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Towards coupling agent-free composites made from regenerated cellulose/HDPE by UV radiation-induced cross-linking

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ABSTRACT

This research aims to enhance fibre-matrix adhesion in bio-based fibre-reinforced polyolefins without using adhesion promoters. The primary focus is to establish a cross-linking mechanism between cellulose fibres and polyethylene by applying UV irradiation to a UV-transparent matrix and UV-absorbing fibres. The influence of UV treatment on the composite properties is evaluated by tensile, interfacial and interlaminar shear strength tests. The UV irradiation decreases the critical fragment length in single fibre fragmentation tests, indicating an improved fibre-matrix adhesion. The UV-irradiated composites' tensile strength and Young's modulus are found to be ~10% (for 3- and 8-minute irradiation) and ~50% (for 8-minute irradiation), respectively, higher than those of the untreated samples. Furthermore, the UV irradiation leads to an improvement in the interlaminar shear strength by 25%. The variation of the UV-irradiation time (3 min and 8 min) and the comparison of the properties of semi-finished composite sheets and composites also reveal chemical and physical changes in the regenerated cellulose fibres due to heat adsorption. The proposed mechanism of interfacial crosslinking is confirmed by FTIR spectroscopy. The results suggest an approach to overcome poor compatibility between hydrophobic polyolefin matrix and hydrophilic cellulose-based fibres, resulting in adhesive-free bio-based composites.

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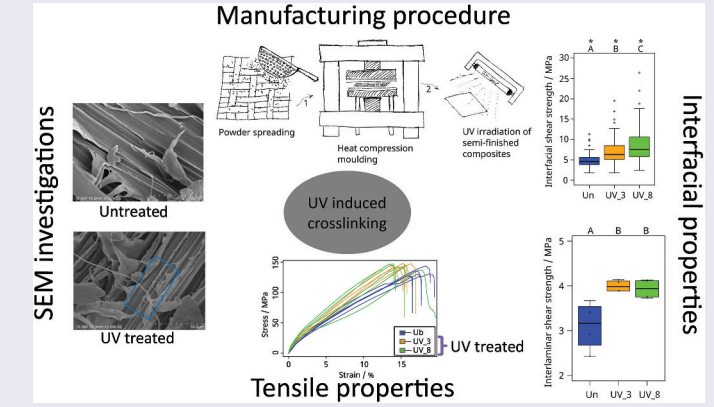

1. Introduction

Over the decades, great efforts have been put into using natural fibres such as hemp, kenaf and flax in composite manufacturing due to the ongoing environmental concerns [1,2]. Recently, bio-based regenerated cellulose (RC) fibres, including viscose, have gained attention for composites due to their unique surface properties, commercial availability, and diverse morphology [3–5]. The mechanical properties of RC fibres vary from 230 MPa to 850 MPa in tensile strength and 11 GPa to 35 GPa in tensile modulus, depending on the manufactururing process and fineness [6–8]. RC fibres, which can be processed into endless fibres, staple fibres, nonwovens, or woven fabrics, offer superior reproducibility and homogeneity compared to natural fibres.

Despite their potential advantages, HDPE composites reinforced with RC fabric remain underexplored, as most studies have concentrated on short-fibre-reinforced HDPE in injection-moulded systems [9,10]. In comparison, rayon fabric-reinforced polypropylene composites have demonstrated significantly enhanced mechanical properties, achieving tensile strength and modulus values of 98 MPa and 3.1 GPa, respectively, compared to 20 MPa and 1.12 GPa for unreinforced polypropylene [11]. The use of RC fabrics in composites offers consistent mechanical properties. Although RC fibres are currently costlier than glass fibres, they are less expensive than hackled long flax and offer significant environmental benefits, such as lower CO_2 emissions compared to traditional reinforcements. With the increasing focus on de-fossilisation and sustainable materials, RC composites are emerging as a viable alternative for areal load-bearing applications, such as automotive interior components. When combined with polyolefin matrices like polyethylene (PE), which are low-cost, chemically inert, and widely available, and with future availability from bio-based sources, these composites offer a promising balance between performance, affordability, and sustainability.

However, incorporating hydrophilic, moisture-absorbing RC fibres into a hydrophobic matrix such as HDPE poses a significant challenge due to poor fibre-matrix adhesion [9–11]. Traditional methods to address this issue often involve the use of chemical coupling agents, such as silane or maleic anhydride. While effective, these methods have significant drawbacks, including hindered recyclability, high water

consumption, health risks, and the need for strict handling protocols due to the chemicals involved. In contrast, irradiation offers a cleaner and more sustainable alternative, as it eliminates the need for additives, does not consume water, and does not interfere with the recyclability of composites. These advantages make irradiation a promising solution for overcoming the limitations of traditional coupling agents. The aim of this work is to achieve fibre-matrix adhesion comparable to those obtained through UV-induced monomers or other physical treatments reported in the literature. However, in this study, we demonstrate that UV treatment alone is sufficient to significantly enhance adhesion in regenerated cellulose fabric-reinforced HDPE composites. This approach simplifies the process by eliminating the need for additional UV-curable monomers, providing a more straightforward and sustainable solution. Various methods of chemical and physical treatments of natural fibres to improve the composite quality have been reported and discussed in detail in numerous recent reviews [3].

Here, we focus on the effect of UV irradiation on the performance of fibre-reinforced composites. UV irradiation was used to change the surface properties of chemically pretreated fibres and to enhance the adhesion and has been studied for grafting photo-induced monomers [9,10] or activating jute [12], coir fibre surfaces [13], as well as glass fibres [14] and carbon fibres [15] to enhance the fibre–matrix adhesion.

In addition to UV irradiation, extensive research has been carried out to improve fibre-matrix adhesion by plasma treatment [16–18], gamma-irradiation [19,20], incorporation of additives [21], or modifying the matrix [21–23]. Some earlier studies used physical treatments to enhance fibre–matrix adhesion by pre-irradiation [13,16,17] on fibres or post-irradiation on manufactured composites [19,20]. Irradiation or post-irradiation of manufactured composites is not novel; in recent years, it has been particularly explored in jute-reinforced thermoplastic [19] and thermoset composites [20] such as jute/PP, jute/polyester, and jute/Epoxy using gamma irradiation. Motaleb et al. [20] achieved notable tensile strength in jute/Epoxy, despite fabric property loss, rather related to testing of the fabric in bulk than to single jute fibre properties. Mina et al. [19] observed a 12% increase in untreated jute/PP composites, rising to 18% with triple superphosphate pre-treatment before irradiation. Despite achieving cross-linking between fibre and matrix, the extent of improvement remains limited, possibly due to sample thickness and process-induced changes in fibre and matrix properties. Several studies suggested that physical treatments could positively or negatively affect the mechanical properties of the fibres, depending on the duration and intensity of the physical treatments [13–16]. Mina et al. [19] and Motaleb et al. [20] reported an increase in Young's modulus of the composite, which was assigned to process-induced changes in fibre and matrix tensile modulus. To support this, no influence of irradiation on the fibres or matrix properties was presented [24]. Unlike gamma irradiation, UV irradiation has not been extensively studied in the post-irradiation of manufactured composites. Therefore, when considering UV irradiation, it is critical to select the suitable duration, as process-induced changes in fibre morphology [13] and matrix [15] exceeding 10 min were reported. While shorter UV durations do not affect RC fibre/HDPE adhesion [5], Bahners et al. [25] found significant improvement with 10-minute irradiation. Moreover, 10 min irradiation on RC fibres increased pore volume [26]. Additionally, research by Quan et al. [15] revealed minimal impact on PPS fibre properties at 3 minutes but stiffening at 10 minutes, indicating UV irradiation has a dual effect on fibres and matrix.

