaline solution. The surface treatment with corona discharge slightly reduced the mechanical properties of the coconut fibers, a behavior similar to that observed by Hassani et al. (2019), who reported a reduction in tensile strength from 800 to 700 MPa and in Young's modulus from 23 to 18 GPa for aloe vera fibers without surface treatment and after 5 min of treatment with corona discharge, respectively.

The electron micrographs obtained using SEM showed that the surface treatments performed on coconut fibers promoted an increase in the surface roughness, as shown in Fig. 5.

In coconut fibers without surface treatment, amorphous components (detritus) adhered to the surface, and they had a slightly rough topography. The fibers treated with hot water became rougher, but it was still possible to observe the presence of debris adhered to the surface of the fibers. This is because the extractives present in plant fibers may be materials that are volatile with water vapor, soluble in organic solvents or soluble in water (Klock and Andrade 2013). Thus, Fig. 5c shows that the surface treatment with NaOH solution resulted in a significant increase in the surface roughness of the coconut fibers, making it possible to identify parenchyma cells and tyloses on the surface of the fibers. When the $Ca(OH)_2$ solution was used, detritus (pectins, terpenes, waxes, greases, etc.) were not completely removed from the surface of the fibers, slightly improving the surface roughness. The alkaline treatments removed the extractives in addition to partially removing the hemicelluloses and lignin, promoting better packaging of the cellulose chains. A similar trend was observed with the fibers subjected to the treatment with corona discharge, in which despite removing the debris adhered to the surface of the fibers, promoted a slight increase in the surface roughness. The surface roughness will help form the mechanical bond between the fiber surface and the resin used so that the adhesion between the fibers and the resin will increase (Arsyad 2017; Renreng et al. 2017).

Unlike synthetic fibers, it is important to specify the growth and harvesting process of plants that produce natural fibers because their effect on mechanical properties is large. The service life of the composite can be reduced due to poor adhesion to the interface. This results in poor mechanical properties for the polymer-reinforced natural fiber composite due to its insufficient adhesion (Sanal and Verma 2019). Thus, after fiber production, chemical or physical treatments should be applied to increase the compatibility between the lignocellulosic fiber and the polymer matrix.

Characterization of composites

Density and void content

Density is a property that directly influences the thermal and mechanical properties of the resin and composites. Table 4 shows the theoretical and experimental densities and void content.

Table 4 shows that the theoretical density values for the resin and the composites produced were similar, which is mainly explained by the fact that it is a calculation that does not consider the specificities of the matrix or its interaction with the reinforcement; only the volumetric fractions and densities of the reinforcement and of the matrix are considered. Conversely, the experimental density shows the behavior of the composites compared to the pure resin, in which higher density values were observed for the composites produced. This behavior is explained by the higher density of the fibers and their insertion in the composites. Thus,

