Flax fiber reinforced PET-G composites with improved interfacial adhesion

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Abstract: Recycled PET-G (rPET-G) composites reinforced with flax fiber were obtained. To improve interfacial adhesion, the fiber was treated by washing and finishing with bio-based epoxidized soybean oil and admixture acid. The effect of fiber surface treatment on its properties and those of the composites was evaluated by TGA, FT-IR and SEM, as well as by contact angle, flexural and tensile measurements. The results indicated delayed degradation of the treated flax fiber with a fiber mass loss of about 4 wt%. The flexural strength and modulus increased for the washed and finished flax fiber compared to the untreated one. While the tensile properties were influenced by structural effects resulting from the fiber treatment. These findings contribute to the development of more sustainable and high-performance natural fiber-reinforced thermoplastic composites.

Keywords: natural fibers, composites, epoxidized soyabean oil, admergin acid, interfacial adhesion.

Kompozyty PET-G wzmocnione włóknem lnianym o zwiększonej adhezji na granicy faz

Streszczenie: Otrzymano kompozyty PET-G z recyklingu (rPET-G) wzmocnione włóknem lnianym. Aby poprawić oddziaływania międzyfazowe, włókno poddano obróbce przez mycie i wykończenie na bazie biologicznej epoksydowanym olejem sojowym i kwasem admerginowym. Wpływ obróbki powierzchni włókna na jego właściwości i właściwości kompozytów oceniano za pomocą TGA, FT-IR i SEM, a także pomiarów kąta zwilżania, właściwości mechanicznych przy zginaniu i rozciąganiu. Wyniki wskazały na opóźnioną degradację poddanego obróbce włókna lnianego z utratą masy włókna wynoszącą ok. 4% mas. Wytrzymałość na zginanie i moduł sprężystości wzrosły w przypadku umytego i wykończonego włókna lnianego w porównaniu z niepoddanym obróbce. Natomiast właściwości mechaniczne przy rozciąganiu zależały od efektów strukturalnych wynikających z zastosowanej obróbki włókna.

Słowa kluczowe: włókna naturalne, kompozyty, epoksydowany olej sojowy, kwas admerginowy, przyczepność międzyfazowa.

The development of high-performance composites from natural resources has gained increasing importance due to climatic changes, with a focus on sustainability and resource-efficient technologies [1–5]. Fiber reinforced thermoplastic composites offer significant advantages over thermoset materials due to their process- and recyclability when compared with thermoset-based composites [6]. However, one of the key challenges in the production of these composites is ensuring a sufficient fiber-matrix interface design [7]. The quality of the interfacial bond condition is critical because it directly affects the mechanical properties of the composite like strength, stiffness, and impact resistance [8, 9].

The utilization of natural fibers, such as flax, jute, and hemp, as reinforcements in composite materials is again a subject of growing interest recently due to their eco-friendly nature, regional availability and sustainable potential [13, 14]. These fibers offer several benefits, including as low density, availability, and biodegradability, which makes them an ideal choice for sustainable lightweight applications [12, 13]. The polar, hydrophilic nature of natural fibers is in stark contrast to the

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typically non-polar, hydrophobic nature of most thermoplastic matrices, such as polyolefins, which results in poor interfacial adhesion [14, 15]. This disparity leads to constrained load transfer, consequently diminishing the composite's static mechanical performance. It is also noteworthy that the majority of natural fibers, in their unprocessed state, are endowed with a protective layer of waxes and pectins that envelop the surface, which, in conjunction with resins, gives rise to adhesion challenges and suboptimal interfacial conditions [16]. Addressing the issue fiber-matrix adhesion problem is therefore critical to realize the full potential of natural fibers in thermoplastic composites [15]. Strategies to enhance the fiber-matrix interfacial adhesion, such as fiber surface treatments or the use of compatibilizers, have been the subject of extensive research to improve the mechanical properties and durability of the composite [9]. Maleated polypropylene (MAPP) has been effectively used as an adhesion promoter to improve mechanical properties of natural fibers reinforced thermoplastics like polypropylene [17]. In [18], the natural fiber was treated with amino silane functionalities and the interaction with a MAPP modified thermoplastic matrix was found to be effective. More information about plant oil-based epoxy′s can be derived from [19]. Thermoset composites made out of natural fiber reinforcement and soybean epoxy were studied in [20, 21].

The objective of this work is to improve the fiber-matrix interface and performance of a more polar thermoplastic matrix combined with pretreated natural flax fibers in a sustainable manner using epoxidized soybean oil and recycled thermoplastic.

EXPERIMENTAL PART

Materials

To study interfacial properties and improve the adhesion of a natural fiber reinforced thermoplastic, a bio-based finish was prepared to coat the reinforcing fabric. Recycled glycolyzed polyethylene terephthalate (rPET-G) as thermoplastic matrix material from Grünstoff® (Frankfurt, Germany) and flax 5040 ampliTex™ from Bcomp® (Fribourg, Switzerland) as reinforcement, were used. The finish was prepared from epoxidized soybean oil and admergic acid, which were supplied by Hobum Oleochemicals GmbH (Hamburg, Germany). Tween 80, a surfactant, was purchased from Sigma Aldrich (Hamburg, Germany). Relevant properties of the used reinforcing fiber and matrix material are summarized in Table 1.

T a b l e 1. Properties of used fiber reinforcements and thermoplastic matrix

Parameter	Abbreviation	Reinforcement	Matrix
Material	–	Flax 5040 ampliTex™	Recycled PET-G
Manufacturer	–	Bcomp®	Grünstoff®
Processing form	–	Fabric	Flakes
Density, g/cm^3	$\varrho_{F/M*}$	1.45	1.38
Areal weight, g/m^2	$AW_{F/M*}$	300.00	414.00
Thickness, μm	d	-	300.00

* F indicates fabric while M indicates matrix

Areal weight of PET-G films was calculated by Equation 1 to further determine the composite composition. Areal weight of ampliTex™ is given by the supplier [22].

$$AW_M = \varrho \cdot d \tag{1}$$

Proposed bonding reactions

The materials utilized and their chemical composition, including available functional groups of the composite components can be derived from Figure 1.