Initial tests showed no changes in fibre properties at 3 minutes but oxidation at 10 minutes, leading to an 8-minute duration adjustment to mitigate effects.

Most of the early works have shown that fibres, matrix or adhesion properties strongly depend on irradiation intensity. Therefore, for a deeper understanding of the impact of UV irradiation on manufactured semi-finished composite sheets, a study of the processing- and parameter-induced changes in fibre and matrix properties is essential. In this case, when UV irradiation is applied to treat the fabricated semi-finished composite sheets, it is crucial to use a UV-transparent matrix such as polyolefins so that the irradiation can be adsorbed at the fibre–matrix interface. Such an approach was reported by Bahners et al. [25] using direct UV irradiation of polyethylene terephthalate (PET)/PE composites.

The present study addresses the primary challenge of avoiding adhesion-promoting additives by exploring direct irradiation as an alternative approach and pioneers the investigation of direct UV irradiation on regenerated cellulose fibre/HDPE composites. Herein, we examine the correlation between composite performance, UV-induced cross-linking and process-induced changes in fibre and matrix. The adhesion between fibre and matrix was characterised through micro- and macro-mechanical investigations of the single fibre fragmentation and tensile and double-notch tensile tests. Furthermore, mechanical tests on HDPE films and infrared spectroscopic analyses of polymers from the composite materials are used to assess adhesion. The SEM investigations will provide insights into the interaction between fibre and matrix. We hypothesise the formation of UV-activated covalent bonds between the UV-transparent HDPE polymer matrix and the UV-absorbing RC fibres.

2. Materials & methods

2.1. Materials

Composites, fabricated using the regenerated cellulose (RC) plain weave fabric (Cordenka® of areal mass 450 g/m^2, Cordenka GmbH, Obernburg, DE) as a reinforcement, were used for the characterisation of interlaminar shear and tensile properties. A low undulation angle RC plain weave fabric (areal mass 220 g/m^2, BÜFA Thermoplastic Composites GmbH & Co. KG, Oldenburg, DE) was used to observe its influence on the composites' Young's modulus. Danufil® single RC fibres with a fineness of 3,3 dtex (diameter of approx. 16.6 µm, Kelheim Fibres GmbH, Kelheim, DE) were used in single fibre fragmentation tests. High-density polyethene (HDPE) from LyondellBasell Industries (Lupolen 5031 L, 0.952 g/cm^3 of density, 8.5 g/10 min of melt flow rate at 190 °C) was employed as a matrix [27].

2.2. UV irradiation

The semi-finished composite sheets were irradiated using a UV broadband lamp with a wavelength spectrum from 250 nm to 300 nm (UVACUBE 2000, Dr Hönle, Munich, DE). The irradiation dose was varied by the exposure time while maintaining a consistent specific power of 50 W/cm. The distance between the lamp and the sample is kept constant to 10 cm. The sheets were exposed to UV light for three minutes (UV_3) and

eight minutes (UV_8) on each side. The duration of the treatment was chosen according to previous research on the composites based on polyolefin matrix [25,28].

2.2.1. Tensile properties of fibres

The tensile properties of RC single fibres prepared from the untreated and UV-irradiated composite samples were tested (DIN EN ISO standard 5079 [29]) using a Fafegraph M (Textechno, Mönchengladbach, DE) with 10 mm/min crosshead speed using a load cell of 100 cN. The single fibres were fixed at 20 mm gauge length in the PVC pneumatic clamps. Before testing, the fibres were conditioned (DIN EN ISO standard 139 [30]) in a climatic chamber (VCL 4003, Vötsch Industrietechnik GmbH, Reiskirchen Lindenstruth, DE). Eighty fibres for each variant were tested, and Young's modulus was evaluated for each fibre in the linear elastic region of the stress–strain curve. The dimensions and morphology of untreated and UV-irradiated RC fibres (for sample preparation, refer 31) were examined using a scanning electron microscope (SEM) JEOL 6510 (SE – electron detection, JEOL GmbH, Eching, DE). The fibre diameter was analysed with ImageJ (version 1.48 v, Wayne Rasband, National Institutes of Health, USA) and used to calculate the tensile properties. A statistical analysis aimed to compare the influence of the UV irradiation duration on the diameter and its respective tensile properties.

2.2.2. Tensile properties of HDPE sheets

The HDPE sheets were produced using the granulates with a hydraulic press (LaboPress P200S, Vogt, Berlin, DE) at 180 °C for 10 minutes under 10 bar areal pressure. Before testing, the samples were conditioned (DIN EN ISO standard 291 [32]) in a climatic chamber. Tests were done (DIN EN ISO standard 527–3 [33]) under axial load on a Zwick universal testing machine (Z020, Zwick/Roell, Ulm, DE; load cell 20 kN).

2.2.3. Single fibre fragmentation test

The fabrication, preparation and characterisation of the single fibre fragmentation test (SFFT) were done according to Graupner et al. [34]. The samples were manufactured using a hydraulic press at 180 °C for 5 minutes under 10 bar areal pressure and cooled down to 40 °C under pressure. SFFT was performed using the Zwick universal testing machine (Z020 Zwick/Roell, Ulm, DE) at 16 mm gauge length and 2 mm/min crosshead speed using a 500 N load cell. The fragment lengths were measured with a polarisation microscope (ADL-601P, Bresser GmbH, Rhede, DE), which allowed the determination of the critical fragment length L_{fc} in mm and IFSS in MPa using the formula by Feih et al. [35]. A direct comparison of L_{fc} was given to assess the fibre-matrix adhesion. A good fibre-matrix adhesion is expected to result in lower critical fragmentation lengths and vice versa.