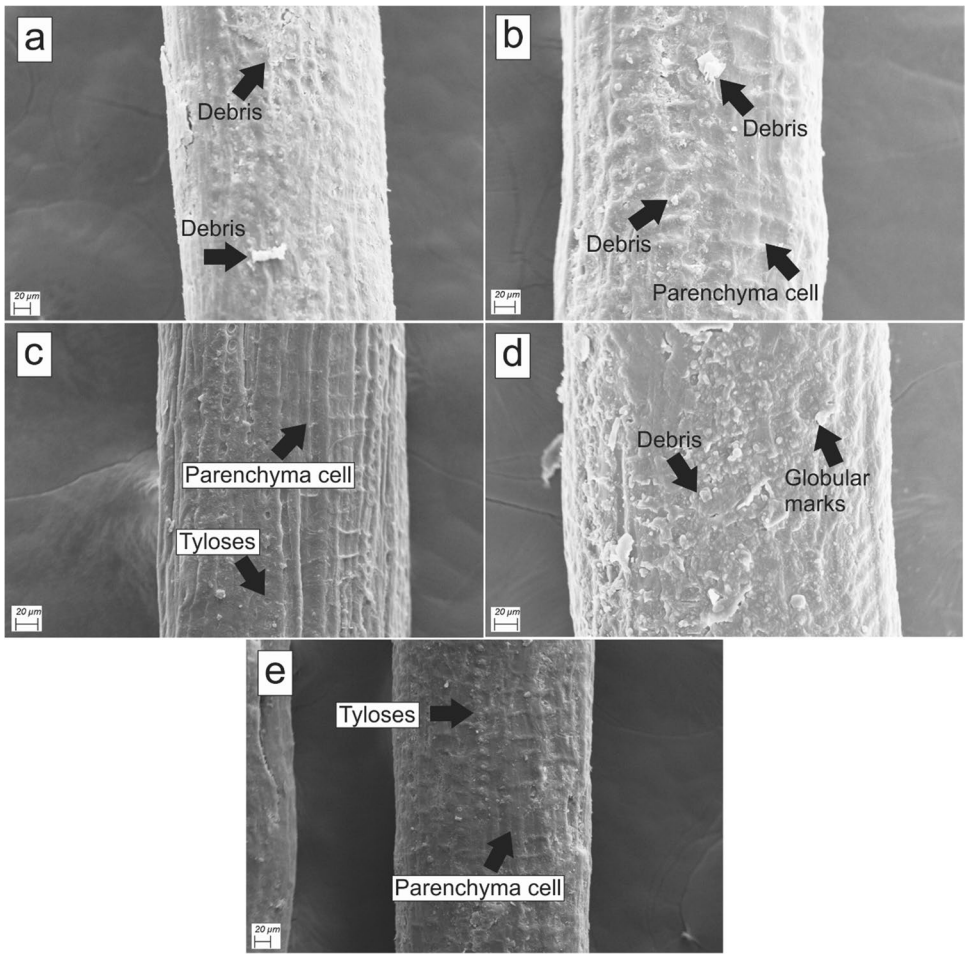

Fig. 5 SEM micrographs of coconut fibers. (**a**) Untreated fibers; (**b**) fibers treated with hot water; (**c**) fibers treated with NaOH; (**d**) fibers treated with $Ca(OH)_2$; (**e**) fibers treated with corona discharge

Table 4 Density and void contents of resin composites

Composition	Theoretical density (g/cm^3)	Experimental density (g/cm^3)	Void (%)
Resin	1.125	0.961	17.06
Without treatment	1.132	1.042	8.84
Hot water	1.129	1.097	2.91
Alkaline treatment (NaOH)	1.127	1.086	3.77
Alkaline treatment $Ca(OH)_2$	1.128	1.083	4.15
Corona discharge	1.131	1.095	3.28

the experimental density of the composites is the combined density of the reinforcement and the polymer matrix.

According to Table 4, the higher void content observed in the composites is due to the porous structure of the polymer. Because it is an exothermic polymerization reaction, about 24 kcal/mol of formed urethane is released, resulting in a porous structure (Vilar 1993). Due to the replacement of the polymer by coconut fibers, a portion of the prepolymer reacts with the hydroxyls on the surface of the coconut fibers. Thus, less polymer is formed and consequently less heat is released, which results in composites with lower porosity. Therefore, due to the replacement of part of the resin by coconut fibers, the void content decreases as a function of the higher density of the fibers in addition to their interaction with the polymer matrix. Zanini et al. (2021) observed an increase in density and reduction of the pore size of the composites produced with plant polyurethane reinforced with palm fibers (185 µm) compared to the resin (256 µm). This behavior was similar to that observed in the present study, in which the void content decreased from 17.06 to 8.84%, which was attributed to the interaction of the coconut fibers with the plant polyurethane matrix. Despite this reduction, the composites produced with coconut fibers treated with NaOH showed void contents of 3.77%, which were much lower than those composites produced with fibers without surface treatment. According to Ramlee et al. (2021), this difference is attributed to the incompatibility between the hydrophobic resin and the hydrophilic fiber during the production of the composites. In addition, trapped air bubbles from the manual manufacturing process of hand lay-up of

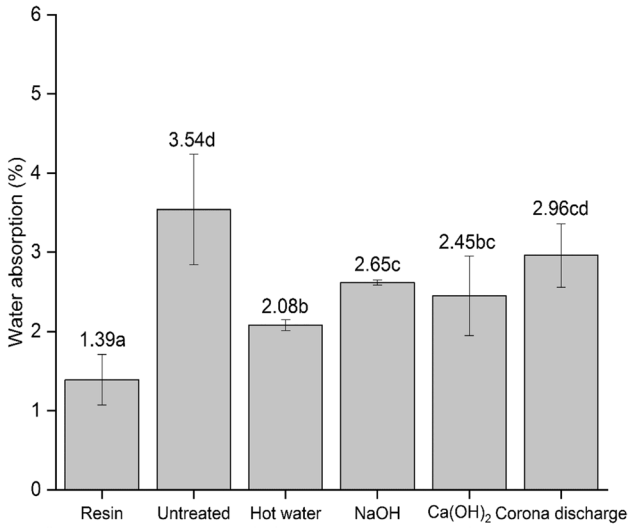

Fig. 6 Water absorption of the resin and composites. Averages followed by the same letter (**a**, **b**, **c**, and **d**) in the column do not differ statistically according to Tukey's test ($p > 0.05$)

the composites influence the void contents, affecting the physical and mechanical properties of the composites (Chua et al. 1992).

