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Suitability of Kahili ginger stem fibres for reinforcement/filler of polymeric matrix composites

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ABSTRACT

Kahili ginger (KG) is one of the world's most invasive species, whose proliferation can be controlled by mechanical thinning. The KG chips produced in that process can be used as reinforcement/filler in polymer matrix composites (PMC), adding value to this waste. The aim of this study is to evaluate the possibility of using fibres obtained from KG stem chips for PMC. The chemical composition, thermal stability, water absorption, hydrophilicity and tensile mechanical properties of the KG fibres were assessed, and the values obtained were compared with those reported in the literature for other natural fibres commonly used in the manufacture of PMC. The results showed that the fibres treated with alkali/silane are thermally stable up to 200 °C, have a contact angle of 70°, a water absorption of 24 %, a modulus of elasticity of 23±3 GPa and a tensile strength of 277±113 MPa. Overall, the results demonstrate the potential of KG as a reinforcement/filler material for PMC.

1. Introduction

Kahili ginger (KG) is an ornamental plant scientifically named as *Hedychium gardnerianum* and is native from the Hawaiian rainforest (India, Nepal). This plant is recognised as one of the one-hundred worst invasive species in the world, spreading in regions such as the Azores, Madeira, Jamaica, New Zealand and Hawaii, South and Central America, Australia and southern Africa. KG spreads through seeds dispersed by birds and through rhizomes. Its rapid growth, adaptability to various environments, and rhizome system allow it to dominate ecosystems, altering the structure of native vegetation and threatening biodiversity. Thus, its rapid growth, adaptability, and potential for cultivation on marginal lands make KG a promising candidate for industrial crops, and it is therefore important to find sustainable economic applications for this plant. According, to ancient cultures, the rhizomes have anti-inflammatory and analgesic properties, turning KG interesting for pharmaceutical applications [1,2]. In addition, the sweet aroma of its flowers may be captured through the extraction of essential oils and fragrances that can be used in aromatherapy and perfumery [3].

The proliferation of this plant is usually controlled through the application of herbicides or mechanical thinning. The latter process generates a residue that might be used as a filler or a reinforcing material for polymer matrix composites (PMC), giving this residue added value. It is known that fibres extracted from plants are rich in cellulose and can be used as an alternative to glass fibre reinforcements [4,5]. In fact, it was reported by Eleutério et al. [6] that the fibres mechanically extracted from the stem of KG have 79 % cellulose, 5.7 % hemicellulose and 12 % lignin, with an ash content of 2.0 %, a crystalline index of 70 % and are

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thermally stable at 200 °C. The amount of cellulose and the high crystalline index reported in this work indicate that these fibres could have greater resistance [7].

The introduction of natural fibres PMC results in lighter materials, with excellent sound absorption properties, exhibiting in some cases higher specific modulus than when are reinforced with glass fibre, easier to manufacture and with a lower environmental impact (manufacture and disposal). Sisal, Flax, jute, kenaf, and bamboo are examples of natural fibres widely used in the composite's manufacture [8,9]. For example, in car industry, natural fibres are used in several parts such as door panels, dash boards, seat backs and trims, due to be light and comfortable, have a pleasing aesthetic, and thermal and acoustic insulation capacity [10,11]. However, there are many issues that need to be addressed when considering its use, such as mechanical and thermal resistance, water absorption and fibre/matrix adhesion [12]. Strategies employed to overcome these challenges include, in most cases, an alkaline treatment, or a silane treatment applied after an alkaline treatment [13,14]. Alkaline treatment, also known as mercerisation, is a process in which the fibres are immersed in a 4–10 % solution of sodium hydroxide (NaOH) for a certain period, at a specific temperature. This treatment allows removing some of the lignin, hemicellulose and other impurities from the fibre surface, therefore exposing more cellulose and increasing the number of hydroxyl groups available for bonding. In addition, the treatment increases the roughness of the fibre's surface, improving its mechanical bond with the polymeric matrix in the composites [15]. Posterior treatment with a silane coupling agent is used to enhance the interfacial chemical bond between the fibres and the polymeric matrix, forming a bridge between the hydrophilic fibre and the hydrophobic polymer matrix [16,17]. In this treatment silanol groups, formed from alkoxy groups via hydrolysis, react with hydroxyl groups present in the cellulose, living an organofunctional group that can increase the adhesion between the fibre and the matrix. Among the silane couple agents, aminopropyltriethoxysilane is one of the most effective silane compounds. This has two distinct functional groups: one that forms a strong covalent bond (Si-O-C) with the hydroxyl (-OH) groups of the fibre and another (amino group) that can form covalent and hydrogen bonds with a variety of polymeric matrices, including thermosets (epoxies and polyurethanes) and thermoplastics (polypropylene and PLA) [17].

The aim of this work was to evaluate if KG stem fibres, obtained from mechanical thinning chips, have potential to be used as reinforcement/filler for polymeric matrix composites. For that, KG was submitted to alkaline treatment and posterior silane treatment. Than the morphology, chemical composition, crystallinity index, thermal stability, water absorption, hydrophilicity and tensile properties were accessed. To evaluate KG suitability, the obtained values were compared to values reported in literature for natural fibres currently used as reinforcement/filler for polymeric matrix composites.

2. Materials and methods

2.1. Materials

KG stem chips were obtained by mechanical thinning (Fig. 1a) from island of San Miguel, Azores Archipelago, Portugal. For alkali treatment, sodium hydroxide (MERCK, 99 %) was used. Regarding to silane treatment, aminopropyltriethoxysilane (Sigma Aldrich, 99 %) and ethanol (Fisher chemical, 99,8 %) were used. The preparation of all solutions, as well as the fibres washing was done with distilled water. Citric acid (LABKEM, 99.5 %) was used for pH adjustments. Diiodomethane (Sigma Aldrich, 99 %) and distilled water were used in the wettability measurements.