2.3. Composite manufacturing

The fibre-reinforced semi-finished sheets were manufactured with a dimension of 180 × 180 mm^2 with a hydraulic press (Labopress P200s, Vogt, Berlin, DE) at 180 °C for 5 min under an areal pressure of 20 bar. The manufacturing process is illustrated in Figure 1. The RC fabric was dried for 24 hours at 60 °C before the matrix impregnation.

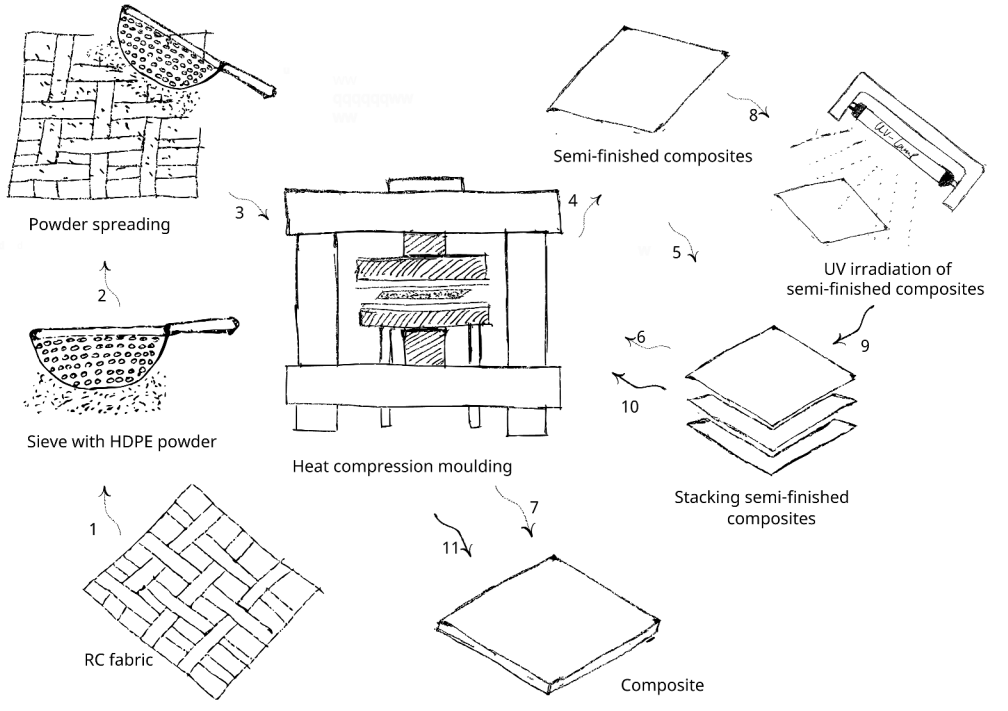

Figure 1. RC fabric (Cordenka) (1) was coated with HDPE powder using a sieve (2); the coated fabric (3) was then compressed in a heat compression mould into a semi-finished composite sheet (4); the untreated semi-finished composite sheets were stacked (6) and processed by compression moulding into a composite (7); the semi-finished composite sheets (4) were processed to the UV irradiation (8), then the irradiated semi-finished sheets were stacked (9) and the process by compression moulding (10) into the UV-treated composite sample (11).

The manufactured semi-finished composite sheets were subjected to UV irradiation, as described in section 2.2. '*Semi-finished composite sheets*' refer to a single layer of RC fabric as a reinforcement; '*Composite*' refers to multiple layers of semi-finished sheets. The composite samples for the tensile and interlaminar shear tests were fabricated by pressing several stacks of semi-finished sheets at 180 °C to the desired thickness. To achieve a fibre volume content of 65%, the composite samples were compacted for 5 minutes under 10 bar areal pressure, followed by 5 minutes under 45 bar areal pressure. This research involved tests on three series of composites: Untreated (Un), treated UV_3 and UV_8.

2.4. Tensile test

The tensile test was carried out according to the DIN standard 527–4 (Type 1B) [36]. Specimens were prepared using a CO_2 laser (150 W, Sabko GmbH, Trierweiler, DE). Before testing, specimens were conditioned for 24 hours at 23 °C and 50% rel. humidity. The test was performed with a Z020 Zwick universal testing machine at 2 mm/min crosshead speed. The strain was optically determined via a video extensometer (Zwick/Roell, Ulm, DE) with a distance between the measuring marks of 50 mm.

2.5. Double-notch tensile test

The double-notch tensile test was used to determine the interlaminar shear strength (ILSS) and, thus, the characterisation of fibre-matrix adhesion. The specimen preparation and testing followed the standard DIN 65,148 [37], except for the specimen length. Five test specimens (65 × 25 mm^2) per series were tested. The notches were created with a Proxxon mill MF 70 (Proxxon, Wecker, LUX) and a file tool. Tests were performed with a Zwick universal testing machine (Z020 Zwick/Roell, Ulm, DE) at 2 mm/min crosshead speed and 40 mm gauge length.

2.6. Thermal analysis

Differential scanning calorimetry (DSC) analyses were performed on untreated and UV-treated fibre samples with 4–5 mg. The tests were conducted using a Q20 instrument (TA Instruments, New Castle, USA). The analyses were conducted under a nitrogen atmosphere, and cooling was achieved with compressed air. A standard heating rate of 10 K/min was applied for the first heating run.

2.7. Fourier transform infrared spectroscopy (FTIR)

The test samples were measured using FTIR spectroscopy with the IR Prestige-21 FTIR spectrophotometer from Shimadzu (Kyōto, JP), using LabSolutions IR software in conjunction with the Golden-Gate Diamond ATR unit. The sapphire press piston for solid samples was utilised.

2.8. Statistical analysis

The statistical evaluation of the results was evaluated with the open-source software R (R studio 1.4.1103/R version 4.0.3). The data's normal distribution ($\alpha = 0.05$) was checked using the Shapiro–Wilk test. The Tukey–Kramer HSD test ($p < 0.05$) was performed to check the significant differences among normally distributed data. A Wilcoxon test ($p < 0.05$) was performed in the case of not normally distributed data. A p-value of less than 0.05 indicates that the probability of the observed differences occurring by random chance is less than 5%, confirming statistical significance. The results were shown as Box–Whiskers' plots showing the median, the Quartile 25, the Quartile 75 and the interquartile length (Whiskers). An asterisk (*) represents the results that do not follow a normal distribution, and different letters are used to indicate significant differences between median values.