Flax hydroxyl groups (–OH) have been found to react with epoxy groups (also known as oxirane rings) through a nucleophilic ring-opening mechanism. In this reaction, the nucleophilic –OH group binds to one of the carbons in the epoxide and most likely forming ether structures. Oxygen from the original epoxide becomes a hydroxyl group on the adjacent carbon. Carboxyl groups (COOH) from rPET- exhibit a higher propensity to form ester bonds with one of the carbons of the epoxide epoxide's carbons, thereby avoiding the formation of water as a by-product. As a secondary mechanism, formed (epoxy) and available (flax, rPET-G) hydroxyl groups can in turn react with one another to form ether linkages through a condensation reaction. The proposed reactions are shown in Figure 2.

However, covalent bonds are more likely to occur in small numbers due to the low number of terminal functional groups of PET-G. In practice, epoxy resin usually adheres to PET-G via physical interactions (mechanical anchoring, hydrogen bonds), not primarily via covalent bonds.

Analytical methods

The fiber coating resin was prepared and initially analyzed to determine its suitability as a coating material in several aspects. As high flowability is helpful for coating large surfaces, temperature-dependent rheological behavior was evaluated to determine the minimum application temperature. Temperature steps were held for 20 s before further heating to ensure sufficient data.

The curing behavior of the finish formulation was determined by differential scanning calorimetry (DSC) at 15 K/min and 10 mg between 25–340°C with a Mettler/Toledo 3+ (Mettler-Toledo GmbH, Germany).

To assess the influence of an aqueous dispersion as a method of coating application, the epoxy equivalent weight (EEW) and total acid number (TAN) of the coating resin were measured at various time points from a resin-water-dispersion according to ISO 3001 and ISO

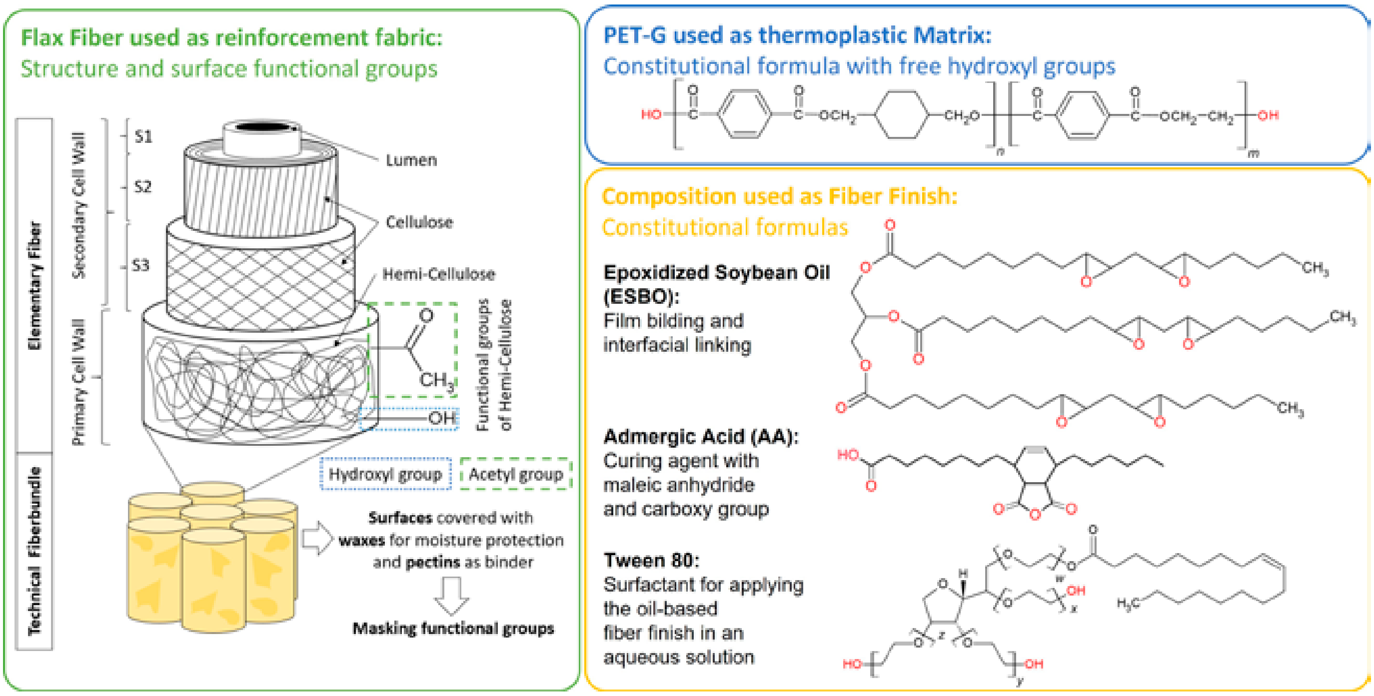

Fig. 1. Materials used as composite components with chemical properties: flax [23], PET-G [24], ESBO [25], AA [26], Tween 80 [27]

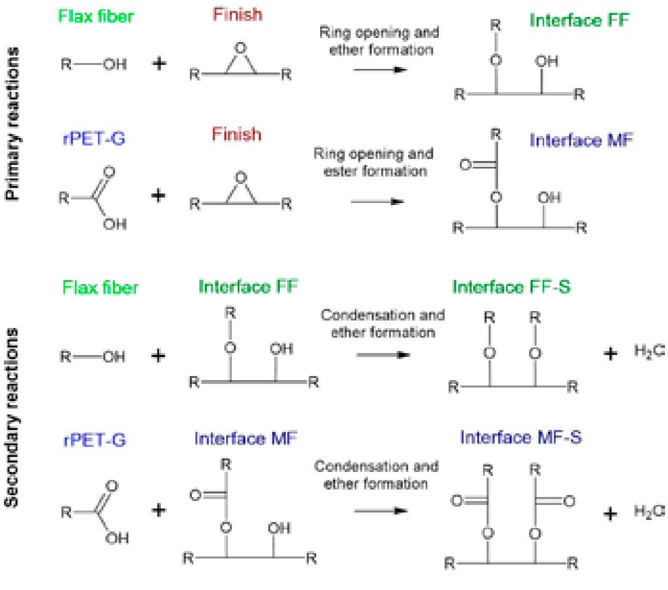

Fig. 2. Assumed interfacial reactions between fiber and finish (FF interface) and matrix and finish (MF interface) in unsaturated and saturated (-S) states