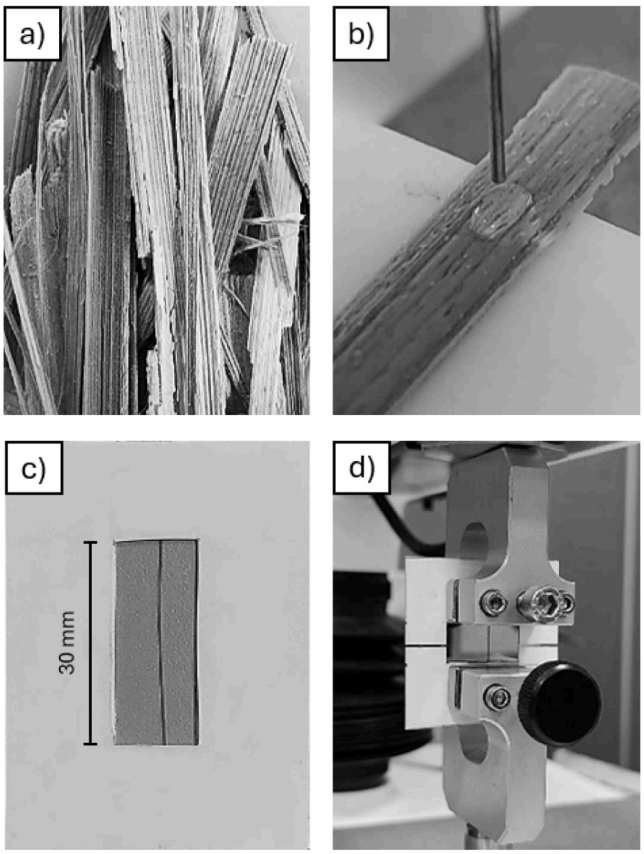

Fig. 1. a) Residue obtained by mechanical thinning of KG stem; b) Deposition of a water drop on a slab of KG for contact angle measurement; c) Card frame with a glued fibre for tensile tests, d) The frame card gripped to the texturometer before testing.

2.2. Methods

2.2.1. Kahili ginger treatments

Kahili ginger (KG) stems obtained from mechanical thinning were first reduced into chips with approximate dimensions of 30–50 mm in length, 10–20 mm in width, and 5–10 mm in thickness. These chips were manually cut into smaller slabs and subsequently manually defibrillated into individual fibres. Prior to chemical treatments, all chips and fibres were thoroughly washed with distilled water until the effluent ran clear, to remove dirt and soluble compounds, and then oven-dried ((Binder® ED115, Binder, Germany) at 70 °C for 24 h.

2.2.1.1. Alkaline treatment. Slabs and fibres were immersed in a 5 wt. % sodium hydroxide solution for 6 h at room temperature. Then they were immersed in distilled water several times until the pH was neutral, and dried in an oven (Binder® ED115, Binder, Germany) at 70 °C for 24 h.

2.2.1.2. Silane treatment. Part of the slabs and fibres of KG previously alkali treated were immersed during 3 h in a solution prepared with 69 % ethanol, 29 % of distilled water and 2 % of *aminopropyltriethoxysilane* with a pH between 4 and 3.5 adjusted with 1 M citric acid solution. After, KG was washed in a solution consisting of 80 % ethanol and 20 % distilled water. Finaly, KG was soaked in ethanol for 24 h and there after dried in an oven (Binder® ED115, Binder, Germany) at 70 °C for 24 h.

2.2.2. Morphology

The fibres morphology before and after treatment, and the tensile fracture surface were analysed using an electron scanning microscope (Hitachi® S-2400 (Japan)) For that the samples were previously coated

with an Au-Pd alloy.

2.2.3. Fourier transform infrared spectroscopy (FTIR)

FTIR analyses of untreated and treated fibres were performed using a FTIR spectrometer (Perkin Elmer®, Waltham, MA, USA) equipped with a diamond crystal ATR accessory (model UATR Two) at room temperature for a Wavenumbers ranging from 4000 to 400 cm^{-1}. The spectra were obtained from eight scans with a resolution of 4 cm^{-1} and normalized using the OriginPro 8.5 software. Measurements were done in triplicate.

2.2.4. X-Ray diffraction

X-ray diffraction (XRD) analysis was performed to obtain the crystallinity index (CI) of KG before and after treatments. XRD scans were performed using Bragg–Brentano configuration with Cu-Kα radiation in 2θ range from 10° to 80°. A step size of 0.03° and an exposure time of 0.5 s per step were used. A beam knife was used to reduce background scattering at low 2θ angles. The crystallinity index (CI) was estimated using the Segal method as expressed in Eq. (1) [18].

$$CI = \left(1 - \frac{I_a}{I_c}\right) * 100 \quad (1)$$

Where I_a is the Intensity of the amorphous phase (calculated by the hight of the value 2θ∼18°) and I_c the Intensity of the peak at the crystalline phase (maximum intensity in the 2θ angle range of 20–23°)

2.2.5. Thermogravimetric analyses (TGA)

The thermal stability of the treated and untreated fibres was analysed using a Hitachi® STA7200 Thermal Analysis System equipment under an air atmosphere (200 mL/min) and a heating rate of 10° C/min (until 600 °C). There analyses were performed per condition ($n > 3$).

2.2.6. Water absorption

For the water absorption, KG Slabs with of 5 × 5 × 0.01 mm, previously dried for 24 h in an oven (Binder® ED115, Binder, Germany) at 70 °C, were weighed (W_0) and immersed in distilled water for 24 h, at room temperature (≈23 °C). After that, the samples were weighed (W_1) again. The water absorption percentage (WA) was calculated according to Eq. (2). At least 10 experiments (n > 10) were performed per condition.

$$WA(\%) = \frac{W_1 - W_0}{W_0} \times 100 \quad (2)$$

2.2.7. Wettability and surface free energy

Water and diiodomethane contact angles with the surface of untreated and treated KG slabs (θ_{water} and θ_{diim}, respectively) were measured through the sessile drop method (Fig. 1b) at room temperature and humidity (≈23 °C and 50 % HR). Before measurement, the materials were dried as referred in the previous section. Drops of 4–6 μL were generated with a micrometric syringe and deposited on the KG surface, at room temperature. The images were obtained using a video camera coupled to a microscope (Wild M3Z) and the angles were determined with the software ADSA-P (Axisymmetric Drop Shape Analysis – Profile). The reported results correspond to the mean of at least 15 experiments (n > 15).