3. Results and discussion

3.1. Effect of UV irradiation on the RC fibres

It is essential to characterise the possible effects of the UV treatment on the single components to facilitate the understanding of the behaviour of a composite material. Single fibres were extracted from the untreated and treated composites for the fibre tensile tests. The results of the tensile tests on the untreated and irradiated RC fibres were

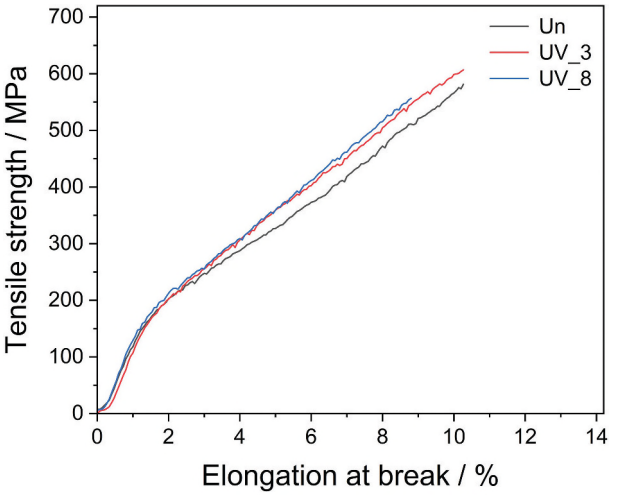

Figure 2. Stress-strain behaviour of the untreated (un) and the treated UV_3 & UV_8 RC fibres.

analysed using descriptive statistics, as explained in the experimental section. The results of the tensile tests in Figure 2 show no significant change between the properties of the untreated and the UV_3 treated fibres (based on statistical analysis). However, a prolonged treatment (UV_8) results in stiffening of the RC fibres with a significant decrease in tensile strength (TS) from 583 MPa in untreated samples to 535 MPa; a 2.3% increase in tensile modulus (TM), from 16.10 GPa to 16.47 GPa; and a decrease in strain at break. Similar observations that the UV-irradiated fibres dependent on the treatment conditions may become more brittle but stiffer were reported earlier that extended exposure resulted in a reduction of the TS due to the decrease in the moisture and the weakening of the fibres as a result of partial chain scission [13,26,38,39].

As seen in Figure 3, the morphology of the RC fibres shows no significant changes in the shape or estimated average dimension (~12.9 ± 1.5 μm) for treated and untreated fibres.

The thermal characteristics of the RC fibres after the applied UV irradiation were assessed using DSC measurements. The DSC thermograms of fibres from untreated and UV-irradiated RC fabrics in Figure 4 show a broad endothermic drop near 100 °C due to the evaporation of physically absorbed water due to the hydrophilic characteristic of the

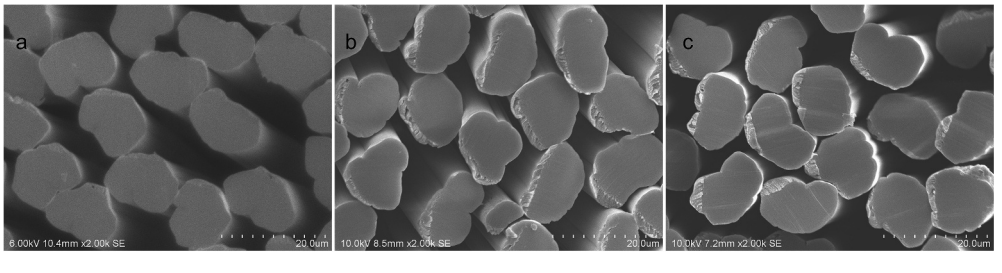

Figure 3. Cross-sectional micrographs of RC fibres of untreated (a) and UV-treated for 3 min (b) and 8 min (c).

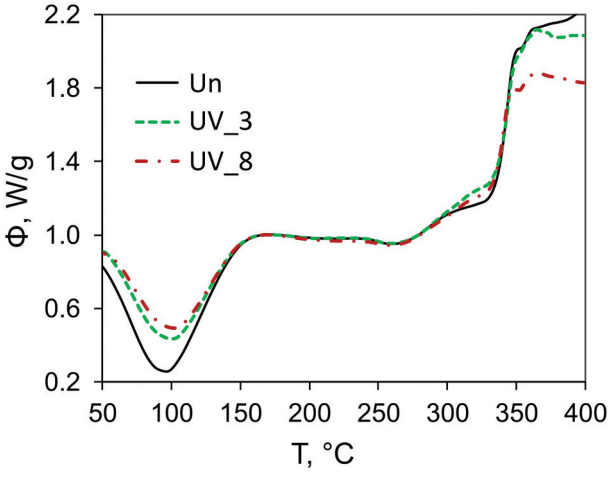

Figure 4. DSC thermograms of untreated (un) and UV-treated for 3-minute (UV_3) and 8-minute (UV_8) RC fibres.

RC fibres. The peak shifts from ca. 94 °C for untreated fibres to 104 °C and 108 °C for the UV_3 and UV_8 fibres, respectively, followed by an exothermic increase extending from 280 °C to 340 °C, which is attributed to the decomposition of hemicelluloses and cellulose [40]. Prolonged irradiation with broadband may lead to partial oxidative degradation of RC fibres when a dehydrogenated structure with more aromatic moieties is partially formed [41]. However, we note that the RC fabric was irradiated through the HDPE matrix in the fabrication procedure, which probably prevented moisture loss during the treatment.

3.2. Effect of UV irradiation on the matrix polymer

The tensile properties of both untreated and UV-treated HDPE sheets are summarised in Figure 5 and Table 1, focusing on the influence of UV irradiation and its impact on Young's modulus at the composite level. UV irradiation slightly improves tensile properties, attributed to HDPE's small absorption in the 250–300 nm wavelength range (Figure A1, Appendices), where HDPE exhibits 90% transparency. This absorption could lead to cross-linking in the HDPE matrix polymer, forming a few radicals that recombine or 'bridge' to secondary chains [42].

3.3. Fibre-matrix adhesion tests

The single fibre fragmentation test (SFFT) results are shown in Figure 6 and Table 2. The main interest is to discuss the effect of UV irradiation on the fibre-matrix adhesion and the influence of UV-induced changes in the fibre tensile properties on the critical fragment length. Fragment lengths were measured using a polarisation microscope, as sketched in Figure 7.

The UV_3 and UV_8 samples demonstrated reductions in critical fragment length by approximately 43%, from 0.519 mm to 0.296 mm and 50.5%, from 0.519 mm to

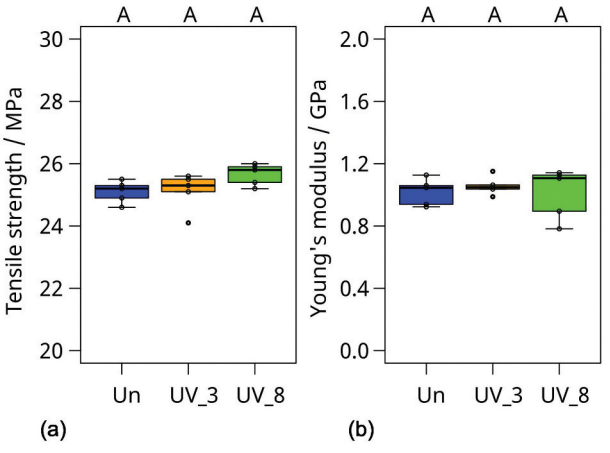

Figure 5. Box-whisker plots of the tensile characteristics, i.e., tensile strength (a) and Young's modulus (b) of the untreated and UV-treated high-density polyethene sheets for 3 and 8 min. An asterisk (*) represents the results that do not follow a normal distribution, and different letters are used to indicate significant differences between the samples.