Water absorption

Figure 6 shows the water absorption levels of the resin and the composites produced.

Due to the hydrophobic nature of the polymer matrix, the resin had the lowest mean value (1.39%), differing statistically from the composites produced. In contrast, the composites produced with fibers without surface treatment had the highest water absorption levels. This behavior is explained by the low wettability of the fibers by the polymer matrix, resulting in weak interfacial adhesion, thus exposing the fibers to contact with water. In addition, coconut fibers not subjected to surface treatment have higher levels of hemicelluloses, and this chemical component is the most hydrophilic of the cell walls of plants. Thus, the higher water absorption levels observed for composites reinforced with coconut fibers without surface treatment are explained by the chemical properties of the fibers used (Fig. 4), in which the cellulose and hemicelluloses present in the cell wall of the fibers consist of hydroxyl (OH) groups that react when they come into contact with water molecules (Pérez-Fonseca et al. 2015). In addition to the chemical components present in holocellulose (cellulose and hemicelluloses), the high total extractives content (19.78%) and insoluble lignin content (45.74%), which have a hydrophobic character, acted by hindering the synergy between the fibers and the matrix, promoting inhibition of the surface area of the fibers (Faria et al. 2020a). The lowest water absorption contents of the composites produced were observed for the surface-treated fibers, where the surface treatment reduced the number of free hydroxyls, improving interfacial adhesion of the fiber with the matrix, which reduced the voids present in the composites and, consequently, reduced water absorption (Sudhakara et al. 2013).

Table 5 Contact angle and degrees per second values for the resin and composites for each measured time

Composition	Measurement time (s)	Contact angle (°)	° per second
Resin	5	99.69	0.032
	55	97.94	
Without treatment	5	78.03	0.063
	55	74.56	
Hot water	5	87.09	0.089
	55	82.18	
NaOH	5	78.64	0.079
	55	74.31	
$Ca(OH)_2$	5	94.12	0.057
	55	90.97	
Corona discharge	5	93.43	0.051
	55	90.64	

Wettability

The higher water absorption levels observed for the composites in relation to the resin can be explained by the lower contact angle values (Table 5), in which the composites showed a reduction in the hydrophobic behavior observed for the resin after 5 and 55 s measurements.

The illustrative images of the contact angles of the resin and the composites after 5 and 55 s after contact with the droplet on the wood surface are shown in Fig. 7.

Table 5 shows that the resin has contact angles of 99.69° and 97.94° after 5 and 55 s, respectively, which are consistent with those observed in the literature (Zanini et al. 2021). As verified in the literature and in this study, the contact angle initially decreased rapidly (propagation phase); however, over time, the contact angle decreased slowly and finally reached equilibrium (diffusion phase) (Laskowska and Kozakiewicz 2017).

Only the composites produced with untreated fibers and fibers treated with hot water and NaOH showed hydrophilic behavior (contact angle < 90°), while the others followed the hydrophobic behavior of the matrix (contact angle > 90°). This trend can be explained by the efficiency of the surface treatment. Due to the severity of the surface treatment, higher extractive contents were removed from the surface of the coconut fibers, and thus the hydrophilic components (cellulose and hemicelluloses) of the cell wall of the fibers