660, respectively. The finish (including surfactant) was mixed in equal amounts with cold distilled water (5, 10, and 20 mL) and stirred at 60°C till all water was evaporated (45, 90 and 180 min).

Thermogravimetric analysis (TGA) was conducted to estimate the absorbed amount of finish on the fiber by comparing it to untreated fibers (the weighing out method is not applicable due to moisture influences) using a Mettler-Toledo TGA 851 (Mettler-Toledo GmbH, Germany).

Contact angle measurements (CAM) were performed using an OCA20 from Data Physics Instruments GmbH (Filderstadt, Germany) to validate the compatibility of the composite components (fiber, matrix, interface). Further details about CAM in regard with natural fibers can be found in [28]. For each measurement, three liquids were used to determine the surface free energy, as shown in Table 2.

Table 2. Total surface tension (SFT_t) with dispersive (SFT_d) and polar parts (SFT_p) of used liquids

Liquid	SFT_t, mN/m	SFT_d, mN/m	SFT_p, mN/m
Diiodomethane	50.00	47.40	2.60
Water	72.80	26.00	46.80
Ethylene glycol	48.20	29.29	18.91

Fourier-transform infrared spectroscopy (FT-IR) was conducted to determine the functional groups of the fiber, cured finish and matrix using a Bruker ALPHA II (Bruker Corporation, USA). To enhance the comparability of the data, the values for rPET-G and cured finish have been normalized to the maximum value of flax ampliTex™. In this context, only relative absorbance can be interpreted.

The flexural and tensile properties of the composites were evaluated using a Zwick/Roell Z050 (Ulm, Germany) device according to DIN 14125 and DIN 527-4, respectively. At least five samples were tested in each test series.

The fracture surfaces of the tensile tested composite specimens were subsequently subjected to selective electron microscopy (SEM) to assess the quality and characteristics of the interface using a Vega MM (Tescan, Czech Republic).

Finish preparation and application

The combination of epoxy and curing agent is considered beneficial in the context of coating fiber finishes, as such combinations have been shown to maintain the structural integrity and ensure the formation of films on the fiber surface [29, 30]. Thus, an under-stoichiometric amount of 75 wt% epoxidized soybean oil (ESBO) and 25 wt% admergic acid (AA) were mixed at 60°C for 10 min. with continuous stirring until the mixture was clear and completely dissolved (supplied by HOBUM Oleochemicals, Hamburg, Germany). Admergic acid is a combination of linoleic acid and maleic acid. It can be used as an anhydride curing agent for epoxies. The epoxy equivalent weight (EEW) of ESBO is 235 g/mol with an epoxy number (EN) of 0.54 mol/100 g, while the hydrogen equivalent weight (HEW) of AA is 189 mol/100 g [31]. An amount of 80.50 g of AA is required for complete cure 100 g of ESBO (or 44.60 wt%), calculated using Equation 2.

$$EN \cdot HEW = \frac{\text{Curing agent (g)}}{\text{Resin (100 g)}} = c \tag{2}$$

$$EN \cdot HEW = \frac{\text{Curing agent (wt\%)}}{c\ \text{(wt\%)}} \cdot 100\,\% = \text{Curing rate (\%)} \tag{3}$$

$$1 - \text{Curing rate (\%)} \cdot EN = \text{remaining } EN \tag{4}$$

Dividing the 25 wt% used AA by the ratio c gives a resulting curing rate of 56.05%, according to Equation 3. The amount of remaining epoxy groups is then calculated from Equation 4 to be 0.24 mol/100 g, available for bonding reactions with the functional groups of either fiber or matrix. The anhydride group of AA is assumed to be fully reacted with the epoxy functions. After preparing the finish and cutting the reinforcement fabric, three liters of an aqueous solution of 5% finish, 1% surfactant (Tween 80®) and 94% distilled water was heated to 70°C. The cut fabrics were then pre-dried (2 h at 110°C), dipped twice separately and dried again at room temperature.

Film casting and composite manufacturing

The thermoplastic matrix material rPET-G was supplied in the form of shredded flakes from (Grünstoff GmbH, Germany). The flakes were compounded into pellets on a Collin ZE25E and cast into film on a Collin flat film line including a smoothing calendar and chill roll (COLLIN Lab & Pilot Solutions GmbH, Germany). Prior to composite fabrication, all cut pieces of flax fabrics (10 × 20 cm) were dried at 110°C for 2 h and the finished pieces were pre-cured at 160°C for 5 min before further processing. Composite fabrication was done by film stacking with alternating reinforcement and matrix layers, using a KV hot-pressing machine (RUCKS Maschinenbau GmbH, Germany). The fabrication parameters are depicted in Figure 3.

To ensure a homogeneous melt flow, pressing frames for tensile and flexural sheets (2.36 mm and 4 mm thickness, respectively) were used. Information about the composite composition is summarized in Table 3.