Table 1. Tensile properties of the untreated and UV_3- and UV_8-treated fibres and matrix.

	Fibre properties		Matrix properties	
Samples	Tensile strength in MPa	Tensile modulus in GPa	Tensile strength in MPa	Tensile modulus in GPa
Un	583 ± 160	16.10 ± 1.65	25.20 ± 0.31	1.04 ± 0.07
UV_3	618 ± 128	15.63 ± 1.32	25.30 ± 0.53	1.05 ± 0.05
UV_8	535 ± 121	16.47 ± 1.18	25.80 ± 0.30	1.11 ± 0.14

0.257 mm, respectively, compared to the untreated sample, indicating increased fibre-matrix adhesion also depends on the irradiation dose. The interfacial shear strength (IFSS) improved by factors of 1.3 and 1.7 for UV_3 and UV_8, respectively, comparable to improvements (1.1–2.0) reported in studies involving fibre or matrix modifications [16,28]. For UV_3, the decrease in the critical fragment length is attributed solely to cross-linking, as tensile properties remain unchanged. However, in UV_8, the reduced fibre tensile strength (TS) suggests that the smaller fragment length may also result from changes in fibre strength (see section 3.5).

3.4. Interlaminar shear strength

The determined ILSS results are presented in Figure 8 and Table 2. UV irradiation significantly improved the ILSS in both UV_3 and UV_8 treated samples. The ILSS increased from 3.165 MPa in untreated samples to 3.985 MPa for UV_3, representing a 25.9% increase, and to 3.940 MPa for UV_8, showing an improvement of 24.5%. Earlier studies reported a 29% improvement in ILSS through UV-induced crosslinking monomers on RC fibres embedded in PP and a 140% increase in ILLS using maleic anhydride [43]. Significant ILSS increases of 72% and 129% were achieved through low radio frequency plasma treatment on jute fibres in jute/polyester [18]. Nevertheless, direct UV irradiation proves nearly as effective as UV-induced monomers on fibres. However,

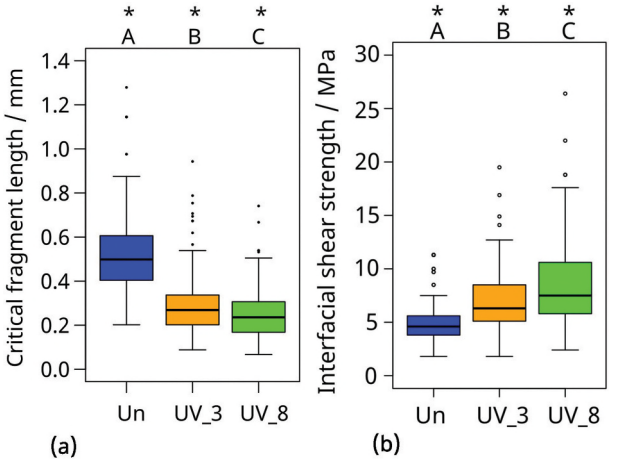

Figure 6. Box-whisker plots of the critical fragment length (a) and interfacial shear strength (b) of the RC fibres reinforced in the HDPE matrix measured with a single fibre fragmentation test. The untreated (un) sample is compared with the UV irradiated samples for 3 min and 8 min. An asterisk (*) represents the results that do not follow a normal distribution, and different letters are used to indicate significant differences between the samples.

Table 2. Mechanical properties of the untreated, UV_3 and UV_8 treated composites.

	Single fibre fragmentation test		Composite properties		
Samples	L_{fc} in mm	IFSS in MPa	Tensile strength in MPa	Tensile modulus in GPa	ILSS in MPa
Un	0.519 ± 0.20	5.20 ± 2.24	131 ± 0.56	1.86 ± 0.37	3.165 ± 0.47
UV_3	0.296 ± 0.14	6.90 ± 2.83	145 ± 2.65	2.28 ± 0.32	3.985 ± 0.11
UV_8	0.257 ± 0.11	8.40 ± 3.93	145 ± 3.60	2.87 ± 0.21	3.940 ± 0.19

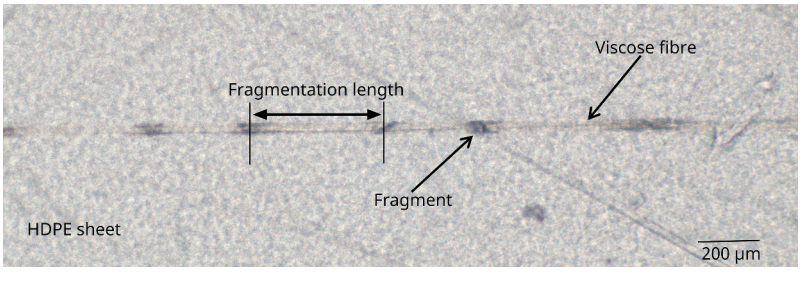

Figure 7. After the fragmentation test, a fragmented RC fibre in a polyethene matrix.

the ILSS improvement here is less pronounced than with pre-fibre irradiation. This discrepancy may arise from UV-induced moisture loss, creating a heterogeneous interface with voids, which weaken fibre-matrix shear resistance. However, this effect is negligible in single fibre fragmentation tests (SFFT) due to fewer fibres involved. Studies have reported void-related ILSS reductions of 11% for 0.5–1.0% void volume and up to 50% for 5% void volume [44]. Moreover, the fibre–fibre interaction could also

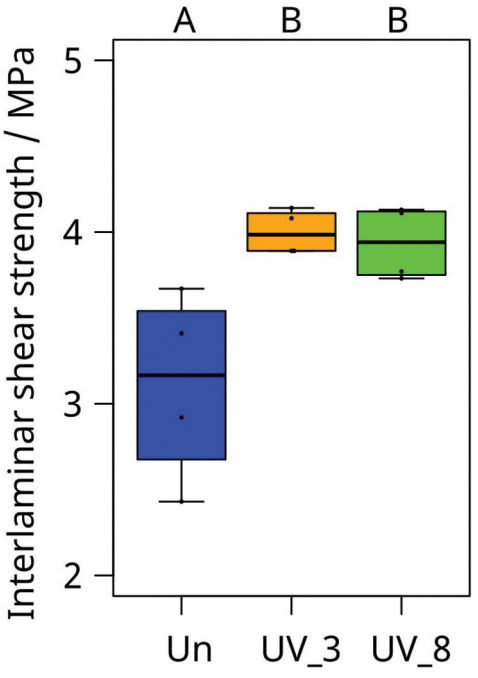

Figure 8. Box-whisker plots of the interlaminar shear properties of the RC fibres embedded in the high-density polyethene measured with the double-notch tensile test. A comparison is made between the untreated (un) and the UV irradiated samples, i.e., UV_3 and UV_8. An asterisk (*) represents the results that do not follow a normal distribution, and different letters are used to indicate significant differences between mean values.