The materials' surface free energy (γ_{sV}), was calculated through the Owens and Wendt method [19], applying the geometric mean approach. This implied solving the following system of equations for water and diiodomethane:

$$\gamma_{LV\,water}(1 + cos\theta_{water}) = 2\left(\sqrt{\gamma^d_{LV\,water}\gamma^d_{sV}} - \sqrt{\gamma^p_{LV\,water}\gamma^p_{sV}}\right) \quad (3)$$

$$\gamma_{LV\,diim}(1 + cos\theta_{diim}) = 2\left(\sqrt{\gamma^d_{LV\,diim}\gamma^d_{sV}} - \sqrt{\gamma^p_{LV\,diim}\gamma^p_{sV}}\right) \quad (4)$$

where $\gamma_{LV\,water}$ and $\gamma_{LV\,diim}$ refer to the surface tension of the liquids. According to the Owens and Wendt method, both the surface free energy and the surface tension of the liquids can be given by the sum of a dispersive and a polar component (superscripts d and p, respectively). The values of the liquid's surface tension and respective dispersive and polar components were retrieved from [20]. The material's polarity, which corresponds to the ratio between the polar component of the surface free energy and the total value of this parameter, was also calculated.

2.2.8. Tensile testing

The tensile tests were performed using a texturometer, TA. XT Express Texture Analyzer (Stable Micro Systems, Godalming, Surrey, UK) with a load cell of 50 N. The software Exponent Lite Express (version 6) was used to acquire the data. Briefly, the treated and untreated KG fibres were glued into a card frame (Fig. 1c). The cards containing the fibres were griped to the texturometer and before the tests the frames were cut as demonstrated in Fig. 1d). The tensile tests were carried out at a rate of 0.4 mm/min with a gage length of 10 mm. The transverse area of the fibres was determined from scanning electron microscopy (SEM) images, using the ImageJ software. At least 8 tests per group were performed(n > 8).

The set-up compliance (C) was calculated using the method described in [21,22]. For that, tensile tests were performed using untreated fibres with gauge length (l) of 10, 20 and 30 mm and the force versus displacement curves were obtained. The calculation was done using the following Equation:

$$K = \left[\frac{1}{EA}\right] l + C \quad (5)$$

where K (mm/N) is the slope of the curve displacement/force obtained for each test, E is the Young's modulus [GPa] of the fibre and A is the transversal area of the fibre [mm^2].

2.2.8. Statistical analysis

Quantitative results are given as mean ± standard deviation. The data distribution normality was assessed using the Shapiro–Wilk test. When they followed a normal distribution, Levene's test was applied to evaluate the homogeneity of variance. When variances were found to be similar between groups, one-way ANOVA was performed followed by Tukey's test for multiple comparisons. In cases where the assumption of equal variances was not verified, Welch's ANOVA and Dunnett's C test were employed to identify different pairs of groups. Non-parametric tests were performed for data not following a normal distribution. In this case, Kruskal–Wallis tests adjusted with Bonferroni correction were used to determine significant differences between groups. All tests were performed using the Statistics Kingdom software.

3. Results

3.1. Morphology

Fig. 2 illustrates the morphological differences in KG fibres before and after surface treatments In Fig. 2a, the untreated KG fibre exhibits visible tears, likely resulting from the manual separation process. Upon closer inspection at higher magnification (Fig. 2d), these tears are pronounced, suggesting a potential impact on the fibre's performance. Fig. 2b, representing the alkaline-treated KG fibre, reveals a significant reduction in these tears, indicating that the surface treatment effectively mitigated such defects. At higher magnification (Fig. 2e), the removal of the outer layer of the fibre is evident, exposing the inner regions and presenting a rougher texture compared to the untreated fibre (Fig. 2d). Fig. 2c shows the alkaline/silane-treated KG fibre, which displays a surface morphology similar to that of the alkaline-treated fibre. However, under higher magnification (Fig. 2f), it is apparent that the fibre

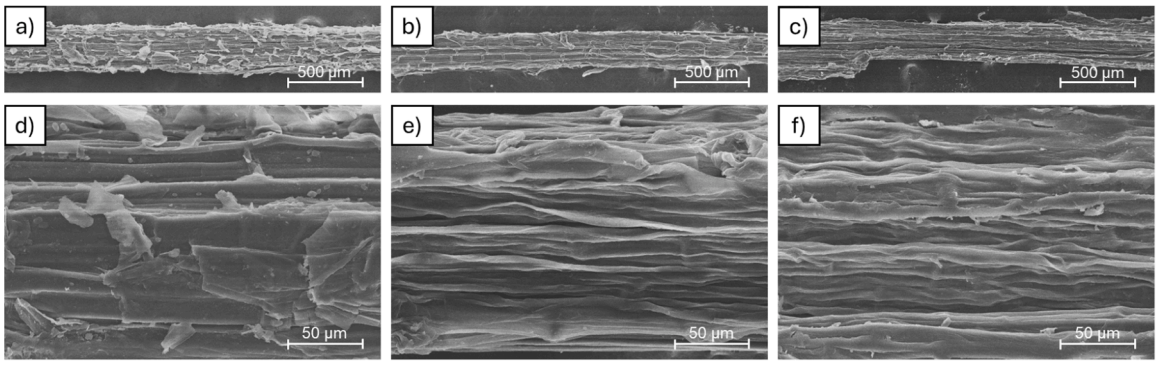

Fig. 2. SEM analysis of the KG fibres before and after the chemical surface treatments: a) Untreated KG fibre (50x); b) Alkaline-treated KG fibre (50x); c) Alkaline/silane-treated KG fibre (50x); d) Untreated KG fibre (500x); e) Alkaline-treated KG fibre (500x); f) Alkaline/silane-treated KG fibre.

surface is more uniform and homogeneous.

3.2. Fourier transform infrared spectroscopy (FTIR)

The FTIR spectra for the untreated and treated fibres are shown in Fig. 3 and the peak assignments are given in Table 1. FTIR analysis shows a few chemical changes induced by the alkali treatment, namely less presence of aromatic compounds represented by the decrease in intensity of the bands at 830 and 780 cm^{-1}, less C = O groups (decrease of the band at 1668 cm^{-1}) and less adsorbed moisture (decrease in the band at 1635 cm^{-1}). On the other hand the bands at 1420 and 1140 cm^{-1} suffered a slight increase in intensity. The FTIR spectrum of the fibres subject to alkaline/silane treatments is quite similar to that of the alkaline treatment, however, it shows a decrease in intensity of the band at ca. 3335 cm^{-1}, its broadening to the lower wavenumber side (ca. 3100 cm^{-1}), and also a broadening of the main band centred at 1050 cm^{-1}.