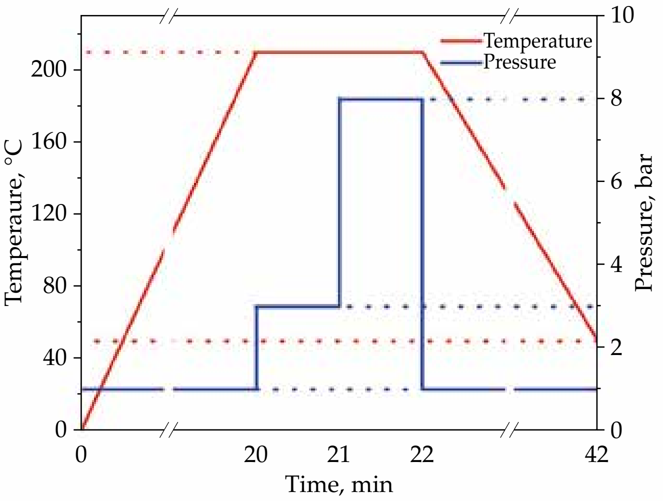

Fig. 3. Film-stacking process parameters

Table 3. Composite sheet composition

Properties	Abbreviation	Tensile test sheets	Flexural test sheets
Layer of matrix	n_M	5	8
Layer of fabric	n_F	4	7
Thickness	–	2.36 mm (standard 2 mm)	4.00 mm (standard)
Standard reference	–	DIN 527-4	DIN 14125
Fiber weight fraction, %	Ψ	36.67	38.78
Fiber volume fraction, %	ϕ	35.35	37.61

Fiber weight fraction was calculated by Equation 5, fiber volume fraction by Equation 6 according to [32].

$$\Psi = \frac{n_F \cdot AW_F}{n_F \cdot AW_F + n_M \cdot AW_M} \tag{5}$$

$$\varphi = \frac{1}{1 + \frac{1 - \Psi}{\Psi} \cdot \frac{\varrho_F}{\varrho_M}} \tag{6}$$

Directional ψ and φ for twill 2/2 fabric is 50% in 0° and 90° each. However, given that the homemade films exhibit thickness distribution and melt accumulation at the edges, fiber wt% and vol% of the composite sheets have only been approximated. After fabrication, the pressed (0°/90° reinforced) composite sheets were cut into specimen and tested for tensile and flexural performance. A practical overview is given in Figure 4.

RESULTS AND DISCUSSION

Finish analysis

Figure 5 shows a temperature dependent dynamic viscosity measurement of the finish preparation, performed

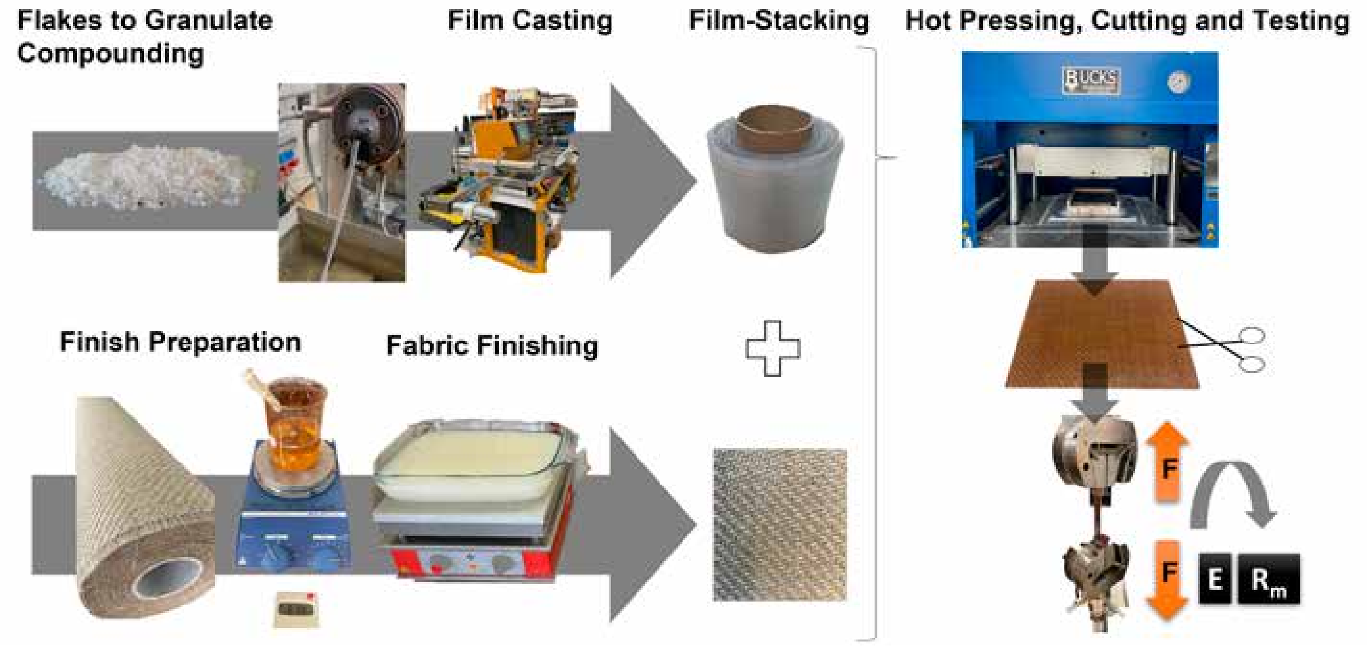

Fig. 4. Practical processing overview

with a Rheotest® RN rheometer (Medingen GmbH, Germany).

The melting point of AA is given at 45°C, where a sharp drop in viscosity can be seen. Minimum viscosity is reached at 70–80°C.

Results from DSC are given in Figure 6. Peak fitting (Gauss) was used to analyze the curing peaks. Since the measurement parameters can strongly influence the DSC results (especially the location and nature of the curing peaks) approximations can be tolerated.

The first curing peak in the region of 185°C is related to the bonding reaction of carboxyl and epoxy groups, the second curing peak at 270°C is primarily related to anhydride opening and reaction with epoxy groups. Also, temperature-based ring-opening of epoxy groups can happen at temperature above 200°C, which can cause uncontrolled, superimposed bonding reactions.