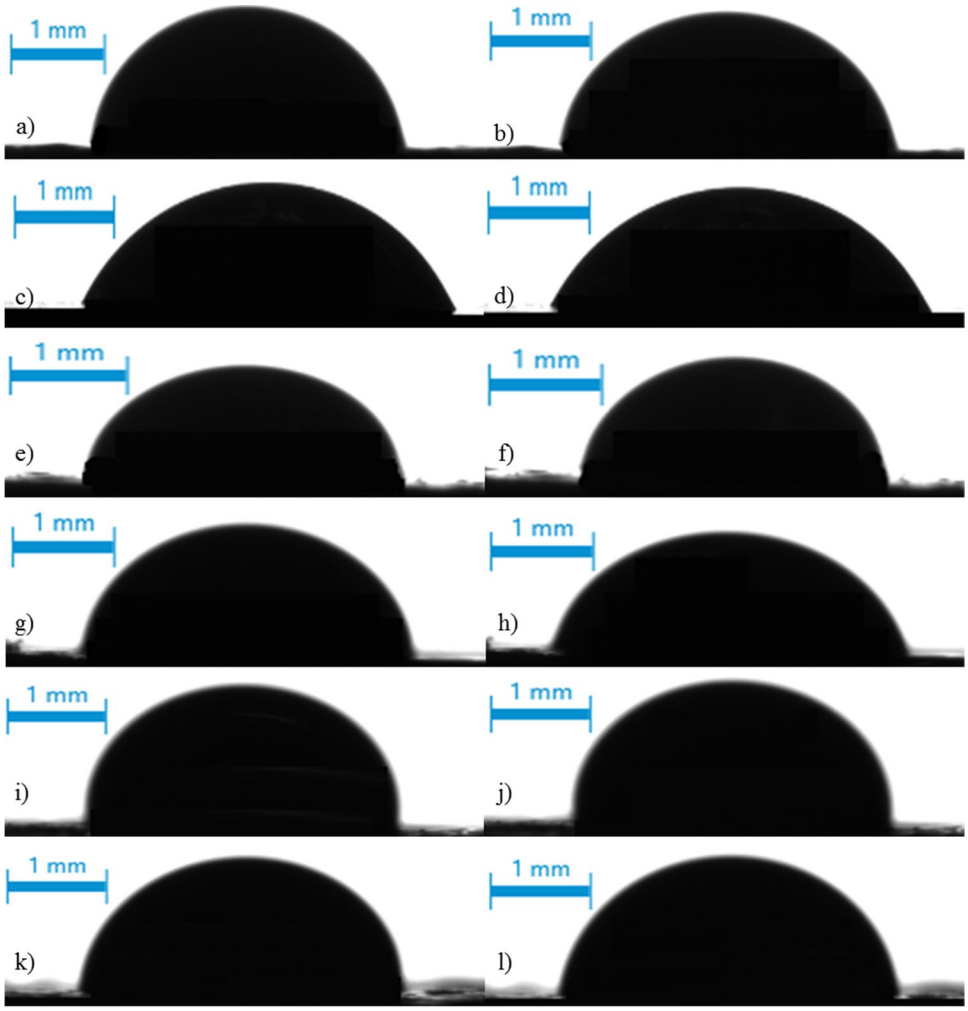

Fig. 7 Contact angles. (**a**) Resin after 5 s; (**b**) resin after 55 s; (**c**) composite with untreated fibers after 5 s; (**d**) composite with untreated fibers after 55 s; (**e**) composite with fibers treated with hot water after 5 s; (**f**) composite with fibers treated with hot water after 55 s; (g) composite with fibers treated with NaOH after 5 s; (**h**) composite resin with fibers treated with NaOH after 55 s; (**i**) composite with fibers treated with $Ca(OH)_2$ after 5 s; (**j**) composite with fibers treated with $Ca(OH)_2$ after 55 s; (**k**) composite with fibers treated with corona discharge after 5 s; (**l**) composite with fibers treated with corona discharge after 55 s

were exposed, resulting in the interaction of the hydroxylic sites with the deposited water droplet. Therefore, the more efficient the surface treatment is, the better the connections between the matrix and the reinforcement, resulting in the composite exhibiting the behavior of the matrix phase, i.e., hydrophobic behavior (Zanini et al. 2021). In addition, the manual processing method of the composites used in the present study may have resulted in poor dispersion of the coconut fibers in the matrix, resulting in points with fiber agglomeration and, consequently, a large volume of free hydroxylic sites to interact with water.

Tensile strength

The typical curve of tensile strength vs. specific deformation (Fig. 8) shows the mechanical behavior of the resin and the composites produced.

The composites produced with coconut fibers treated with the alkaline NaOH solution showed the highest mean values of maximum tensile strength, significantly higher than the resin and the composites produced with other treatment methods. The insertion of the coconut fibers in the composites promoted an increase in their specific deformation in relation to the resin, except for the composites produced with fibers treated with the NaOH solution, which showed specific deformation results close to the resin. This is due to the good interaction between the fibers treated with the NaOH solution and the polymer matrix, in which there was an effective transfer of stress from the matrix to the fibers, resulting in the increased rigidity of the composites.

Based on the experimental procedures applied to the six specimens under the tensile test, the mean values and standard deviations for the tensile strength, Young's modulus and elongation at break were calculated, and the results are shown in Table 6.

