

Environmentally Resistant Flax Fiber-Reinforced Composites for Aircraft Applications: Aviation Stress Tests with Optical and Mechanical Analyses

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Abstract

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Keywords Flax fibers · Mechanical properties · Coatings · Environmental conditions · Aviation

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1 Introduction

In aviation, every additional gram of basic weight results in increased fuel consumption for a given flight distance [1], which is why carbon fiber-reinforced composites (CFRCs) are very attractive for this area of application given their superior specific strength [2, 3]. However, natural fiber-reinforced composites (NFRCs) which utilize plant fibers for mechanical reinforcement are also used for lightweight design, as they exhibit excellent sound-absorbing and vibrational damping properties [4, 5] and thus ensure passenger comfort and health during transport. Although they do not come close to the outstanding mechanical properties of CFRCs [6–9], flax fiber-reinforced composites (FFRCs) as prominent examples of NFRC are cheaper [3] and have very good mechanical properties comparable to glass fiber-reinforced composites (GFRCs) [10]. The specific stiffness of flax fiber-reinforced composites is especially excellent. Therefore, due to their mechanical properties and renewable origin FFRC exhibit a better CO₂ footprint for stiffness-related automotive applications compared to GFRC [11, 12], making them suitable for sustainable lightweight construction. NFRCs are currently used in automotive interior trim [13]. Additionally, in 2022 the company BMW presented its M4 GT4 G82 race car in which they used NFRCs even in exterior parts like doors and tailgate. As these materials become more popular in engineering, hybrid structures of flax fibers and glass or carbon fibers are being investigated to determine possible synergistic effects [8, 14]. NFRC materials are also finding their way into application in aerospace. For instance, the airline Lufthansa and the company Bcomp have jointly developed the commercially available product system AeroFLAX, which is based on flax fiber-reinforced composites and allows to replace GFRC interior panels by saving weight. As the example shows, interior applications in particular are currently being identified as attractive substitution candidates in the aviation sector. Nevertheless, just like in the automotive sector, an expansion of possible applications to semi-structural and structural exterior parts is not merely theoretical.

To extend their area of application in the aviation sector, NFRCs must fulfill special requirements. For example, the RTCA/DO160G standard describes which liquids airborne equipment or materials for the protection of airborne equipment must withstand [15]. In Sect. 11 of the document, jet fuel and hydraulic fluids are named as critical fluids whose influence on the material should be determined in immersion tests, when it is likely that a contamination of the materials with the specific fluid occurs. In the European norm EN 2379:2018 hydraulic oil and jet fuel are specified as test liquids for evaluating nonmetal materials for aerospace applications as well. For the commonly used CFRC materials, their property changes due to exposure towards jet fuel and hydraulic oil have already been tested [16–19]. Flax fiber-reinforced composites as relatively new materials for light weight design have not yet been sufficiently investigated in this regard, especially as they are usually favored more for interior applications. To broaden the possible range of application to semi-structural and structural components, NFRCs need to withstand jet fuel and hydraulic oil. To the authors knowledge, the only study on this topic was done by Krzyżak et al. [20]. They investigated the tensile properties of flax, cotton and jute fiber-reinforced composites after 48 h immersion in water, oil and jet fuel among others. However, an assessment of the optical properties, longer immersion times as well as of the effectiveness of protective measures like coatings has not been performed yet and is provided by this study. In addition, a comparison of the influence of hydraulic oil and jet fuel with standard weathering

procedures that ensure reproducibility is still pending. For weathering tests, defined conditions of UV irradiation, temperature and water spraying or condensation simulate a natural environment. Several studies already focused on the deteriorating effects of weathering on FFRCs [21–24]. Most studies include alternating UV irradiation and water spraying cycles in the climate chamber. In the study by Vellguth et al. [23] direct water spraying and UV light were replaced by temperature-controlled ageing cycles with a high humidity of up to 93%. In contrast to hydraulic oil and jet fuel, it is well known that the penetration of water into the matrix of NFRC damages the material. The hydrophilic natural fibers absorb the water, causing swelling and deformation of the composite which leads to the deterioration of mechanical properties like flexural strength and modulus [25–27]. This effect was observed in direct water immersion [25, 27] but also in warm, humid conditions (55 °C / 85% humidity) [26]. Water also activates the biodegradation of the fibers, causing them to lose strength [28]. Water is also included in the list of test fluids. Therefore, this study allows comparing the severity of the damage caused by hydraulic oil, jet fuel, water and standard weathering conditions defined in the EN ISO 16474-3 norm.

To protect structural parts from environmental influences, typically coatings are applied to the load-bearing structure. However, these are often fossil-based, so there is potential for saving finite resources by using (partially) biobased coatings [29]. Partially biobased alternatives e.g. on polyurethane basis show effective protection of NFRCs from moisture uptake [30], but commercially available (partially) biobased aircraft coatings are still rare. However, the development of new biobased high-performance coatings is a hot topic in the industry. To be able to classify the protective efficiency of coatings - biobased and petrochemical - against the degradation of FFRCs, a commercially available epoxy/polyurethane based aircraft coating system is applied to NFRC and exposed to environmental influences. Secondly, a (partially) biobased polyurethane automotive interior coating is used to demonstrate the protective effect of a state-of-the-art (partially) biobased high-performance coating. (Partially) biobased aviation coatings are currently still uncommon. Therefore, an automotive coating was used to assess the potential of partially bio-based high-performance coatings for application under aviation conditions. Both coatings are compared in their ability to protect the NFRC and in their robustness towards environmental influences.

Higher fiber contents can contribute to improved mechanical properties of FFRCs as long as the fibers are impregnated by the matrix to ensure force transfer [31, 32]. Therefore, a relatively high fiber content is beneficial for optimal mechanical properties. The effect of an increased fiber content on the composite's resistance to aviation environments is therefore of interest for the application. Hence, two uncoated reference materials with different fiber contents are investigated.