The results of EEW and TAN measurements are presented in Figure 7, with reference value (0 min). It is evident that water is reacting with ESBO and AA over time, leading to a decrease in epoxy and acid functions in asymptotic form, as anticipated. Consequently, the effects are predominantly superficial and water-exposed groups, with minimal to no water permeation observed.

However, the finishing step should be taken quickly to obtain available functional groups. The saturation appears to be around a maximum of 20–25% loss of functional groups.

Results from TGA are depicted in Figure 8. It is evident that the coating leads to a delayed degradation of fiber

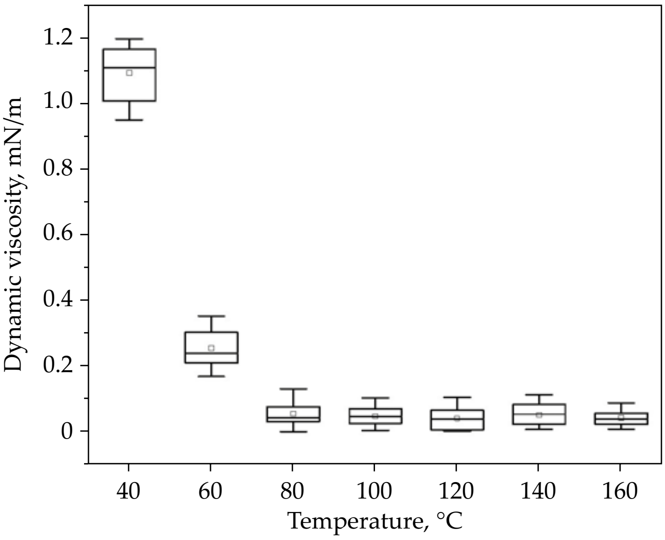

Fig. 5. Rheological analysis of the finishing formulation

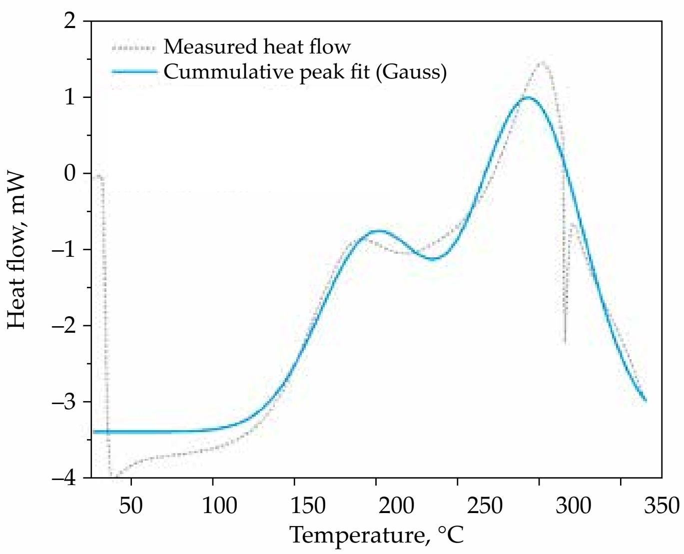

Fig. 6. DSC curves of finish resin (ESBO) and curing agent (AA)

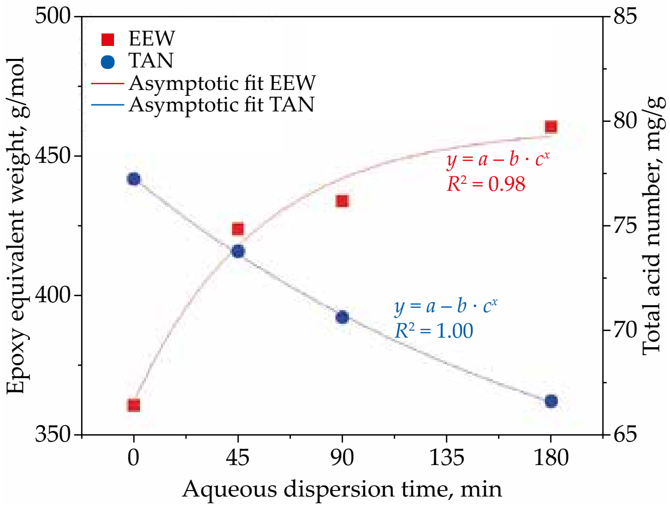

Fig. 7. EEW and TAN of prepared finish resin in dependency of aqueous dwell till

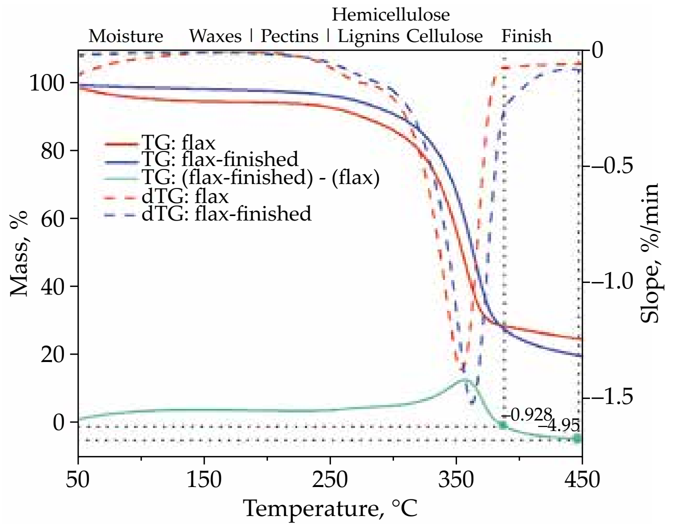

Fig. 8. TGA and DTG curves of untreated and finished samples

components. By comparing the TG and dTG curves, an estimation of the mass content of the fiber finish can be made, which is approximately 4 wt%.

The chemical composition of flax fibers is reported to be approximately 62–72 % cellulose, 18.6-20.6% hemicellulose, 2–5% lignin, 2.3% pectin, and 1.5–1.7% waxes, with an equilibrium moisture content of 8–12% [33, 34]. As the thermal degradation of the flax components is superimposed, the dominant areas of each component are indicated at the top of Figure 8 [35, 36].