be a factor due to higher fibre-volume ratios [38]. The fibre volume ratios for untreated and treated composites were 62.69% and 63.16%, respectively, indicating negligible differences. Porosity measurements, derived from density using mass and volume calculations, showed values of 8.75% for untreated composites and 10.34% and 5.28% for UV_3 and UV_8-treated samples, respectively. Despite the increased pore volume in UV_3, ILSS was unaffected, emphasising the dominant role of UV-induced crosslinking in improving fibre-matrix adhesion. In contrast, UV_8 samples, despite reduced pore volume, showed no further ILSS improvements, likely due to prolonged UV exposure creating nano-scale voids and a heterogeneous interface. These nano-voids, undetectable by density-based methods used here, are presumed to have a more significant effect on ILSS than micro-porosity. Further studies using advanced characterisation techniques are necessary to better understand pore volume, distribution, and their effects on composite performance.

3.5. Composite tensile properties

The stress–strain curves of untreated and UV-treated samples are presented in Figure 9, while the summary of the tensile properties is presented in Figure 10 and Table 2. The UV irradiation improved TS by approximately 10%, increasing from 131 MPa to 145 MPa for

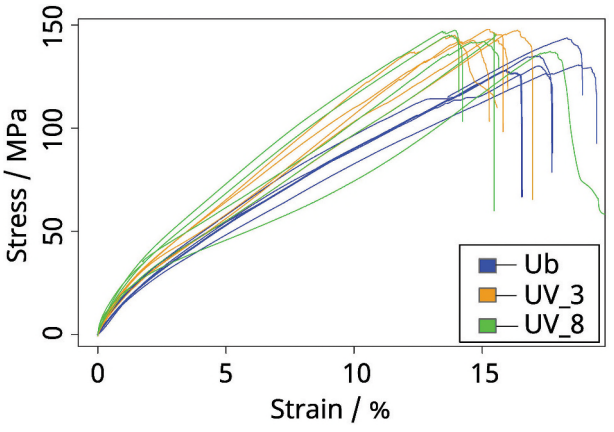

Figure 9. Stress-strain behaviour of the untreated (un) and the treated UV_3 & UV_8 composite samples manufactured with RC fibres and HDPE matrix.

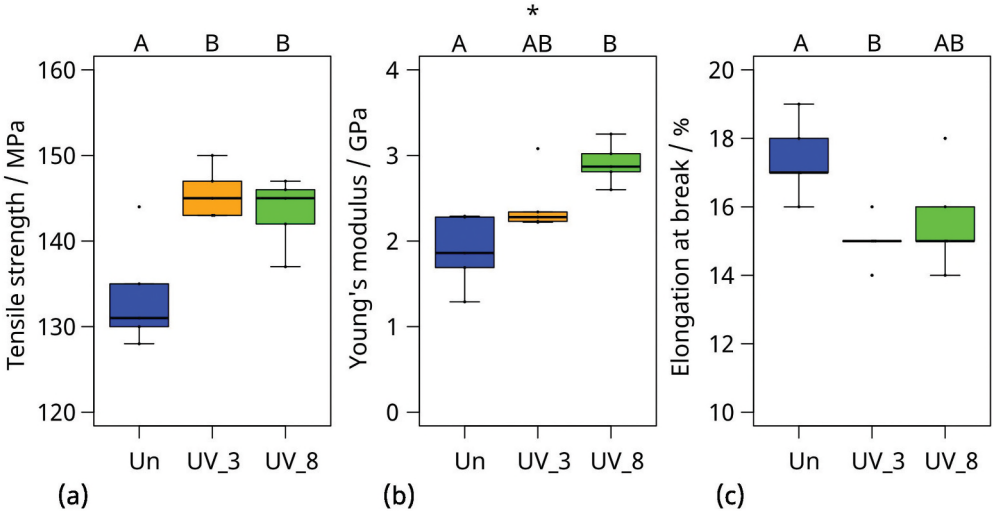

Figure 10. Box-whiskers plots of the tensile characteristics, i.e., tensile strength (a), elastic modulus (b) and elongation at break (c) of the untreated (un) and UV-treated (UV_3 & UV_8) RC fibres/HDPE composites. An asterisk (*) represents the results that do not follow a normal distribution, and different letters are used to indicate significant differences between the samples.

both UV_3 and UV_8 samples, but no further improvement was observed at UV_8. Similarly, gamma irradiation of jute/PP yielded a 12% increase in TS [19], and UV-induced monomers on RC fibres achieved a 20% increase in TS [43]. Pre-physical treatments on jute and henequen fibres showed a 30–62% increase in TS, which also depends on the treatment-induced change in fibre properties [18,45,46], and high reactive lignin on the jute fibre surface (absent in RC fibres). Graupner et al. [47] reported that lignin-modified cellulose fibres in TS of cellulose/PLA composites show a 10% increase in TS. Although the 10%

improvement in tensile strength (TS) may appear modest, it aligns with previously reported results. For example, Mina et al. [19] observed a 12% increase using gamma irradiation combined with chemical treatment of jute fibres. In our study, the observed TS enhancement is attributed to UV-induced cross-linking between fibre and matrix, and despite a reduction in fibre TS at UV_8, the TS of UV_8 treated composites surpassed that of untreated ones. Furthermore, the TS results of UV_8 treated composites indicate that the interfacial shear strength (IFSS) increase in single fibre fragmentation test (SFFT) samples stems from cross-linking rather than decreased fibre TS.

However, prolonged exposure (UV_8) results in a plateau in TS and ILSS; to understand this behaviour, SEM investigations were conducted to evaluate the fibre-matrix adhesion in untreated and UV-treated composites. Due to the fuzzy fracture observed on the tensile fracture surfaces caused by high fibre volume ratios, qualitative analysis was inconclusive. To address this, fibres were manually extracted from the polymer matrix, leaving behind a matrix skeleton for examination, as presented in Figure11. SEM images revealed elongated matrix detachments in UV-treated samples, which were absent in untreated samples, indicating improved fibre-matrix adhesion due to UV treatment. Furthermore, increasing the UV exposure time to 8 minutes resulted in more pronounced matrix detachments, suggesting that longer exposure enhances the adhesion further. These findings provide clear evidence of the positive effect of UV treatment on fibre-matrix adhesion.