3.3. X-Ray diffraction

The obtained x-rays diffractograms for untreated and treated fibres are given in Fig. 4. Three major picks were found for 2θ ~16°, ~22° and 35°. The IC results showed that the application of the treatments lead to an increase of the amount of crystalline cellulose. The obtained IC values

Table 1
KG FTIR peaks assignment [23,24].

Wavenumber (cm^{-1})	Assignments
3335	O-H stretching vibrations, primarily from the hydroxyl groups in cellulose and hemicellulose. It may also reflect the presence of absorbed water within the fibre structure.
2915	C—H stretching vibrations, indicating the presence of aliphatic C—H bonds in cellulose, hemicellulose, and lignin.
1687	C = O stretching vibrations, from carbonyl groups in hemicellulose, lignin, and possibly pectin's.
1635	O—H bending vibrations due to absorbed water and C = C stretching vibrations in the aromatic structures of lignin.
1420	C—H bending (scissoring) vibrations, indicating the presence of aliphatic C—H bonds in cellulose, hemicellulose, and lignin
1140	C-O stretching vibrations, indicating the presence of C—O bonds in cellulose, hemicellulose, and lignin.
1000	C—O stretching vibrations and C—H bending vibrations, indicating the presence of C—O bonds and aliphatic structures in cellulose, hemicellulose, and lignin.
830	Aromatic compounds out-of-plane C—H bending vibrations.
780	
560	

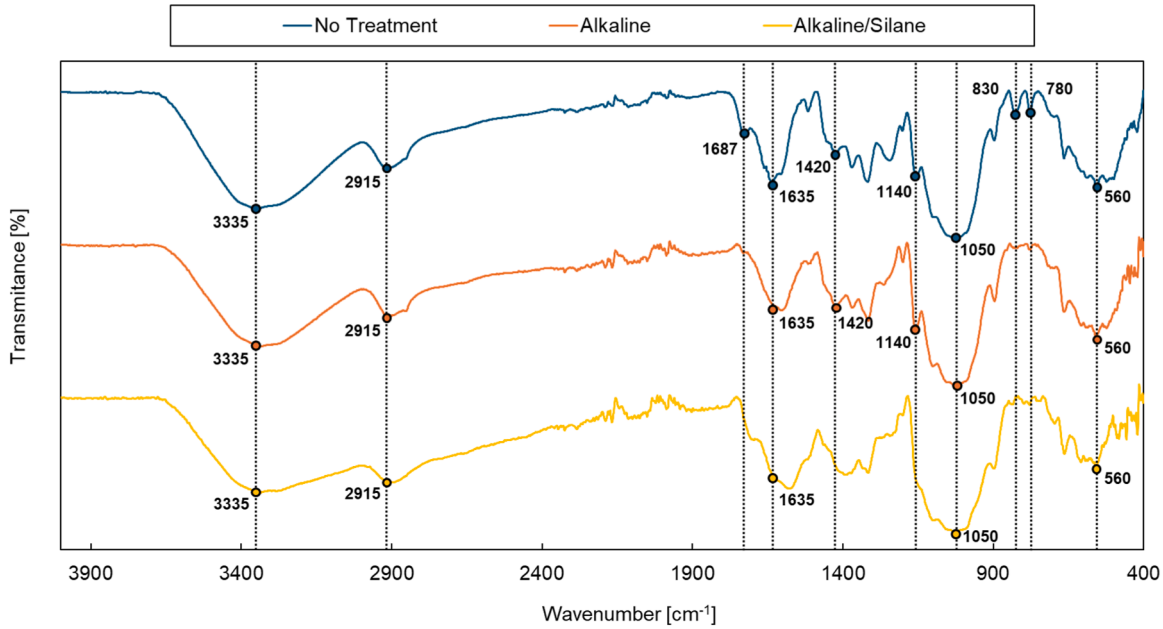

Fig. 3. FTIR spectra of the KG fibres before and after the alkaline and alkaline/silane treatments.

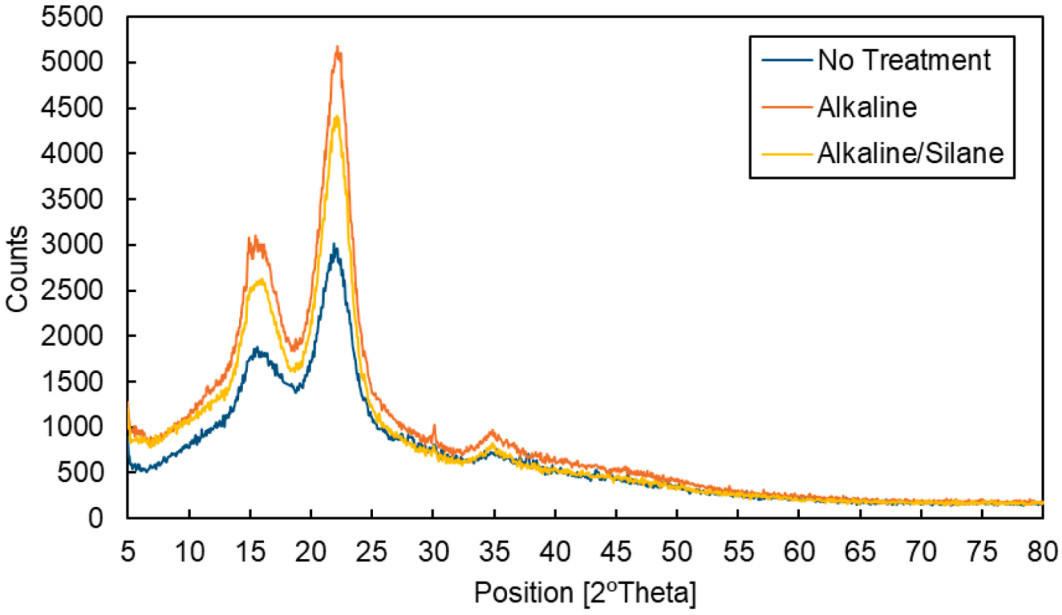

Fig. 4. X-Ray diffractogram for treated and untreated KG.

were 62.5 % for untreated fibre, 71.1 % for alkaline treated fibre and 76.1 % for Alkaline/silane treated fibre.