The composites produced with coconut fibers treated with the alkaline NaOH solution had the highest mean values of tensile strength and Young's modulus, much higher than those of the pure resin. Increases of 157% for tensile strength and 102% for Young's modulus and a reduction in elongation at break of 7% were observed. The same trend was observed for composites produced with coconut fibers

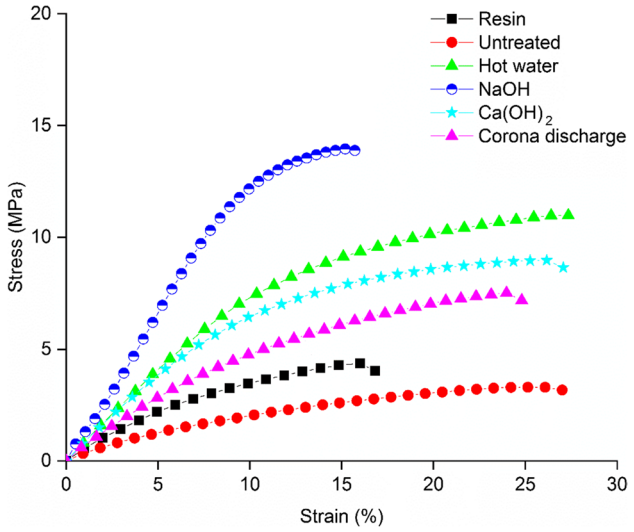

Fig. 8 Tensile strength vs. specific deformation of the resin and composites

subjected to alkaline treatment with $Ca(OH)_2$, corona discharge and hot water. Conversely, the resin and composites produced with coconut fibers without surface treatment showed the lowest tensile strength values and are therefore mainly related to the weak interfacial adhesion between the fiber and matrix. Furthermore, due to the incompatibility of the hydrophilic nature of the fibers with the hydrophobic polymer matrix, there is a tendency to form aggregates, originating in points without the presence of fibers (Ku et al. 2011). The polymerization mechanism of vegetable polyurethane with coconut fibers for the formation of composites occurs by the interaction between the urethane functional groups with the hydroxyls of coconut fibers.

Figure 9 shows electron micrographs obtained from the fracture surface of the composites produced. Figure 10a shows the detachment of the coconut fiber without surface treatment of the polymer matrix, i.e., during the polymerization of the plant polyurethane, the extractives present on the surface of the coconut fibers acted by reducing the surface wettability of the fibers, inhibiting the adhesion mechanisms between the matrix and reinforcement.

As shown in Table 6, the improvement of interfacial adhesion for composites produced with treated fibers is justified by the removal of extractives and hemicelluloses from the surface of coconut fibers, with consequent increases in crystallinity and relative cellulose content (Liu et al. 2019), resulting in the reduction of the gap between the fiber and the plant polyurethane matrix observed in Fig. 9. In addition, it was observed that there was an efficient transfer of stresses since the fiber broke without detaching from the polymer matrix (Fig. 9c). According to Sanal and Verma (2019), this behavior is due to the insertion of chemical groups that act by modifying the physical and chemical interactions between the lignocellulosic fiber and the polymeric matrix.

Widnyana et al. (2020) found an increase in tensile strength from 17.6 to 22.3 MPa in composites produced with an unsaturated polyester matrix reinforced with coconut fibers treated with an alkaline NaOH solution and without surface treatment, respectively, resulting in an increase of 27%. An 18% increase in tensile strength was observed in composites produced with coconut fibers treated with an NaOH solution compared to untreated fibers using an epoxy matrix (Yan et al. 2016). The authors also noted the same behavior observed in the present study, in which there was an increase in Young's modulus and a reduction in elongation at break for the composites produced with treated fibers.

Despite the slightly lower tensile strength for the composites produced with fibers treated with hot water compared to the composites produced with the NaOH solution, it was noted that the treatment with hot water was more effective than the treatments with $Ca(OH)_2$ and with corona discharge, promoting an improvement of 108% in tensile strength, 28% in Young's modulus and 39% in elongation at break. In addition, it is a relatively simple surface treatment, without the use of chemical reagents that acts in the removal of water-soluble impurities, such as polysaccharides, fats, resins and phenols. Because it is less aggressive to plant fibers, there is almost no removal of hemicelluloses and lignin and is therefore a superficial treatment.

Thermogravimetric analysis

Figure 10 shows the results of the thermogravimetric analysis for the resin and the composites produced.