Compatibility analysis

Since physical and non-chemical interactions occur at the interfaces of thermoplastic fiber reinforced composites, CAM provides valuable information. CAM demonstrates compatibility between the cured finish and the rPET-G utilized as the matrix material, as illustrated in Figure 9. Measurements on fiber surfaces are typically challenging to execute; however, the flat roving structure of amplitex™ facilitated droplet deposition. The contact angle was measured at least ten times, immediately following the droplet's complete spread to minimize capillary effects. A comparison with sized or unsized (finished) carbon fibers, epoxy resin and different thermoplastic materials can be found in [37].

Furthermore, the binder material utilized during the processing of roving's (Fig. 9.) serves to bridge the interstitial spaces between the fibers. However, it is important to note that this binder material may also exert an influence on the outcomes. The flax fiber surface in its natural

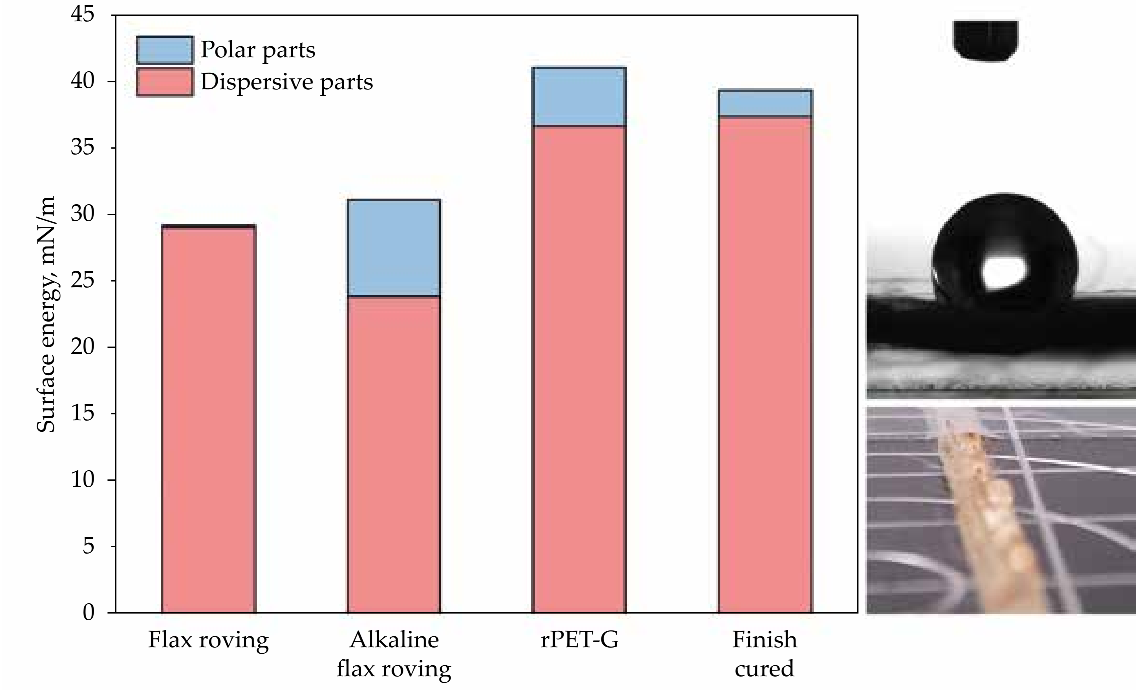

Fig. 9. Contact angle with polar and dispersive parts of surface energy (left), water droplet on flax roving (right)

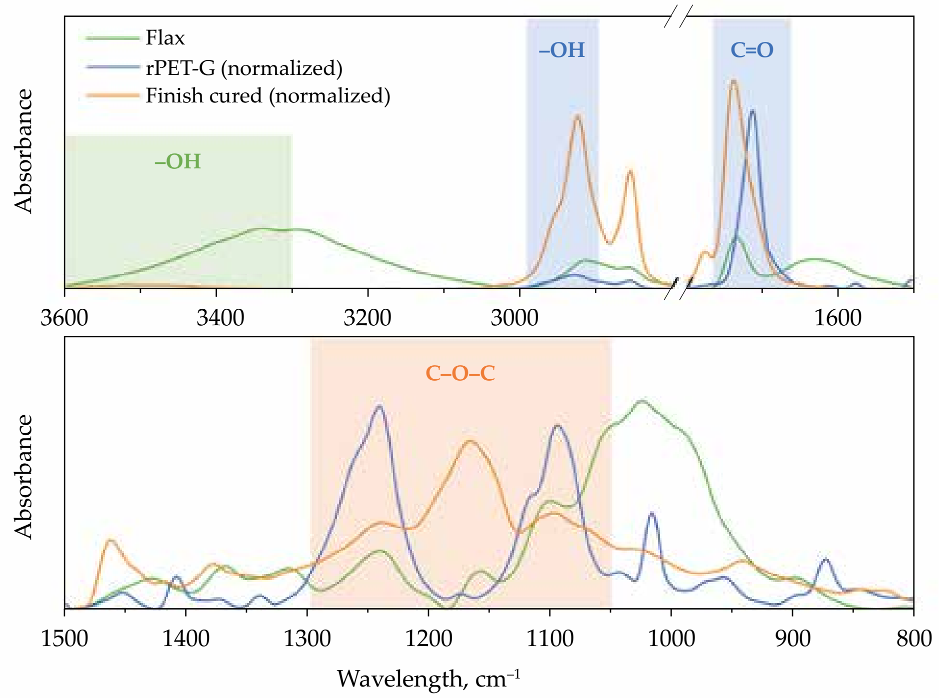

Fig. 10. FT-IR spectra of untreated flax, rPET-G and cured finish, relevant available functional groups are marked

state exhibited negligible polar energy, a phenomenon that can be attributed to the presence of superficial waxes and fatty acids. This observation is consistent with the findings reported in [38, 39]. A low-concentration caustic treatment, such as alkalization, has been proven to effectively swell and clean the fiber from accompanying substances. Mercerization, on the other hand, involves the alteration of the molecular structure through the application of a high concentration of caustic soda. It is imperative to acknowledge that precise CAM is only feasible on flat and closed surfaces. The presented results are intended to illustrate tendencies for the flax roving and do not claim to be exhaustive. However, natural fibers are known to possess a substantial number of hydroxyl groups (–OH) on their surface, a consequence of their cellulose-based molecular structure [38]. The formation of strong covalent bonds necessitates the presence of compatible functional groups.