3.4. Thermogravimetric analysis (TGA)

The TGA curves (Fig. 3a) showed three stages of weight reduction (Fig. 5a). The first stage is characterised by a weight reduction of <10 %. The TGA analysis showed a weight reduction of <10 % up to a temperature of ~200 °C for the untreated fibre and ~260 °C for the alkali-treated fibre and ~250 °C for the alkaline/silane-treated fibre. After these temperatures (2nd phase), there is a dramatic reduction in the amount of material up to ~460 °C for the untreated fibre, ~475 °C for the alkali-treated fibre and ~553 °C for the alkaline/silane-treated fibres. After this temperature, the weight reduction is very low (3rd phase). The shift of the TG curve to higher temperatures, for the treated materials, evidences their increased thermal stability. Through differential thermogravimetric analyses (DTG, Fig. 5b), it is possible to confirm this finding, revealed by the increase of the temperature at which the maximum degradation occurs, for the treated samples, both for the first and second most intense degradation steps. The untreated samples showed a maximum peak at 285 °C, while the samples subject to alkaline and alkaline/silane treatments showed a maximum at 300 °C and 320 °C, respectively.

3.5. Water absorption

Fig. 6a shows that KG slabs water absorption is dramatically affect by the application of the treatments. The alkaline treatment leads to a reduction in water absorption of 40 % while the application of silane treatment after the alkaline treatment leads to a reduction of 56 % in the fibres water absorption (in both cases p-value<0.01).

3.6. Wettability and surface free energy

The water contact angle measurements show that all the tested materials are hydrophilic (see Fig. 6b). The application of the alkaline treatment to KG led to a significantly decrease of the contact angle (p-value<0.01). However, when these KG treated fibres were submitted to a subsequent silane treatment, the water contact angle returned to a value similar to the one obtained for untreated KG (p-value=0.7).

Diiodomethane contact angles (Fig. 6b) were also measured in order to determine the surface free energy of the fibres. The untreated material and the one submitted to the alkaline treatment present similar contact angles with this non-polar liquid, although the dispersion of the values is quite superior for the treated surfaces. The posterior treatment with silane led to a significant decrease of the diiodomethane contact angle.

Regarding the surface free energy and polarity of the fibres (Fig. 6c), both increased with the alkaline treatment. The subsequent silane treatment almost did not affected the surface free energy of the samples, but led to a significant increase of the dispersive component and also to a

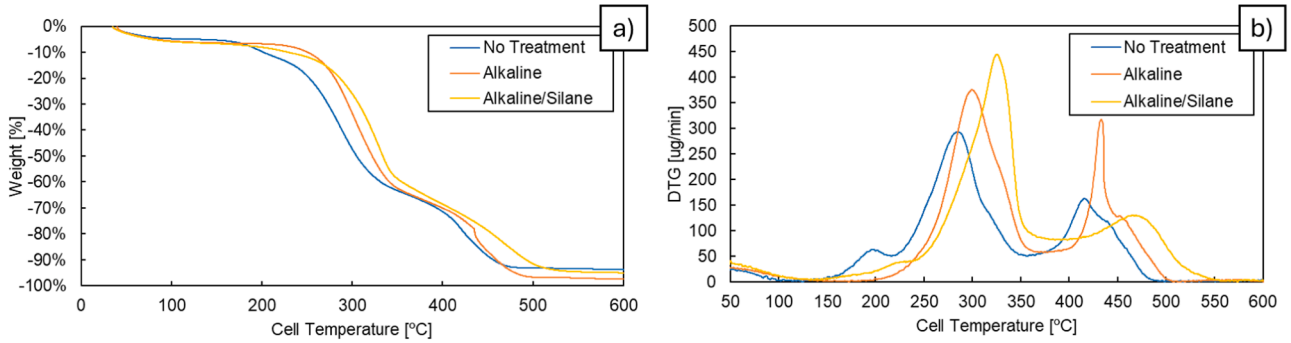

Fig. 5. a) TGA thermogram and b) DTG thermogram of the KG fibres before and after the alkaline and alkaline/silane treatments.

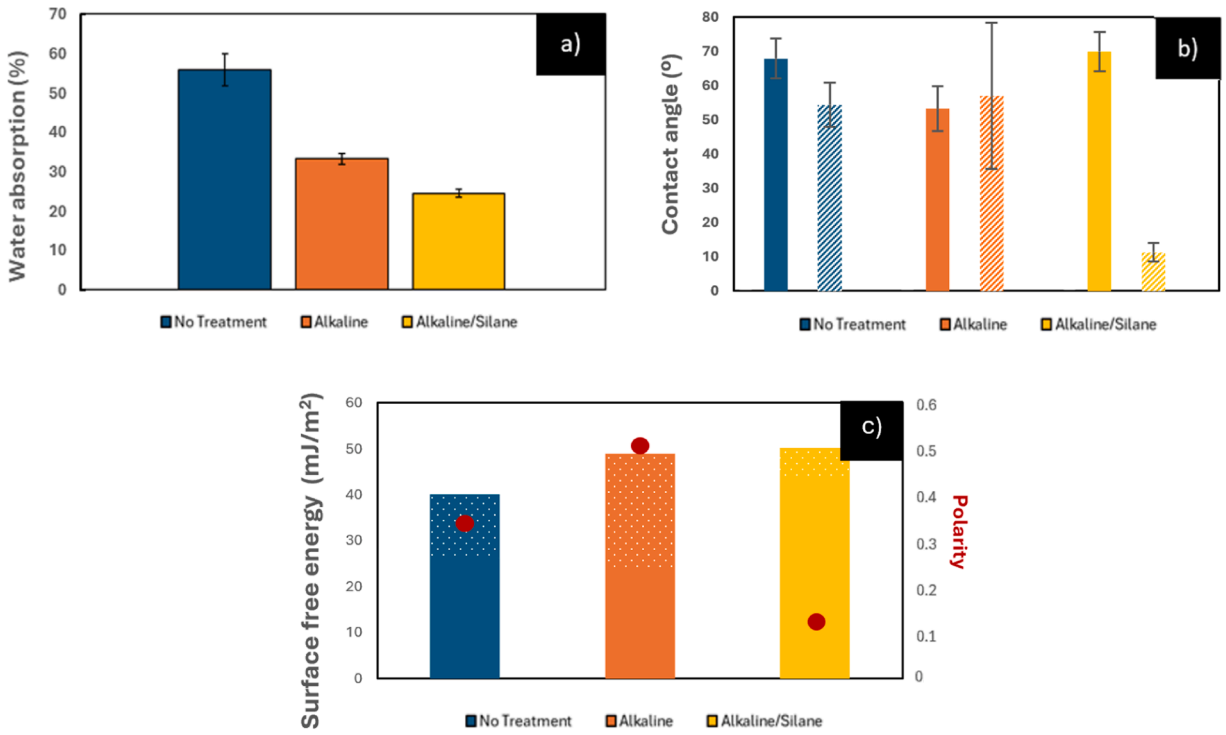

Fig. 6. a) Water absorption, b) water (solid) and diiodomethane (dashed) contact angle and c) surface free energy, dispersive component (solid) and polar (dotted) component (left axis) and polarity (red marks, right axis) of KG before and after the alkaline and alkaline/silane treatments.

notable decrease of the polar component, resulting in an accentuated decrease of the polarity.