The thermal degradation of the plant polyurethane begins at 224 °C and ends at 410 °C, with a mass loss of 60%. This

Table 6 Average tensile properties of the resin and composites

Composition	Tensile strength (MPa)	Young's modulus (MPa)	Elongation at break (%)
Resin	5.68 (0.43)	61.61 (8.29)	26.97 (2.71)
Without treatment	4.13 (0.55)	16.97 (6.24)	35.60 (4.17)
Hot water	11.83 (0.42)	79.06 (6.68)	37.38 (1.65)
NaOH	14.61 (0.77)	124.33 (6.14)	25.01 (3.26)
$Ca(OH)_2$	10.10 (0.66)	69.69 (9.20)	33.05 (0.71)
Corona discharge	9.18 (0.48)	45.35 (10.01)	41.66 (6.37)

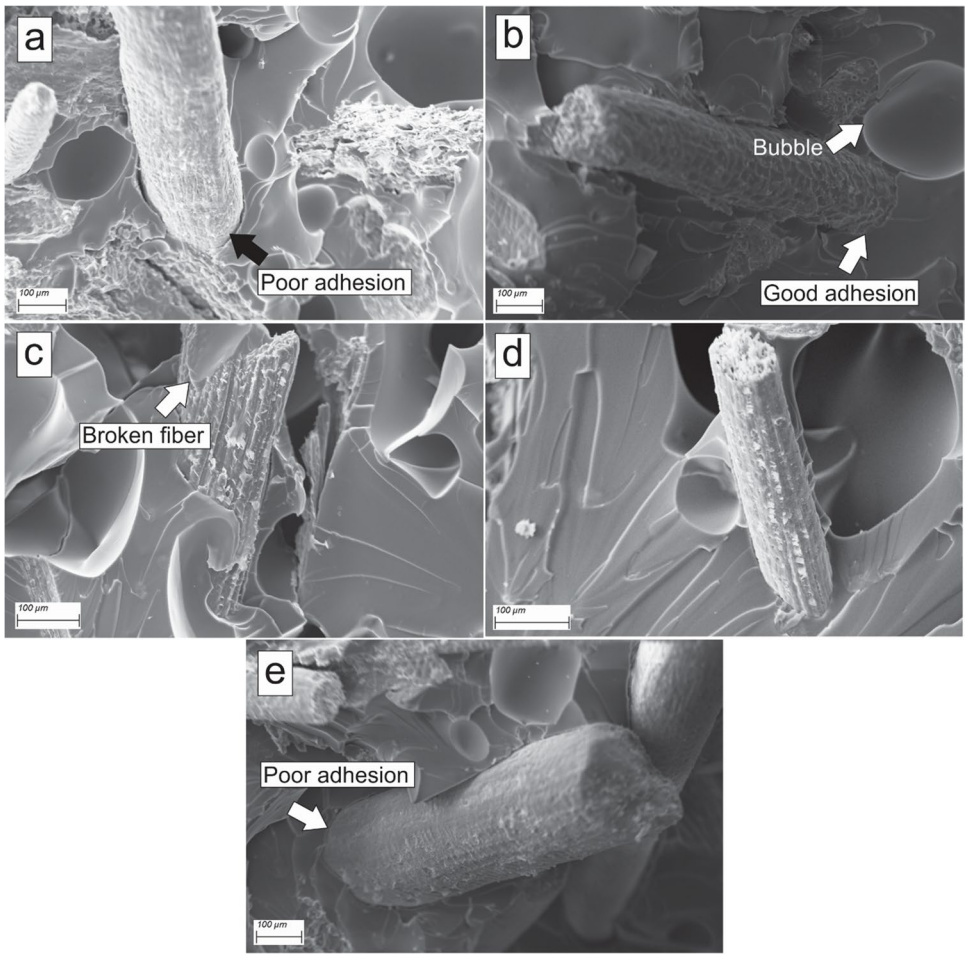

Fig. 9 SEM micrographs of coconut fibers. (**a**) untreated fiber; (**b**) treated with hot water; (**c**) treated with NaOH; (**d**) treated with $Ca(OH)_2$; (**e**) treated with corona discharge