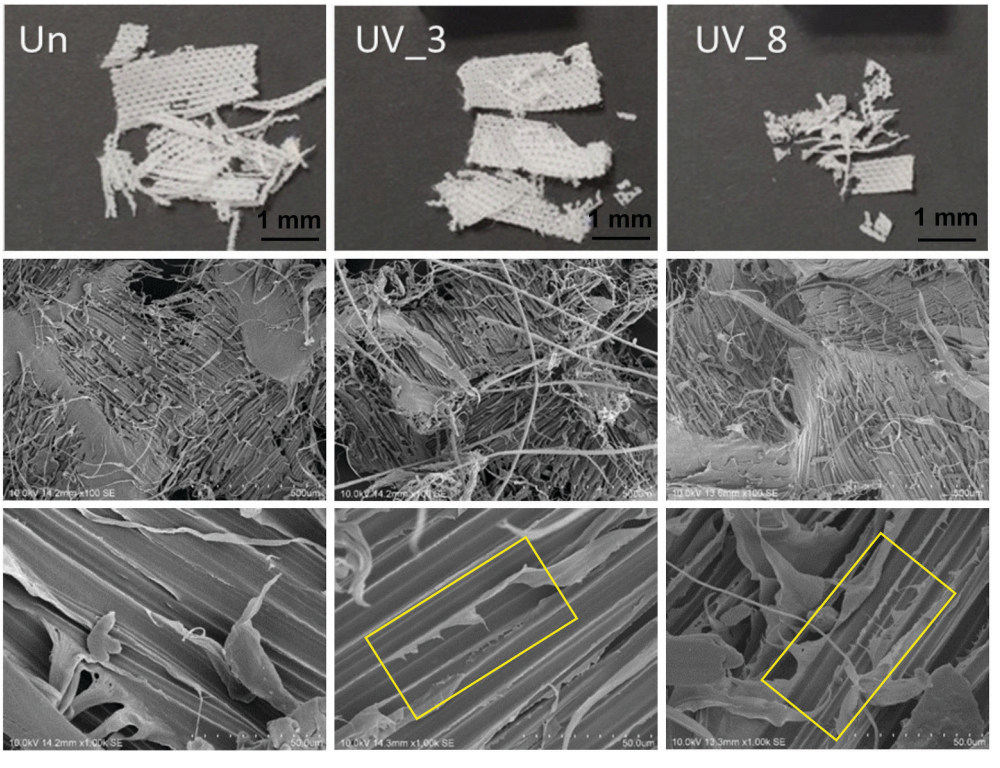

Figure 11. Photographs (top row) and SEM images of polymer matrix skeletons prepared after double-notch tensile tests and observation of matrix pull-out to be seen in UV_3 and UV_8 treated samples.

Despite the increase in the fibre detachment in the UV_8 treated composites and the observed increase in the IFSS from the SFFT results, the plateau in TS and ILSS is likely due to fibre degradation and the formation of a heterogeneous interface, which offsets additional gains despite the cross-linking effect. This assumption is supported by tensile tests of the semi-finished sheets after irradiation, and a second pressing process eliminated the inhomogeneity at the interface (results are not shown). Studies reported that 2% voids in composites resulted in an 8–15% drop in TS, while Young's modulus is less affected between 0-15% of voids [44,48].

Furthermore, the skeletons have been analysed spectroscopically to assess the presence of the fibre rests bound to the matrix. FTIR spectroscopy was employed to analyse the chemical structure and interfacial adhesion between RC fibres and the HDPE matrix under UV irradiation. The characteristic HDPE absorption peaks observed at 2914, 2847, 1470, 1350, and 718 cm^{-1} were consistent with previous studies [49,50], while UV-treated samples exhibited additional features such as O-H stretching around 3400 cm^{-1} and C-H stretching around 2900 cm^{-1} from viscose fibres. Figure A2 (in appendices) presents the FTIR spectra for untreated, UV_3, and UV_8 samples, highlighting UV-induced radical formation that leads to structural changes in both the fibres and the matrix. A spectrum between 800 cm^{-1} and 2000 cm^{-1}, as shown in Figure 12, highlights significant structural changes, likely caused by radical formation induced by UV treatment. These changes manifest to electronic excitations in both RC fibres and the HDPE matrix. In the 800–2000 cm^{-1} range, significant changes in C – C stretching at 960 cm^{-1} and C – O – C asymmetric stretching at 1155 cm^{-1} confirmed cross-linking between RC fibres and HDPE, as reported in the literature [10]. The increased absorption at 1155 cm^{-1} was indicative of the elongation of C – O –

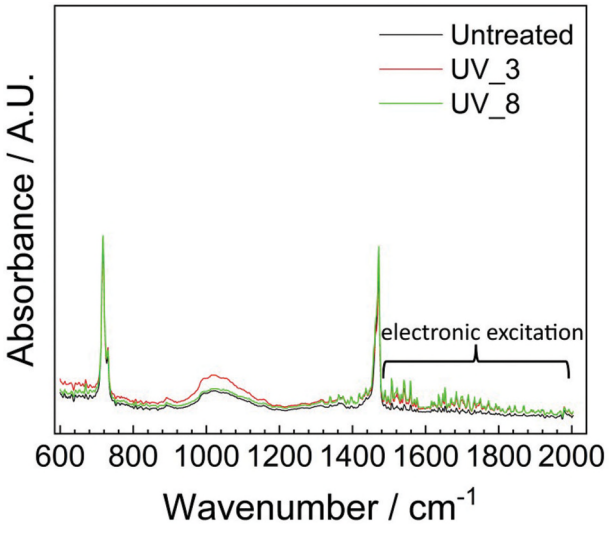

Figure 12. FTIR spectra of the matrix skeletons prepared from the untreated and UV-irradiated composites in the region from 800 to 1250 cm^{-1}.

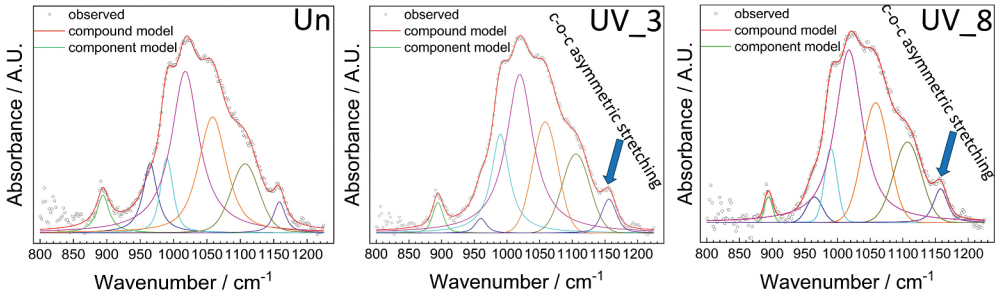

Figure 13. The fitted FTIR spectra with respective components of the untreated and irradiated composites for 3-min (UV_3) and 8-min (UV_8).