3.7. Tensile testing

The compliance from the tensile test set up, determined from the displacement/force versus gage length curve, was 0.0316 mm/N (Fig. 7a). Fig. 7b shows an example of the correction of the measured stress-strain curve data, showing an increase in the slope (Young's modulus) and a decrease in the strain values until fracture. All the stress-strain curves obtained show that the untreated and treated fibres have only an elastic behaviour (Fig. 7c).

The results showed that the application of the alkali and the alkaline/silane treatments led to an increase of the mean values of ultimate

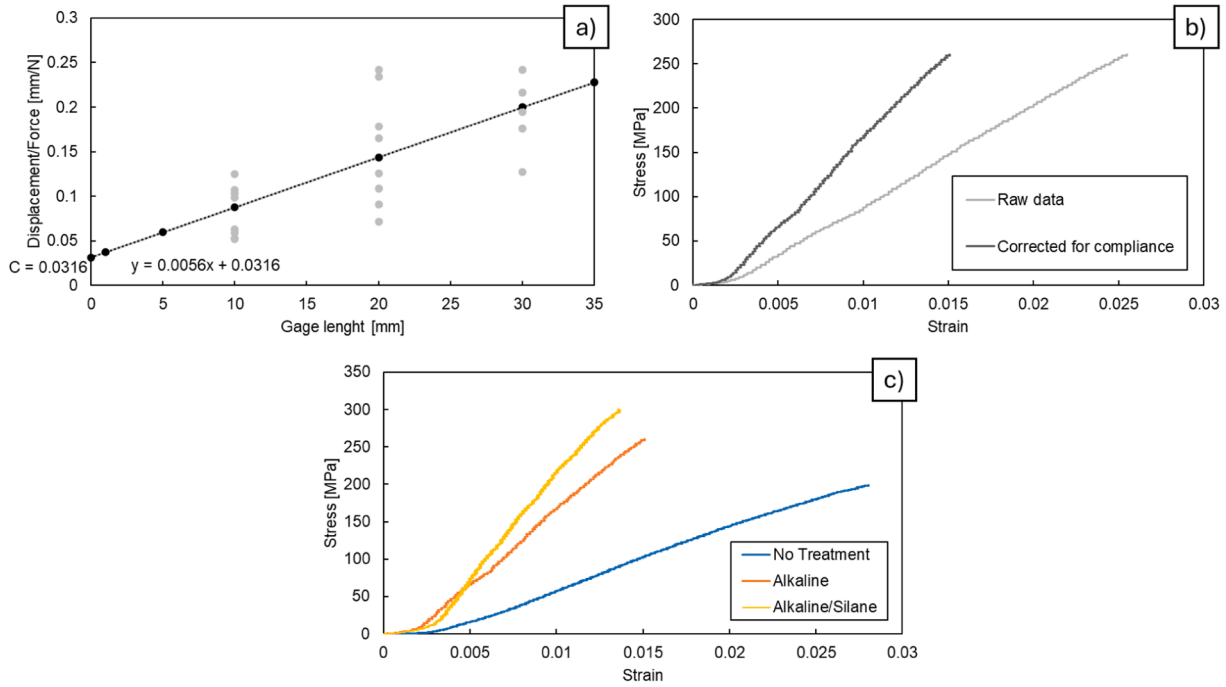

Fig. 7. a) Displacement/force versus gage length for compliance calculation, b) Example of the stress-strain curve data correction, c) Corrected stress-strain curves for the 3 conditions tested.

tensile strength and elastic modulus, and a decrease of the mean values of strain to fracture of the fibres, in comparison to not treated fibres. However, only the elastic modulus increase, when the alkaline/silane treatment was applied was statistically significant, (p-value<0.05).

SEM analysis of the fracture surface reveals no permanent deformation of the surface, which is in line with the elastic behaviour revealed by analysis of the stress-strain curves (Fig. 8), and that fibres presented diameters in the range of 50–250 μm and lengths of 10–50 mm. The approximate fibre yield from raw stem chips was about 35 wt. %, calculated as the mass of recoverable fibres relative to the initial chip mass.

4. Discussion

As reported above, KG is an invasive plant whose dissemination can be controlled by mechanical thinning, leaving a residue that might be used for short fibres production that can be used as reinforcement/filler of polymeric matrix composites. Thus, the aim of present work was to evaluate if KG stem fibre, obtained from mechanical thinning chips, has potential to be used as reinforcement/filler for polymeric matrix composites. To this end, KG was subject to chemical treatments usually used to improve natural fibres properties: an alkaline treatment followed by a treatment with silane. The alkaline treatment conditions used in the present work were chosen according to the parameters often applied to other natural fibres [25]. The silane treatment conditions were based on the work performed by Nafis et al. [26]. After, the effect of the treatments on the most important properties of the fibre was evaluated.

The fibre morphology is affected by the treatment (Fig. 2). The application of the treatments removes surface defects due to the manual extraction of the fibber and defibrillation, that will have impact in the fibre mechanical properties and mechanical adhesion between fibber and matrix. Several studies report defibrillation due to alkaline treatment.