This study closes important knowledge gaps and evaluates new biobased and more sustainable alternatives (flax fiber-reinforcement, partially biobased coating) in regard to their environmental performance compared to the fossil status quo to be able to understand their suitability for use in aircraft.

2 Materials and Experimental

Figure 1 provides an overview of the materials used in this study and the experiments and analysis methods applied. These are described in detail in the following subchapters.

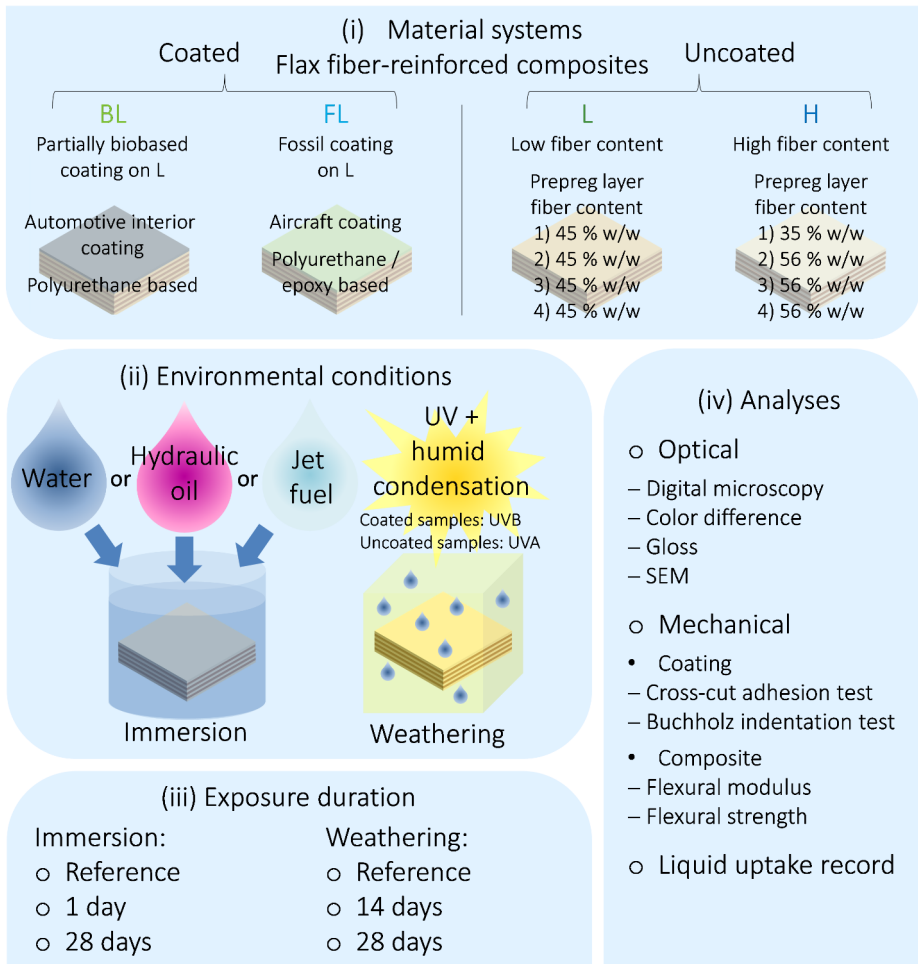


Fig. 1 Overview of materials, type and duration of applied environmental conditioning and analytic methods

2.1 Materials Systems

2.1.1 Fabrication of the Composite Plates

Prepregs from Fiberpreg GmbH were used to fabricate the flax fiber-reinforced composites. The flax fibers were prepared as 2/2 twill weave fabrics and came pre-laminated from the manufacturer. These were processed in an autoclave under vacuum following the manufacturer's recommended procedure. Marbocote[®] 625X Eco was used as a release agent for all samples. Plates measuring 400 mm x 350 mm and 260 mm x 440 mm were produced. For the samples with the lower fiber content, four layers of Fiberpreg N 200 T EP 4.5 S bio 55 100 with a resin content of 55% w/w were layered and cured in a vacuum bag under -0.9 bar vacuum and 7 bar in an autoclave. The heating rate was 2 °C/min and the plates

were cured for 60 min at 120 °C. For the samples with the higher fiber content, Fiberpreg NAT-200-T-EP1.5 44 100 with 44% w/w resin content and Fiberpreg NAT 200 T EP1.5 65 100 with 65% w/w resin content were used and layered. First, one layer of NAT 200 T EP1.5 65 100 and then three layers of NAT 200 T EP1.5 44 100 were stacked on top of each other to increase fiber content and still aim at a good surface finish. These were cured in an autoclave molding process under -0.9 bar vacuum in an autoclave at 7 bar. This took place at a heating rate of 2 °C/min and finally holding a plateau at 120 °C for a period of 90 min. The manufacturing process resulted in one flat side of the composite. The prepared composite plates had a final thickness of 2 ± 0.1 mm.

2.1.2 Coating of Prepared Composite Plates

The fossil-based aviation system from Akzo Nobel N.V. consists of three layers: “Alumi-grip 10P30-8 Sanding Surfer” with the hardener “Curing Solution EC-284”, the primer “Aerowave 2002” with the hardener “Curing Solution 6005”. These two layers are epoxy resin-based. The top coat is a green polyurethane coating “Aerowave 5001” with the hardener “Curing Solution 6002”. The layer thickness varies due to the unevenness of the one side and the manual application of the paint. The coatings were applied to the composite panels using compressed air spraying.

The partially biobased polyurethane coating system is an automotive interior coating from Weilburger Coatings GmbH. It consists of a primer with the label “senocar-2K-ecoHydroLaserprimer black 26-5350-116877E” and a top coat with the label “senocar-2K-Hydro-ecoHaptiklack Soul 6PS 05-5091-118214”. Both coatings were processed with the hardener “19-5090-118213” according to the manufacturer’s instructions and sprayed onto the FFRC plates using compressed air. The layer thickness varies due to the unevenness of the non-flat side and the manual application of the paint.