Figure 10 illustrates the ATR FT-IR spectra with the pertinent bands of relevant functional groups.

FT-IR analysis based on [40, 42] reveals a substantial amount of hydroxyl groups between 3500–3300 cm^{-1} for flax fibers and between 3300–2500 cm^{-1} for rPET-G as part of carboxylic groups, which are characteristic of carboxylic acid structures. The ester groups (C=O) of rPET-G at 1700 cm^{-1} primarily reacted, though some free carboxyl groups (COOH) persist. Ether groups (C-O-C) are located around 1290–1050 cm^{-1} and show the epoxide groups of ESBO and anhydride ring structures of AA. Notably, the presence of hydroxyl groups from the primary cell wall of flax is discernible, despite the presence of thin superficial wax layers, due to the penetration of infrared radiation.

Mechanical analysis

In addition to the untreated and finished samples, a cold water rinsed sample was included as a control for structural effects of the finishing process and to consider cleaning effects. As illustrated in Figure 11, the three-point flexural test results demonstrate that both the cold water-washed (16.82% and 9.57%, respectively) and finished (22.55% and 19.23%, respectively) samples exhibited increased stiffness and strength when compared to the untreated reference. As presented in Figure 12, the tensile test results indicate a significant reduction in tensile strength, with a 11.24% decrease observed in the untreated reference sample and a 6.03% decrease in the treated sample. While the value of Young's modules remained relatively unchanged after the processes of washing and finishing (-2.57% and 0.45%, respectively), tensile strength exhibited a substantial decrease (11.24% and 6.03%, respectively) in comparison to the untreated reference.

As anticipated, tensile strength was found to be more influenced by the structural effects of fabric treatment, such as alignment with the tensile axis or inhomogeneity of fiber distribution. These factors can lead to critical stress concentration and crack initiation. A substantial improvement in mechanical properties was observed when comparing the test results to those of the reference sample devoid of any reinforcements. An investigation of ampliTex™ combined with different thermoplastic matrices yielded similar mechanical results, with a 5% higher fiber volume fraction [43].

According to the technical datasheet provided by the supplier, Bcomp® [22], the mechanical values are notably higher for a fiber volume fraction of 50% (35% in this