The impact of the treatments on the organic constituents of the KG fibres was studied by comparing the obtained FTIR spectra for untreated, alkaline and alkaline/silane treated fibres (Fig. 3 and Table 1). FTIR analysis shows a few chemical changes induced by the alkali treatment, namely less C = O groups (decrease of the band at 1668 cm^{-1}), suggesting the removal or significant reduction of the hemicellulose and pectin contents, less adsorbed moisture (decrease in the band at 1635 cm^{-1}) and less presence of aromatic compounds represented by the decrease in intensity of the bands at 830 and 780 cm^{-1}. This indicates reduction or removal of lignin because these latter peaks are associated to vibrations of C—H bonds in aromatic rings of the lignin structure. On the other hand, the bands at 1420 and 1140 cm^{-1} suffered a slight increase in intensity, revealing a higher contribution of C—H and C—O bonds. The FTIR spectrum of the fibres subject to alkaline/silane treatments is quite similar to that of the alkaline treatment, however, it shows a decrease in OH groups, associated with the less intense band at ca. 3335 cm^{-1}, which suffers a broadening to the lower wavenumber side (ca. 3100 cm^{-1}), revealing the presence of -NH bonds, from the amino silane. The presence of the silane is also revealed by the broadening of the main band centred at 1050 cm^{-1}, which comes from the presence of Si-O-C and eventually Si-O-Si bonds, if condensation reactions happened to occur following the silane treatment.

The IC quantifies the proportion of crystalline (ordered) regions relative to amorphous (disordered) regions within the fibber's structure. The increase of crystallinity has impact on thermal stability, water absorption, and tensile properties. The diffractogram pick's 2θ ≈16,1°, ≈22° corresponds to (200) and (110) lattice planes of cellulose I and the pick 2θ ≈35 to (004) lattice plane of cellulose IV [27]. The IC obtained for the untreated KG fibres (62.5 %) similar to sisal, hemp [28,29]. The results shows that the application of the alkaline treatment results in an increase of the IC of the fibre. According to FTIR analyses, alkaline treatment leads the removal of hemicellulose and lignin. Compounds that are predominantly amorphous [30]. This effect has been reported for several alkaline treated fibres. In addition, the exposure to alkali causes fibre swelling, allowing cellulose chains to rearrange into a more ordered, crystalline structure promoting stronger hydrogen bonding between cellulose chains, contributing to higher IC.

The thermal stability of the fibres limits the manufacturing method of the composite and the component's service temperature. The TGA results showed that the KG fibres are stable in air up to 150 °C, with water only evaporating up to this temperature (Fig. 5a). Thereafter, a DTG peak appears at 200 °C (Fig. 5b) probably due to the decomposition of hemicellulose. This peak was not found for Eleutecterio et al. [6], probably because the TGA experiments on KG fibres were carried out in a nitrogen atmosphere, leading to a shift in the degradation of hemicellulose to higher temperatures, resulting in an overlap between the hemicellulose and cellulose degradation peaks. The application of the alkali and alkali/silane treatments increases the stability of the fibre up to temperatures close to 200 °C, which can be attributed to the partial/total removal of hemicellulose during the treatments [31–33]. It should be noted that the DTG peak at 200 °C (Fig. 5b) found for the untreated fibres has disappeared. The higher DTG peaks observed for the untreated and treated fibres (peaks between 250–350 °C, Fig. 5b) are probably associated with lignin and cellulose decomposition. The increase of temperature leads to the appearance of other peaks (above 400 °C, Fig. 5b that are associated to the late stages of lignin decomposition and char oxidation. The DTG of the alkali treated fibres showed a shift in the peaks towards higher temperatures, showing that the treated fibres are more thermally stable, probably due to the increase of the cellulose crystallinity. The application of an additional silane treatment to the fibres led to a shift in the DTG peaks to even higher temperatures (Fig. 5b), revealing an increase in the cellulose's thermal stability, probably due to the silane layer formed. In fact, there was an increase in residual mass for the alkali/silane-treated fibres compared to the alkali-treated fibres (Fig. 5a) that can be associated with the silicon

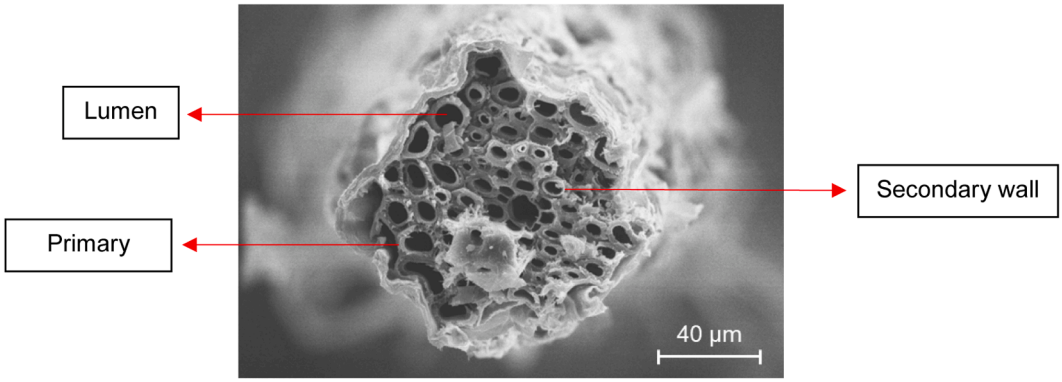

Fig. 8. Example of the fracture surface obtained after tensile testing of alkaline-treated KG fibre.

introduced during the silane treatment. It should be noted that is very difficult to directly compare the results obtained with those found in the literature because TGA data depend on several parameters, such as flow rate, heating rate, and atmosphere, However, the increase in the thermal stability of KG fibre with alkali and alkali/silane treatments observed in the present work is consistent with what has been reported in the literature for the majority of the lignocellulosic fibres [34].

The fibre's water absorption is one of the concerns when using of natural fibre composites. The water absorbed by the fibres can lead to several problems such as reduction of the interfacial bonding between matrix and fibres, nucleation of cracks at the composite matrix and decrease of the mechanical resistance of the fibres. Thus, it is important to apply an effective treatment to the fibres to reduce water absorption. The present results show that the application of the alkaline treatment reduces the water absorption (Fig. 6a) probably due to the removal of hydrophilic compounds such as hemicellulose and pectin, during the treatment and increases in IC. The application of a silane treatment to the previous alkaline treated fibres resulted in additional reduction of water absorption (Fig. 6a). This reduction can be attributed to the formation of a silane layer at the KG fibre's surface, that usually more hydrophobic in their native form.