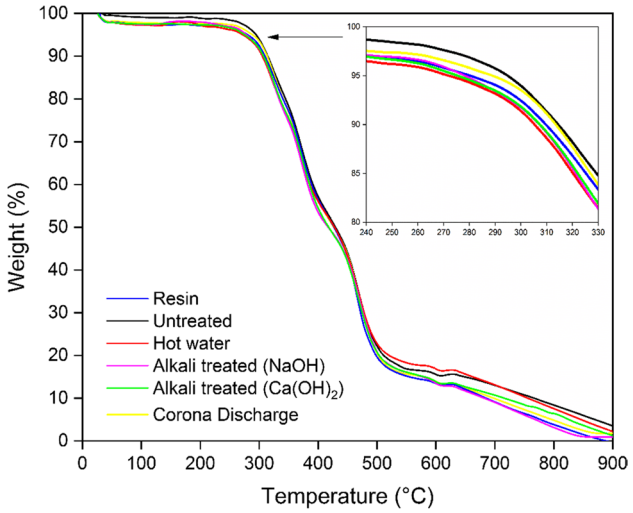

Fig. 10 Thermogravimetric analysis of the resin and composites

decomposition is related to the breakdown of the urethane bond and loss of the diphenyl methylene diisocyanate (MDI) present in the prepolymer (Claro Neto 1997; Cangemi et al. 2006; Pereira 2010). The 38% mass loss from 410 to 588 °C occurs due to the thermal decomposition of the polyol and is related to the breakdown of the fatty esters and double bonds present (Carvalho et al. 2014).

The thermal stability of the composites depends on the combined thermal behavior of the resin and fibers, as shown in Fig. 10. As the temperature increases, the mass loss of the composites up to approximately 150 °C is attributed to the evaporation of water from the fibers; and from 150 to 200 °C, some extractives with low resistance to thermal degradation are removed; between 200 and 260 °C, there is degradation of the hemicelluloses; between 240 and 350 °C of cellulose; between 280 and 500 °C of lignin, and above 500 °C, there is carbonization of the fibers (Salazar et al. 2011; Morandim-Giannetti et al. 2012; Tahir et al. 2019). The composites produced with treated fibers had a lower thermal degradation temperature, which was caused by the removal of cell wall components, especially total extractives. The decrease in temperature at the beginning of thermal degradation is mainly related to the lower total extractive content, in which fibers without alkaline treatment showed higher onset temperature. This is due to the fact that some

Table 7 Temperature and mass/weight loss (%) of the resin and composites

Composition	Onset temperature (°C)	End set temperature (°C)	Decomposition temperature (°C)	Residue value at 900 °C (%)
Resin	287.84	486.29	462.63	- -
Without treatment	302.58	496.63	463.14	3.45
Hot water	301.65	488.85	468.44	2.21
NaOH	299.86	491.18	470.79	0.92
$Ca(OH)_2$	297.73	493.49	465.79	1.20
Corona discharge	297.38	507.99	477.83	1.77

extractives are not very resistant to thermal degradation compared to the macromolecular constituents present in plant materials (Protásio et al. 2021). The main thermal events observed in the thermogravimetry for the resin and the composites are shown in Table 7.

The composites showed a thermal degradation onset temperature (T_{onset}) close to the resin, varying between 287.84 (resin) and 314.38 °C (corona discharge). Close values were also observed for the end set temperature and decomposition temperature. However, regarding the residue contents, the resin was already totally degraded at 900 °C, leaving only contents varying between 0.92% (composites produced from fibers treated with the NaOH solution) and 3.45% (composites produced from fibers without surface treatment), and these contents are attributed to the mineral constituents (ash) present in the coconut fibers.

Thus, when the thermal degradation onset temperature (T_{onset}) is reached, the degradation of the composites is irreversible; therefore, it is the maximum temperature for the processing of the composite.

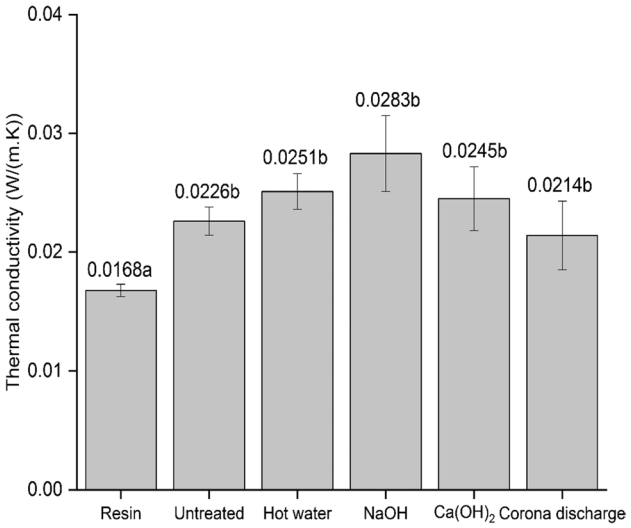

Fig. 11 Thermal conductivity of the resin and composites. Averages followed by the same letter (**a** and **b**) in the column do not differ statistically according to Tukey's test ($p > 0.05$)

Thermal conductivity

Among the materials used for thermal insulation of buildings, rigid polyurethane foams stand out due to their efficiency in ensuring greater thermal comfort to users (Kaikade and Sabnis 2022). Figure 11 shows the mean thermal conductivity values obtained for the resin and the composites produced, and it was observed that the insertion of coconut fibers in the composites promoted an increase in thermal conductivity.