Both coating systems were applied to the composite plates with the lower fiber content and subjected to further tests. The uncoated samples with higher and lower flax fiber content were also examined and served as references. This resulted in four material systems with following sample codes: Partially biobased coating on FFRC with lower fiber content (BL), fossil-based coating on FFRC with lower fiber content (FL) and the uncoated flax fiber-reinforced composites with lower fiber content (L) or higher fiber content (H).

2.2 Environmental Conditioning

2.2.1 Immersion Tests

For exposure in aviation fluids, the coatings were applied to both sides of the composite plates. These and the uncoated samples were then sawn into 125 mm x 85 mm specimens. The thickness remained approx. 2 mm.

Following the European standard EN 2489:1995 recommendations, the sample plates were stored separately in covered glass containers, completely immersed in water, jet fuel and hydraulic oil. The samples were placed upright in laboratory holders so that the liquid could reach the samples from all sides. Distilled water, Jet A-1 from BP and “AeroShell Fluid 41” mineral hydraulic oil from Shell were used as test liquids. The samples were stored at room temperature. For the analyses, reference samples without liquid contact and

Table 1 Irradiation based on EN ISO 16474-3 (Cycle 1). This cycle was used for the uncoated samples L and H

Duration	Type of lamp	Irradiance / wavelength	Black panel temperature
4 h	UVA-340	0,83 W/(m ² ·nm) / 340 nm	(60±3) °C
4 h	Condensation	UV lamps off	(50±3) °C

Table 2 Irradiation based on EN ISO 16474-3 (Cycle 4). This cycle was used for the coated samples BL and FL

Duration	Type of lamp	Irradiance / wavelength	Black panel temperature
4 h	UVB-313	0,71 W/(m ² ·nm) / 310 nm	(60±3) °C
4 h	Condensation	UV lamps off	(50±3) °C

samples that had been immersed for 1 d and 28 d were examined. The edges have not been sealed. Therefore, based on preliminary tests, the area of the composite plates into which the liquid could penetrate from the edge was removed after immersion so that the values of the following analyses were not falsified.

2.2.2 Weathering Tests

For irradiation with UV light, only the smooth side of the composites was coated and the samples were then sawn into 110 mm x 75 mm specimens with the thickness remaining approx. 2 mm. These were tested in a climate chamber (QUV Spray from Q-Lab) based on the European standard EN ISO 16474-3. No water was sprayed in the climate chamber, the effect of UV radiation and water condensation from the air flow was examined. The uncoated samples L and H were irradiated with UVA radiation as described in Table 1. The coated samples BL and FL were exposed to the smaller wavelength UVB radiation as shown in Table 2. The cycles were performed for two weeks and 4 weeks respectively. The irradiated samples were then compared with the non-weathered reference samples.

2.3 Analyses

The micrographs were taken with a Keyence VHX 5000 digital microscope. As required by the EN 2489:1995 standard for the examination of fiber-reinforced composites, gloss and color measurements are performed on the samples that were immersed in aviation liquids. For comparison, this is also done on the samples irradiated with UV light. The “spectroguide 45/0 gloss” color and gloss meter from BYK-Gardner GmbH was used for this purpose. The norm also specifies that the liquid uptake of the samples should be recorded. The samples were wiped dry for the weight measurements. Standard tests were performed on the coatings to determine their quality. These are cross-cut adhesion tests in accordance with EN ISO 2409:2020 and measurements of Buchholz hardness in accordance with EN ISO 2815:2003. The cross-cut and Buchholz indentation tests were done using the byko-cut device from BYK-Gardner GmbH. However, the digital microscope VHX 5000 from Keyence was used instead of the built-in microscope to measure the indentation length. The evaluation of the cross-cut tests can be found in the supplementary information (Fig. S1). As the flexural properties are very important for the most attractive field of application, the flexural modulus and flexural strength of the samples were determined using 4-point flexural

tests in accordance with EN ISO 14,125. For this purpose, the specimens were cut out of the plates used, measuring 40 mm x 14 mm x approx. 2 mm. The samples were taken from the center of the sample plates. Scanning electron microscopy (SEM) images of the coated samples were created with Auriga 40 from ZEISS and the SEM images of the uncoated samples with Supra 55 VP from ZEISS.

3 Results and Discussion

3.1 Surface Quality and Color

Figure 2 shows light microscope images of all the immersed and irradiated samples after the respective exposure durations. The pictures show that the sample surfaces do not exhibit any large cracks. For the coated samples (BL and FL), there is no difference in surface appearance in any of the parameters in a superficial examination. This also applies to uncoated samples stored in jet fuel and hydraulic oil. In comparison, uncoated UV-weathered samples and water exposed samples show significant differences in surface appearance. The color of the fibers of the uncoated UV-irradiated samples fades over time. It changes from a medium brown to a light beige. For the samples with higher and lower fiber content (H and L), the color faded similarly over the 28-day period. The fading is probably due to the photooxidation of lignin [33]. The lignin component is primarily responsible for the color of flax, although extracts can also influence the color. In contrast to hemicellulose and cellulose, lignin absorbs UV light very well due to its chromophores [34]. Sunlight causes