C bonds, a key indicator of crosslinking, while changes at 960 cm^{-1} suggested structural modifications in the HDPE matrix due to radicals formed during UV exposure. To further validate these findings, Figure 13 presents the FTIR fitting curves, distinguishing between the composite spectrum and individual molecular vibrations. The analysis highlights a distinct increase in the 1155 cm^{-1} peak, confirming the formation of chemical bonds facilitated by radical-induced crosslinking.

In UV_3-treated samples, the area under the 1155 cm^{-1} peak shows a pronounced increase, indicating significant crosslinking and improved fibre-matrix bonding. UV_8-treated samples also exhibit an increase in this peak compared to untreated composites, though smaller than UV_3, suggesting prolonged UV exposure promotes crosslinking but also induces chain scission, weakening the fibre structure. Optimal crosslinking in UV_3 results in superior interfacial adhesion and mechanical performance, while UV_8, despite improvements over untreated samples, demonstrates reduced fibre tensile strength due to excessive radical formation. Enhanced bonding in UV_3 and UV_8 samples, reflected in the 1155 cm^{-1} (C–O–C stretching) and 3350 cm^{-1} (O–H stretching) bands, aligns with mechanical test results, highlighting a balance between crosslinking and degradation in UV_8 with extended exposure.

Regarding the composite tensile modulus, the relatively low values for all composites, compared to the rule of mixtures (ROM), can be attributed to the high yarn undulation angle in the RC fabric (areal mass of 450 g/m^2). The TM of the samples with low undulation angle (areal mass of 220 g/m^2) is in line with ROM, which confirms that the reported low TM is related to textile structure. Results in Figure 10 indicate a 22.5% increase in TM, from 1.86 GPa to 2.28 GPa, with UV_3 treatment (not significant from statistical analysis), and a 54.3% increase, from 1.86 GPa to 2.87 GPa, with UV_8 treatment (significant from statistical analysis). Generally, fibre–matrix adhesion has a negligible effect on the composite TM; rather, it is influenced by changes in the fibre and matrix properties [24]. In this study, prolonged UV_8 irradiation slightly increased the Young's modulus of the fibre and HDPE polymer. This effect seems significant when the PE matrix is irradiated in the presence of RC fibres, which ultimately enhances the bulk composite Young's modulus.

4. Conclusion

This study highlights the potential of UV radiation as a promising method to enhance fibre-matrix adhesion in regenerated cellulose fibre (RC) and high-density polyethylene (HDPE) composites, eliminating the need for coupling agents. UV irradiation of semi-finished composite sheets significantly improved their mechanical performance by facilitating cross-links between the UV-absorbing RC fibres and the UV-transparent HDPE matrix. This enhancement in adhesion was evidenced by a reduction in the critical fragmentation length, as well as improved tensile strength and interlaminar shear strength, indicating better load transfer between the fibres and matrix. These improvements were confirmed through FTIR spectroscopy, which showed changes in the chemical structure of the fibres and matrix, particularly in the C-O-C bond region. However, prolonged UV exposure (8 minutes) caused some fibre degradation, reducing tensile strength, which underscores the need to optimise irradiation times. Future research should focus on the long-term durability of UV-treated composites, particularly under varying environmental conditions like humidity and UV exposure. Overall, the findings suggest that UV irradiation could transform the fabrication of sustainable composites, reducing reliance on coupling agents and contributing to greener, more efficient manufacturing processes.

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Disclosure statement

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Appendix

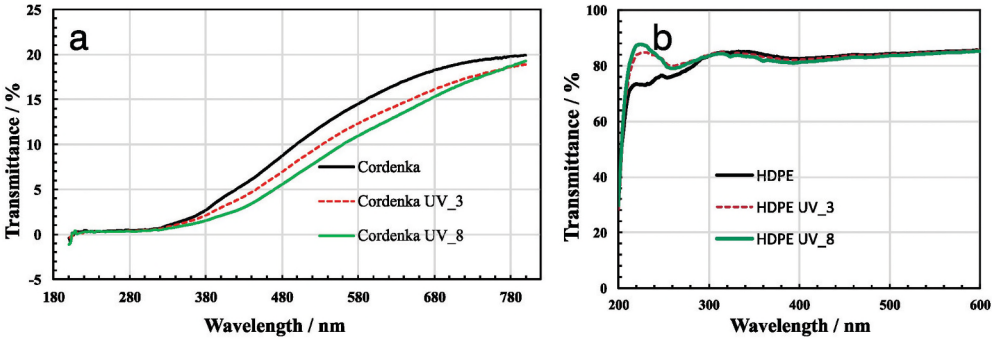

Figure A1. Transmission spectra of the RC fabric and HDPE sheets, untreated and irradiated for 3 min and 8 min; (a) fibres and (b) polymer.

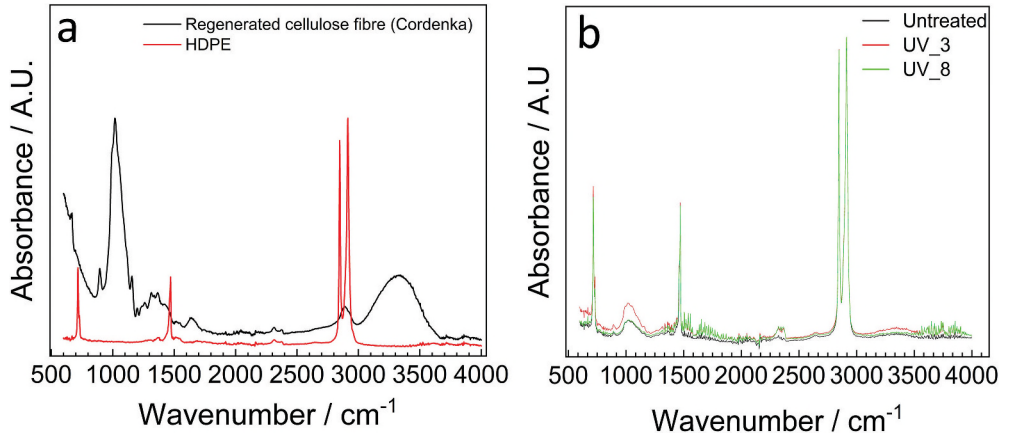

Figure A2. FTIR spectra of HDPE and RC fibre (a), along with the FTIR spectra of untreated, UV_3, and UV_8 treated composite samples in the range of 600 cm^{-1} to 4000 cm^{-1} (b).