The increase in thermal conductivity, i.e., the reduction of thermal insulation for the composites, is attributed to the decrease in the number of voids, as shown in Table 4. Due to the higher void content, the pure resin showed higher porosity and thus better thermal performance caused by the presence of air inside the pores. Several authors evaluated the thermal conductivity of polyurethanes produced with polyol from renewable sources. For example, Malchiodi et al. (2022) observed a thermal conductivity of 0.044 W/(mK), Tan et al. (2011) noted values ranging from 0.023 to 0.024 W/(mK), while Zhou et al. (2013) obtained a thermal conductivity between 0.027 and 0.028 W/(mK) for polyurethanes produced with soybean-based polyol.

The thermal conductivity of the composites is also influenced by the higher thermal conductivity of coconut fibers compared to pure resin. Thermal conductivity values ranging from 0.024 to 0.052 W/(mK) for fibers with densities between 0.03 and 0.12 g/cm^3 were observed by Bui et al. (2020). Thus, despite the low content of coconut fibers inserted in the composites, the coconut fibers, which have a low density, occupy a large volume, filling part of the voids. Thus, the higher thermal conductivity of the coconut fibers promotes the reduction of the thermal insulation of the composites. Sair et al. (2018) concluded that the insertion of hemp fibers in a polyurethane matrix results in an increase in thermal conductivity. The authors noted an increase from 0.030 (polyurethane) to 0.034 W/(mK) in composites produced with 10% hemp fibers.

Despite the increase in thermal conductivity, the composites can be classified as highly insulating materials because they have low thermal conductivity (0.01–0.05 W/(mK)) (Cardoso et al. 2011). In addition, the thermal properties of

composites reinforced with natural fibers are lower than the heat transfer coefficients of synthetic fibers, such as glass fibers (0.043 W/(mK)). The composites produced in the present study with plant polyurethane reinforced with coconut fibers showed thermal conductivity values close to other materials and insulation systems, such as paper-coated fiberglass mats (0.035–0.046 W/(mK)), extruded expanded polystyrene plates (0.027 W/(mK)), mineral fiber plates (0.049 W/(mK)) and mineral wool granules with sprinkled asbestos cements (0.046 W/(mK)) (Bergman et al. 2018).

Conclusions

The high mechanical strength of the coconut fibers, combined with the greater surface roughness promoted by the surface treatments, resulted in a decrease in voids and an increase in the mechanical properties of the composites, with an increase of 157% in tensile strength and 102% in Young's modulus for the composites produced with fibers treated with NaOH in relation to the resin. Although the insertion of coconut fibers promotes a slight increase in the thermal conductivity of the composites compared to the resin, the composites showed values similar to other materials used as thermal insulators in buildings. In addition, the use of coconut fibers as a substitution of the plant polyurethane matrix promotes the reduction of the consumption of the polymer, with a consequent reduction in the final cost of the composite. The ecological advantages are also noteworthy, since it is a biodegradable matrix reinforced with plant fibers, which in itself brings a much more ecological solution compared to composites made with matrices or reinforcements from nonrenewable sources applied as thermal insulators.

Acknowledgements The authors thank the PostgraduateProgram in Biomaterials Engineering (PPGBIOMAT) of the FederalUniversity of Lavras (UFLA) for providing material and infrastructure.The authors are also grateful to the National Council for Scientifcand Technological Development (CNPq) and Coordination for the Improvement of Higher Education Personnel (CAPES).We also thank the Fundação de Amparo à Pesquisa do Estado de Minas Gerais (FAPEMIG) for the postdoctoral scholarships (Process APQ-04368-22).

Author contribution Douglas Lamounier Faria: conceptualization, methodology, investigation, writing the original draft; Lourival Marin Mendes and José Benedito Guimarães Junior: supervision, funding acquisition, and project administration. All authors read and approved the final manuscript.

Data availability The datasets supporting the conclusions are included in the manuscript. Furthermore, the datasets analyzed in this study are available from the corresponding author upon request.

Declarations

Ethics approval Not applicable. This manuscript does not involve researching humans or animals.

Consent to participate All of the authors consented to participate in the drafting of this manuscript.

Consent for publication All of the authors consented to publish this manuscript.

Conflict of interest The authors declare no competing interests.

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