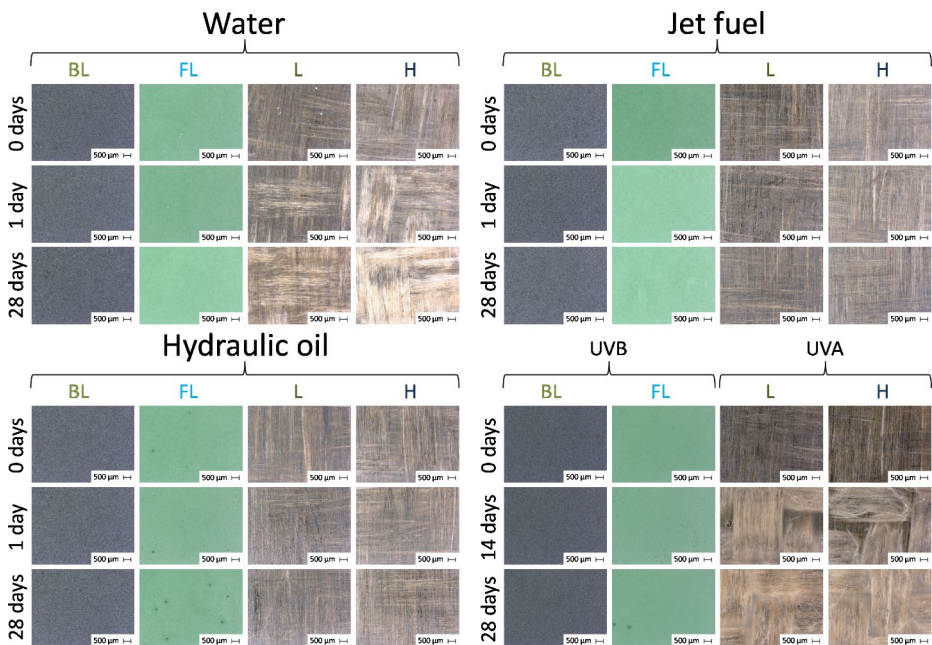


Fig. 2 Digital light microscopy of uncoated and coated samples exposed to water, jet fuel and hydraulic oil, and weathered with UV light depending on different exposure times. UVA light was used for the uncoated samples and UVB light for the coated samples

photooxidation of the lignin and scission of the chemical structure with the formation of phenoxy radicals. With further oxidation, these lead to the formation of quinone structures, new chromophoric groups, which greatly alter the optical impression [35–37]. Due to the degradation of the lignin, the fibers therefore appear light yellow over time. The fact that the color change is more pronounced with L than with H is unintuitive, as the fibers should be better protected with L due to the higher matrix content. A possible reason could be production deviations and variation of the thickness of the protective epoxy resin layer.

After just one day of exposure to water, individual fibers began to lighten. The water already penetrated these areas. After 28 days, the lightening increased as the water continued to penetrate. The reason for this is probably that due to water penetration the connection between the matrix and the fibers is disrupted. The water can diffuse through the matrix and penetrate through microcracks. The hydrophilic natural fibers absorb the water and swell resulting in further cracks that accelerate the process. In addition, the water is transported further along the fibers by the capillary effect. The swelling and release of soluble substances from the natural fibers by the water results in delamination between the fibers and the matrix [38, 39]. Before lamination in resin, the fibers looked lighter. Due to the occurring delamination the color impression probably changes back.

This effect is stronger for the higher fiber content than for the lower fiber content ($H > L$). The reason for this is further discussed in Chap. 3.4.

Figure 3 shows the quantified color difference of the sample set-up. As already observed, there are hardly any color differences for immersion in jet fuel and hydraulic oil, and the coated samples stay very similar in color. One exception is found for the fossil coating system under UV radiation where a slight color change of $3 \Delta E^*$ can be observed at 28 days exposure. The differences in the uncoated samples due to UV irradiation and water immersion are much greater. The uncoated weathered sample reaches the maximum observed color difference of $29.5 \Delta E^*$ already at 14 days exposure. The reason for the color changes has been discussed in the first section of Chap. 3.1.

3.2 Gloss

Figure 4 shows the change in gloss due to exposure to UV weathering and immersion in aviation fluids. It can be seen that the untreated FFRC achieves higher gloss values than the coated samples. This is due to the fact that both coatings aim at a matt finish. The gloss values of the references of the untreated FFRC vary widely in some cases, because the surface does not achieve the same finish everywhere due to production deviations. This is particularly evident in the references for hydraulic oil, which have a higher gloss. It is interesting to note that the gloss of the samples does not change significantly over the immersion duration in water, jet fuel and hydraulic oil. This is an indication that none of the fluids cause any major chemical changes to the epoxy resin topcoat, maintaining the original surface quality of the samples. It should be emphasized that immersion in water leads to a significant change in color of the NFRC probably due to delamination (see Fig. 3). The gloss, on the other hand, is not affected by the water, as it seems not to severely chemically alter the epoxy resin matrix, which is responsible for the gloss. This is in stark contrast to the weathered sample, which is not only brightened (color change), but also loses much of its gloss. After just 2 weeks of cycling, the gloss value dropped by approx. 60%. Loss in gloss indicates a roughening of the surface. Ghasemi-Kahrizangi et al. [40] showed that the surface of an epoxy resin coating