One of most important issues in the design of a composite material is the degree of adhesion between the fibre and the matrix, that needs to be high to guarantee an efficient stress transfer from the matrix to the fibres. To ensure it, the matrix needs to wet the fibres during the composite manufacturing process. Thus, it is important that both fibres and matrix have the same characteristics in terms of hydrophilicity/hydrophobicity. This can be studied thought water contact angle measurement. The contact angle measurement results showed that the application of the alkaline treatment increases the hydrophilicity, surface free energy and polarity of the KG fibre (Fig. 6b and Fig. 6c). This was probably due to the removal of waxes, oils, and lignin, during the treatment. In addition, the reaction of NaOH with the cellulose introduces more hydroxyl groups on the fibres surface, according to the Eq. (6) [35]:

$$Fibre--OH + NaOH \rightarrow Fibre--O--Na + H_2O \quad (6)$$

However, the application of a silane post treatment led to an increase of the hydrophobicity (Fig. 6b) and, although the surface free energy almost does not change, to a significant decrease of the polarity (Fig. 6c). This was expected since aminopropyltriethoxysilane molecules graft onto the fibres surface through covalent Si-O-C bonds, while exposing their aminopropyl moieties, which alter the balance of polar interactions at the interface and thereby decrease the polar component of the surface energy. Silane also lowers the possibility of fibre agglomeration during the composite manufacture. In addition, the amino groups can increase the adhesion between the fibres and certain polymeric matrix (epoxy, phenolic). The contact angle value obtained for KG alkaline/silane treated fibres was higher than the one obtained by Singh et al. for sisal treated with gamma-methacryloxy propyl trimethoxy silane–A-174 (54.°) [36].These authors reported similar values to the one obtained in the present work (~ 70°) for pyro-phosphato titanate–LICA-38, neopentyl (di-allyl) oxy triacryl zirconate–NZ-39, and N-substi-tuted methacrylamide–QB-012 couple agents applied to sisal fibres.

The effect of both treatments on the KG mechanical properties was assessed using tensile tests. At least 8 tests per group were performed, which is in line with accepted practices for single-fibre tensile testing of natural fibres. The results showed that the KG fibres behaved elastically during all test (Fig. 7c). This behaviour was also observed for other fibres, such as cornua, jute and sisal [37]. The elastic modulus obtained for untreated KG fibres (14.6 ± 7.3 GPa) is close to the values reported for sisal (8.4–19 GPa, [38,39]) using a similar experimental method. However, the tensile strength was lower. This was probably due to greater damage to the fibres' surface during defibration. The variability of the results was evident despite careful preparation of the tests. This phenomenon is in line with the variability found by other authors for lignocellulosic fibres using the same experimental method [40,41]. According to Silva et alI [41], this variability in results is due to differences in the structure of the tested fibres such as the number of lumens, the internal area of the lumens, cell's wall thickness and occasional surface tears generated during manual defibrillation.

The application of alkaline followed a silane treatment led to an increase of 54 % in the elastic modulus and of 30 % in the ultimate tensile strength results averages (Table 2). This is due to the removal of lignin and hemicellulose during alkaline treatment, which are more amorphous than cellulose [30]. In addition to the greater alignment of the cellulose chains along the fibre axis due to the alkaline treatment, increasing IC.

Overall, the results showed that KG stem fibres have potential to be used as reinforcement material/filler in polymeric matrix composites. The TGA results showed absence of significant fibre degradation up to 200 °C, especially if the fibres are pre-treated, revelling that the KG can be used in most of composite production processes such as layup, extrusion, injection, compressing moulding, or additive manufacturing like fused deposition modelling [42,43]. In addition, the results showed that the application of alkaline/silane treatment reduces effectively the fibres water absorption, increasing the compatibility between the fibre and the polymeric matrix. The application of alkaline/silane treatment led to a contact angle of 71° that is not far off the contact angles found for some polymeric matrix (PLA, 80° [44]; Polyamid 77° [45]; Epoxy, 71° [46]). The tensile tests revealed that the KG fibres mechanical properties are within those observed for commonly used natural fibres. In fact, according to values reported by Elfaleh et al. [47], the elastic modules obtained for KG untreated and treated fibres are similar to the values found in literature for jute (10–55 GPa), kenaf (2.86–60 GPa), sisal (9–38 GPa), HardWood (5.2 −37.9 GPa) and Bambo (11–17 GPa).

5. Conclusions

The present work aimed to evaluate the potential of KG stem fibres as reinforcement/load material for polymeric matrix composites. For that KG was submitted to an alkaline treatment and posteriorly to a silane treatment. After, the chemical composition, thermal stability, water absorption, hydrophilicity and tensile mechanical properties of KG were accessed. The obtained results showed that:

- The alkaline treatment led to a reduction of hemicellulose and lignin fibre content (decrease in C = O bonds and aromatic compounds) and increase of crystallinity index. The application of posterior silane treatment provided the presence of Si-O-Si, Si-O-C and amine groups in the samples.
- KG fibres were thermically stable in air until 150 °C without major decomposition of hemicellulose. The treated fibres showed thermal stability up to 200 °C, with the samples subject to silane treatment presenting the highest thermal resistance.
- The water absorption of the untreated KG fibres was 56 %, being reduced for 33 % when the fibres were alkaline treated and 24 % when the fibres had an alkaline/silane treatment.
- The contact angle of the untreated KG fibres was 68°, while for the alkaline treated was 53° and for the alkaline/silane treated was 70°.
- The elastic modulus of the untreated KG fibres was 14.6 ± 7.3 GPa, increasing significantly for 23 ± 3 GPa when the fibres were treated with alkaline/silane.

Table 2
Ultimate tensile strength (σ_{UTS}), elastic modulus (E); strain to fracture (ε_f), cross section area (A) for untreated and treated KG fibres.

	σ_{UTS} [MPa]	E [GPa]	ε_f	A [mm^2]
No Treatment	212 ± 90	14.6 ± 7.3	0.033±0.005	0.025±0.009
Alkaline	261 ± 142	22.5 ± 3.3	0.031±0.008	0.036±0.018
Alkaline/Silane	277 ± 113	23.5 ± 9.3	0.017±0.005	0.019±0.011

- The ultimate tensile strength of the untreated KG fibres was 212 ± 90 MPa. This value raised 23 % for alkaline treated fibres and 30 % for alkaline/silane treated fibres, however the values were not statistically different.

By comparing the obtained values for KG fibres with those reported in literature for natural fibres often used as reinforcement/filler material, it can be concluded that KG stem fibres have a great potential to be used as reinforcement/filler material for polymeric matrix composites.

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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