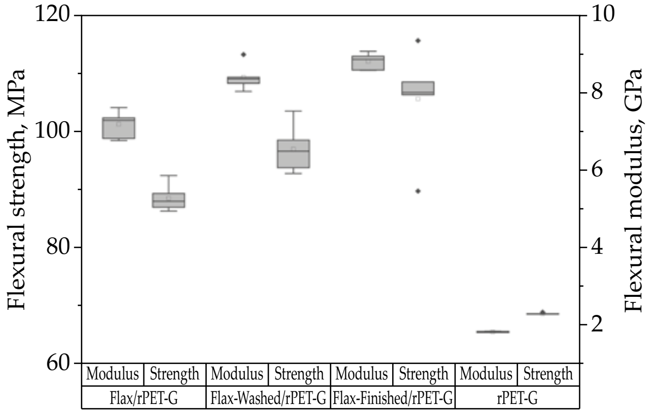

Fig. 11. Flexural properties

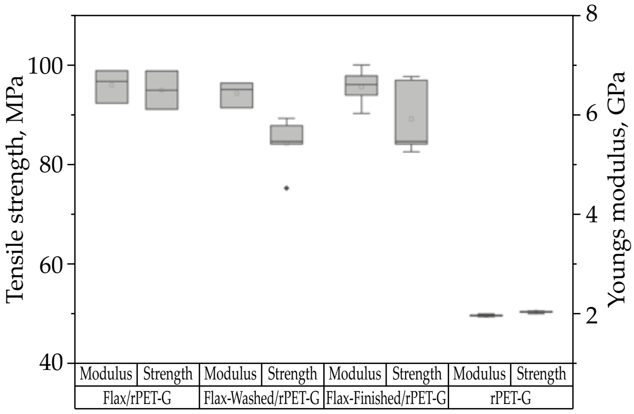

Fig. 12. Tensile properties

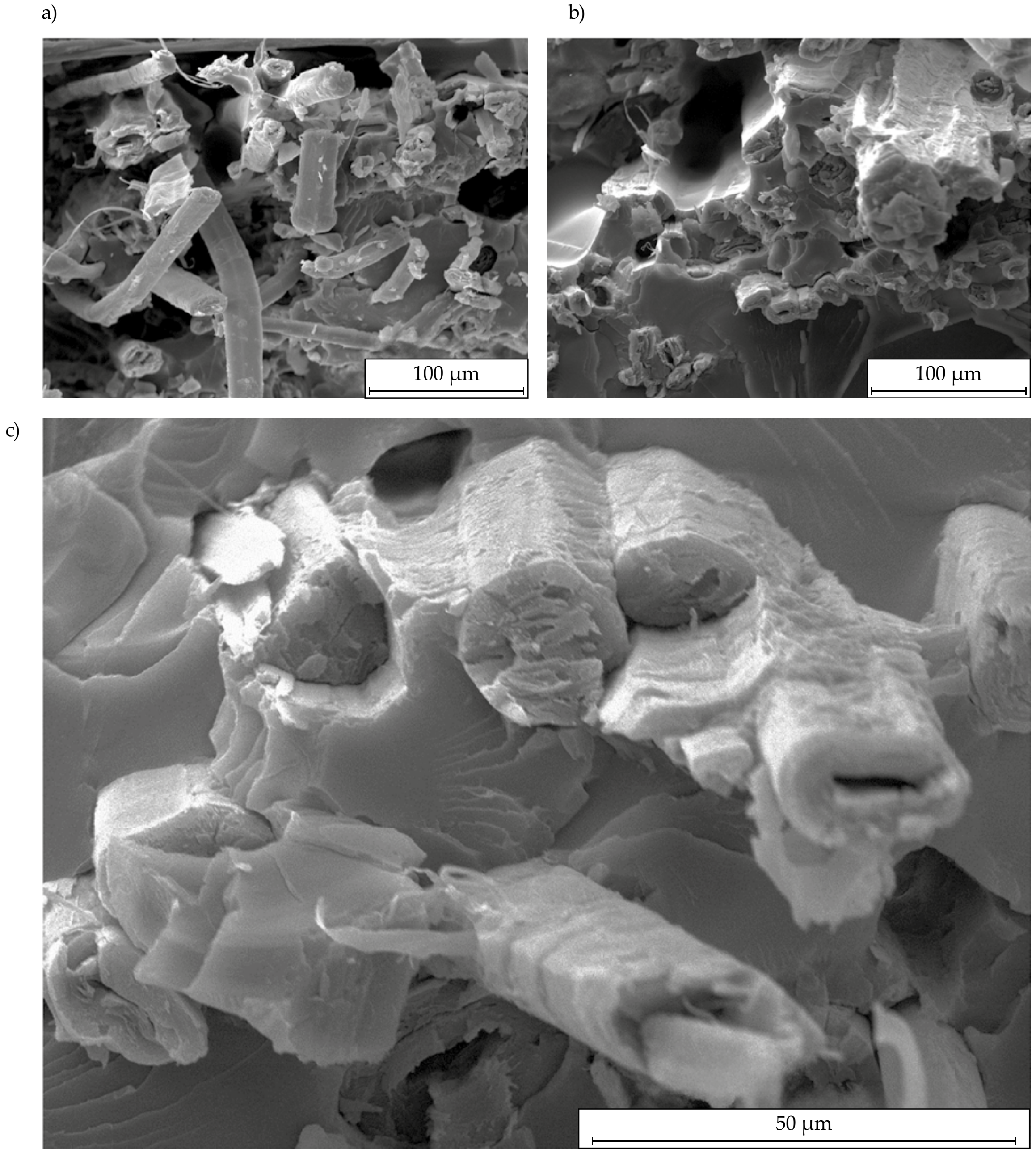

Fig. 13. SEM images of cracks in samples after tensile tests: a) untreated flax reinforcement, b) washed flax reinforcement, c) finished flax reinforcement

study) in a thermoset composite. Of note is the significantly increased Young's modulus using a thermoset matrix system compared to a thermoplastic one in this study. However, strain was measured through traverse movement for this study, which is a rather imprecise method and might have affected the results for Young's modulus.

Fracture surface analysis

To check the mechanical properties and evaluate the interfacial interactions, SEM images were taken, and examples are given in Figure 13.

Untreated flax exhibits substandard interfacial properties, manifested by various forms of fiber draw-out and interfiber breakage. It has been observed that fiber-matrix adhesion is significantly improved by finish-treatment, and the washing step results in moderate improvement, as evidenced by the results of the flexural test. The presence of strong adhesion has been shown to cause fiber breakage; however, draw-out still occurs, but to a lesser extent compared to untreated flax. Fabric pre-treatment also tends to induce structural changes, having a detrimental effect on mechanical performance, as evidenced by the results of the tensile test. Ideally, fibres should be prepared before further processing of the textile. In addition, pre-opening technical fiber bundles by treatment with a weak acid or alkali may be beneficial in maximizing fiber surface wetting and minimizing individual draw-outs from the fiber bundles.

Improvements and sustainable aspects

In considering further strategies to enhance fiber-matrix adhesion, the implementation of additional pre-treatments (e.g., weak alkaline or acid treatments) has been demonstrated to be advantageous [8]. These pre-treatments have been shown to facilitate the wetting of fiber surfaces and mitigate the occurrence of individual fiber pullouts from fiber bundles by prior separation.

Given that epoxidized soybean oil is fully bio-based, and admergic acid is 75% bio-based, the total finish composition is approximately 94% bio-based [31]. The utilization of recycled PET-G as a matrix material in conjunction with natural fibers and an epoxy-based finish appears to be a promising approach, owing to its compatible processing temperature (below 220°C), commendable adhesion properties, and extensive availability at comparable low costs.

CONCLUSIONS

An analysis of flax fibers before and after surface treatment (washing and finishing) revealed significant differences. TGA demonstrated a retardation of degradation of structural components, such as hemicellulose and cellulose, due to the finish coating. The extent of finish uptake was estimated to be approximately 4 wt% of the fiber mass. Three-point flexural tests demonstrated increased stiffness and strength for both washed (17% and 10%) and finished (23% and 19%) fibers compared to untreated fibers. However, tensile tests indicated a decline in tensile strength for the washed (11%) and finished (6%) samples, with Young's modulus remaining unaltered. This reduction can be ascribed to the structural effects of the preparation treatment. Excluding the structural effect of pre-treatment, the finish also increased tensile strength due to enhanced adhesion. SEM revealed inadequate fiber-matrix adhesion in untreated samples, characterized by multiple fiber pullouts. It is noteworthy that the washing and finishing processes led to a substantial enhancement in adhesion, although the fracture characteristics remained variable.

Authors contribution

D.H. – concept, methodology, visualization, writing; H.S. – validation, revision; T.O. – concept, writing; B.S. – validation, revision; R.E.P. – methodology, consultation; M.K. – material testing, data analysis.

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Conflict of 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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