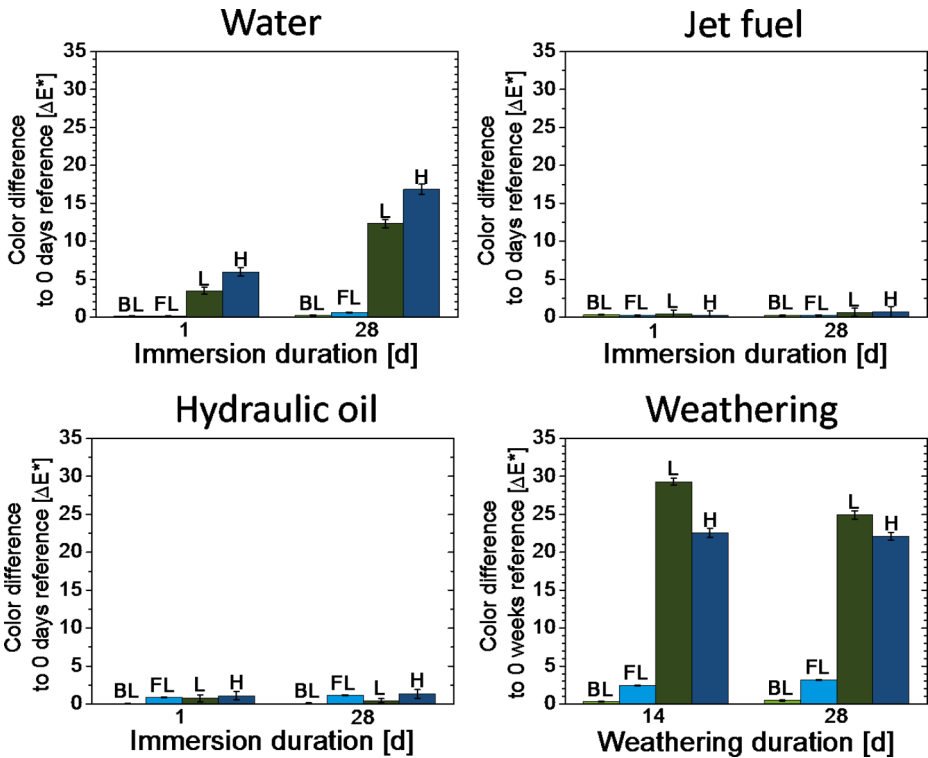


Fig. 3 Color difference ΔE^* of uncoated and coated samples exposed to water, jet fuel and hydraulic oil, and UV weathered in regard to pristine samples (0 d exposure)

was riddled with microcracks after long-term UV irradiation (1000 h) which is associated with roughening of the surface. They attribute this behavior to photooxidation, the resulting scission of the polymer chains and the formation of radicals. Similarly to the lignin of the flax fibers, epoxy resin is particularly susceptible to UV light due to its aromatic rings, as these absorb UV light particularly well. The generated radicals can react with the polymer chains and cross-link them further. This could be shown in FT-IR measurements by Peterson et al. [41] The bands indicating the typical benzene rings and C-O compounds are reduced, but C=C and C=O bands are added. This additional cross-linking embrittles the material, which favors the formation of microcracks. The cracking itself leads to an increase in the surface area and accelerated photooxidation. The roughening can also be associated with material removal [42]. As the UV light causes the breaking of the epoxy resin chains on the surface, the components of the epoxy resin volatilize over time due to the breaking of the bonds and the energy supplied by the UV light [43]. Subsequently, there is also erosion of the epoxy resin surface i.e. a loss of mass [41–44]. As described in the literature, it was also observed in this study that the surface of the weathered samples was roughened. After four weeks the epoxy resin was partially removed and the fibers were exposed. This led to the severe loss of gloss. No difference in the loss of gloss was found between H and L. The gloss is determined by the surface structure. Therefore, a thin layer of epoxy resin is sufficient to produce a glossy finish. Although the SEM images (Fig. 7g-h) showed that the surface of

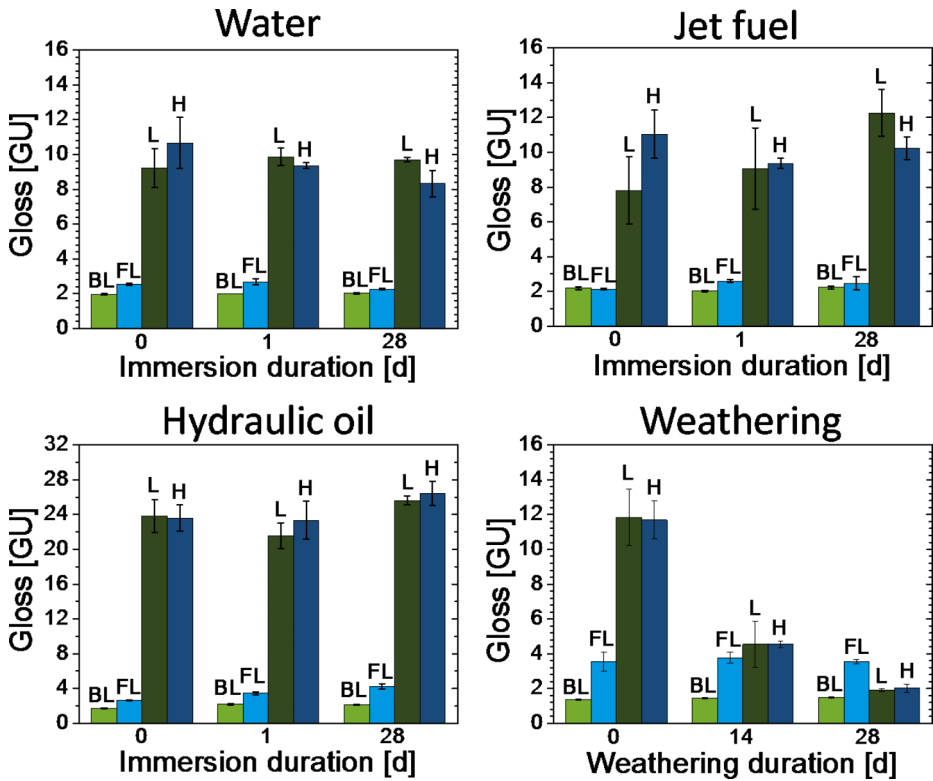


Fig. 4 Gloss of uncoated and coated samples exposed to water, jet fuel and hydraulic oil, and weathered with UV light

the FFRC with the lower matrix content H is not completely closed in some areas, the epoxy resin layer is thick enough to result in a glossy surface that does not differ significantly from the sample with the higher matrix content L. The loss of gloss over the irradiation time is also similar for H and L. This is due to the fact that only the surface is responsible for the gloss which is roughened by photooxidation regardless of the respective total matrix content in the composite.

3.3 Coating Adhesion and Hardness

To investigate the effects of weathering and exposure to aviation fluids on the quality of the coatings, cross-cut adhesion tests were performed and assessed according to the evaluation scheme. The results can be found in the supplementary information (Fig. S1). It was found that the coating adhesion of both coats was almost completely unaffected by exposure and weathering. Only the exposure to weathering conditions led to a decrease of the layer adhesion of the fossil aircraft coating to the FFRC substrate. The partially biobased alternative still performed excellently after four weeks and is very well suited as a coating system for FFRC materials under the environmental influences tested. To detect a possible chemical change, Buchholz hardness tests were performed as well. The results can be seen in Fig. 5.

The shorter the indentation length, the higher is the hardness. It was found that the partially biobased coating and the aircraft coating system achieve very similar values within the scope of the measurement accuracy. The reference samples were tested in an analysis of variance and no significant difference was found for the Buchholz indentation length of BL and FL based on $p < 0.05$. No softening or hardening could be detected over the duration of exposure under any of the aviation conditions, which is a further indication of the suitability of the coatings as protective layers against the given conditions.

3.4 Liquid Uptake

As already described in Chap. 3.1, the color fading of the samples immersed in water is probably triggered by the penetration of the water into the matrix. Figure 6 shows how the sample mass increases over the immersion time. It can be seen that up to 16% w/w water is absorbed resulting in a saturation profile. On the contrary, almost no jet fuel and hydraulic oil is accumulated in the samples. A comparison shows that significantly more water was absorbed by the FFRC with the higher fiber content than by the FFRC with the lower fiber content. This is due to the hydrophilic nature of the natural fibers. Flax fibers consist of 80–90% cellulose and hemicellulose [45, 46]. The biopolymer chains have a large number of hydrophilic hydroxyl groups so they can adsorb a lot of water and the entire fiber swells as a result. This leads to microcracks as well as delamination which can promote the capillary

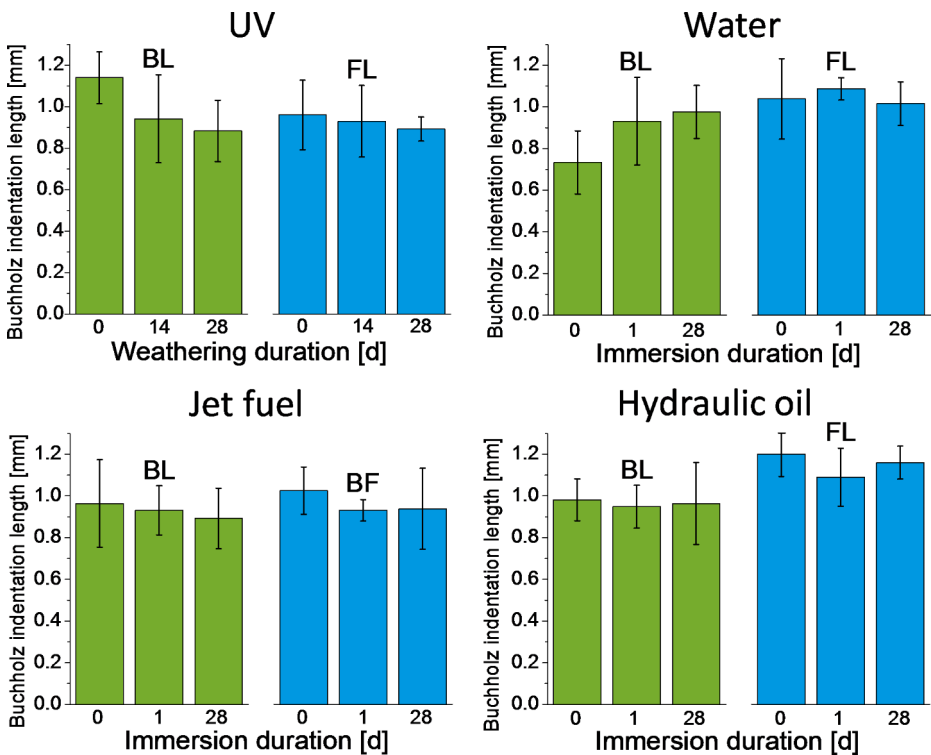


Fig. 5 Buchholz indentation length for coated samples that were immersed in water, jet fuel or hydraulic oil or weathered

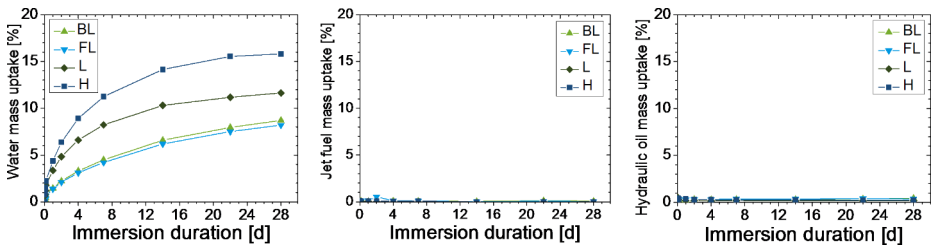


Fig. 6 Liquid uptake of samples stored in water, jet fuel or hydraulic oil over the immersion time

effect. These factors can accelerate water penetration [38, 39]. Therefore, an increased water absorption rate is measured for H than for L. It can also be seen that the water absorption of the uncoated samples seems to approach a plateau, but has not but has not fully reached it after 28 days of immersion. The uptake of water until a plateau is reached is typical for FFRCs, as shown by Chilali et al. [47] The approached plateau is shifted to higher values for the sample with the higher fiber content. It is very likely that it is not only a matter of water absorption rate, but an actual difference in water absorption capacity due to the larger fiber/matrix ratio as the epoxy resin matrix itself hardly absorbs any water [48]. Muñoz et al. also observed that water uptake was increased with higher fiber content [49]. Another factor is discussed in Chap. 3.5. Water absorption is significantly delayed by the two coatings tested. The protective effect of coatings on FFRCs in contact with water is known and has already been demonstrated, e.g. by Mokhothu et al. [30]. It is interesting to note that both the bio-based coating (BL) and the fossil-based coating (FL) show a very similar retarding effect on water absorption, although the fossil-based coating is significantly thicker and shows a more advantageous surface morphology (see Fig. 7).

Although cellulose is a hydrophilic biopolymer, it is insoluble in water. Therefore, Lindman et al. [50] postulated that hydrophilic hydrogen bonds are not the only crucial interactions that determine the processes between the biopolymer and solvents. Lindman et al. indicated that hydrophobic interactions are also important to understand cellulose chemistry and that cellulose has significant amphiphilic properties. Based on this, hydraulic oil and jet fuel as apolar hydrocarbons are interesting candidates to study their interaction with the cellulose-based flax fibers. In our experiments, it became clear that flax fibers hardly absorb hydraulic oil and jet fuel. It could still be possible that the liquids penetrate the composite through microcracks and other defects. However, there is no significant liquid absorption that could lead to swelling of the fibers, which means that the damaging mechanism described in Chap. 3.1. is probably not active. Nevertheless, damage by other mechanisms like chemical reactions cannot be excluded. This possibility is examined later in Chap. 3.6 when testing the mechanical properties.

3.5 Surface Structure of Coatings and Uncoated Composites

Figure 7a-f shows micrographs and SEM images of the coated samples as well as the schematic structure of the applied coating. The light microscopic cross-sectional images (Fig. 7c and f) show that the aviation coating system adds up to a thicker structure (approx. 220 μm) than the partially biobased automotive interior coating system (70 μm) due to the three layers. The SEM images also show that although both coatings are matt systems, the par-

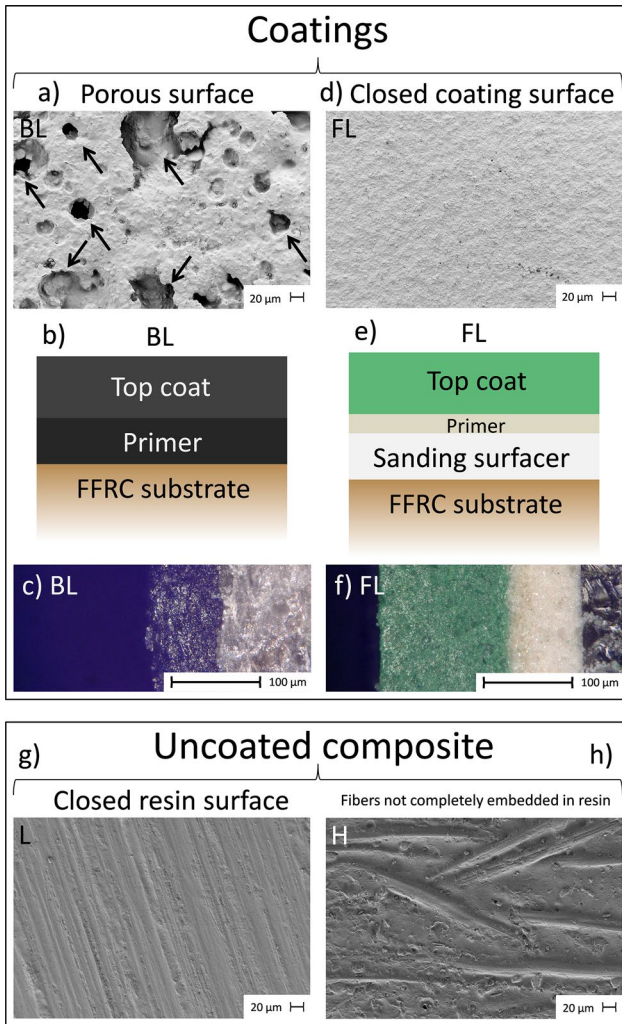


Fig. 7 a) SEM image of the surface of the partially biobased automotive interior coating on FFRC substrate (BL). The surface is permeated by many pores (indicated by black arrows). b) Scheme of the BL setup. The scheme illustrates the stacking. The layers are not shown in the correct thickness ratio. c) Micrograph of the BL cross-section showing the coating on FFRC substrate. The two coating layers are indistinguishable as they are both very dark. d) SEM image of the surface of the fossil aircraft coating (FL). e) Scheme of the FL setup. The scheme illustrates the stacking. The layers are not shown in the correct thickness ratio. f) Micrograph of the FL cross-section showing the coating on FFRC substrate. g) SEM image of the uncoated FFRC surface with the lower fiber content (L). h) SEM image of the uncoated FFRC surface with the higher fiber content (H)

tially biobased coating has a more porous, pitted and uneven surface (Fig. 7a). In contrast, the surface of the fossil aviation system is free of pores (Fig. 7d). Due to the lower coating thickness and more porous surface, one would expect the partially biobased system to absorb considerably more water. As can be seen in Fig. 7, there is only a slight difference,

so that even the lower coating thickness can be classified as good protection against water penetration.

Figure 7g-h shows SEM images of the surfaces of the uncoated samples. In the sample with the higher fiber content (H), the fibers are also visible on the surface. They are not completely laminated in resin even though a surface prepreg layer with a reduced fiber content was used during processing. In contrast, the surface of the sample with the lower fiber content (L) is completely laminated in epoxy resin. This difference may be an additional reason for the higher water absorption by the sample with the higher fiber content (Fig. 6a), because the water can reach the surface fibers easily and use them to penetrate into the composite. This mechanism could be particularly effective at the beginning, when the composite does not yet exhibit swelling-related microcracks through which the water can penetrate.

3.6 Flexural Properties

Figure 8 shows the flexural modulus of the immersed and irradiated samples. The references for L and BL are not significantly different based on an analysis of variance with $p < 0.05$. However, they both diverge significantly from the FL references which show a decreased flexural strength, although the same prepreg system was used for all of them. The coating layer is thick enough to have a significant influence on the thickness of the samples. Since the flexural modulus is normalized to the cross-section of the sample and the coating itself is less stiff than the composite substrate, this leads to lower flexural moduli. However, the

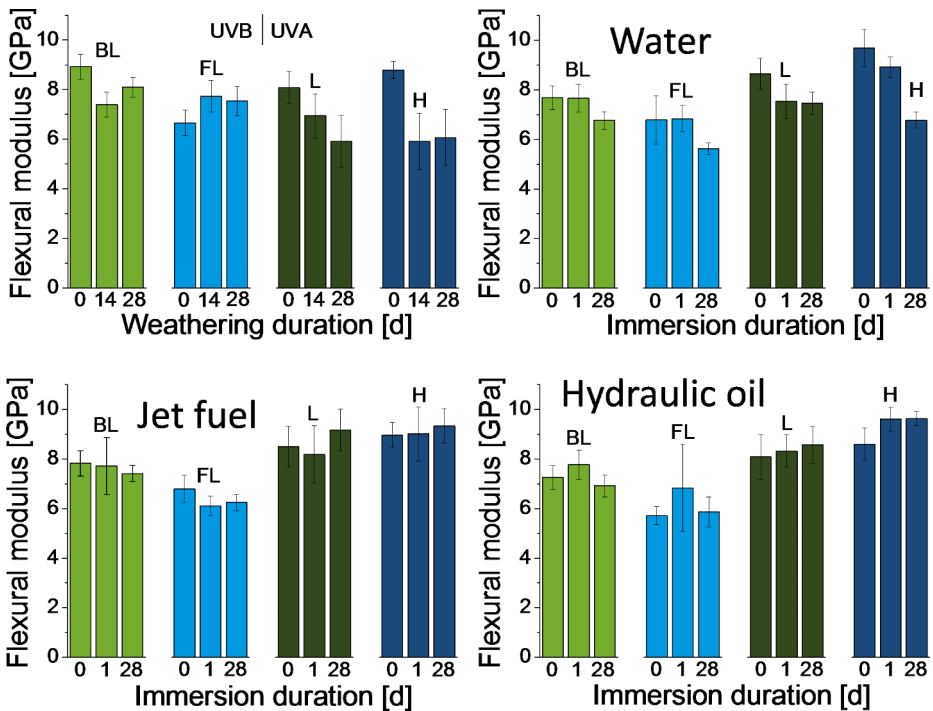


Fig. 8 Flexural modulus of uncoated (L and H) and coated (BL and FL) samples exposed to water, jet fuel, hydraulic oil and weathering conditions

influence of the different coatings is too small to explain the difference. Since the fibers in NFRC are of natural origin, the deviation in the mechanical properties is accepted to be generally high, and therefore varies from plate to plate. This also results in high standard deviations within a sample set.

It can be seen that the references of the uncoated system with the higher fiber content (H) achieve the highest values, as the excess fiber additionally stiffens the composite material. Despite the high standard deviation, it can be shown that weathering and exposure to water leads to a significant deterioration of the flexural modulus of the uncoated samples, i.e. resulting in a loss of stiffness. The damage due to water penetration, swelling of the fibers and following delamination was already discussed in Chap. 3.1. If the force transmission between the load bearing fibers and the matrix is interrupted, the mechanical properties of the composite break down. In addition, the water promotes microbial and fungal growth on the natural fibers, which can also impair Young's modulus, for example [28]. The decrease in stiffness between the references and the samples aged for 28 days is 30% for H, but only 13% for L. Although H had a higher stiffness than L at the beginning, the mean value of H is even lower than that of L after 28 days of ageing. This behavior is due to the higher water absorption of H compared to L, which was observed in Fig. 6. The higher fiber content leads to increased water absorption and thus to amplified swelling, cracking and delamination, which deteriorates the mechanical properties of the NFRC.

The damage caused by UV light is attributed to photooxidation of the fibers and the epoxy resin matrix. Nasri et al. [51] showed for a polypropylene matrix that microcracks formed in the matrix due to the breaking of the bonds, which led to the deterioration of the mechanical properties. They also found that UV light in dry conditions has a deteriorating effect over time, but the presence of moisture accelerated the degradation strongly as the condensed water damages the composite additionally. In this study, condensation was possible during the weathering cycles resulting in the observed severe deterioration of the flexural modulus. The coated samples did show a deterioration for the samples immersed in water for 28 days. Contrary to intuition, the difference is clearer for the sample with the thicker coating layer (FL). For the weathering, they didn't show any significant variation. Therefore, it could be determined that the coating is an effective protection against weathering, but cannot protect the composite from extended immersion in water entirely.

In contrast, there is no decrease in the flexural modulus for the samples aged in jet fuel and hydraulic oil. The values remain constant within the standard deviation. This proves the resistance of the composite to jet fuel and hydraulic oil. For hydraulic oil, the uncoated sample with the higher fiber content even shows a slight increase in the flexural modulus. However, the difference is too small to be attributed to a physical mechanism.

Figure 9 shows the flexural strength of the samples. The observed effects are slightly less severe than for the flexural modulus, but the patterns continue and show the degradation due to weathering and water immersion. For example, the flexural modulus of H immersed in water for 28 days decreased approx. 29% compared to the reference whereas the flexural strength decreased approx. 21%. Saha et al. [52] investigated the properties of flax fiber-reinforced epoxy resin composites that were exposed to water for approx. 260 h. They also found that the flexural stiffness decreased with approx. 46% to a greater extent than the flexural strength with approx. 38%. In contrast, when irradiated with UV light, it has been reported in the literature that the strength deteriorates more severely than the modulus. Alam et al. [53] investigated the influence of UV radiation without condensation/

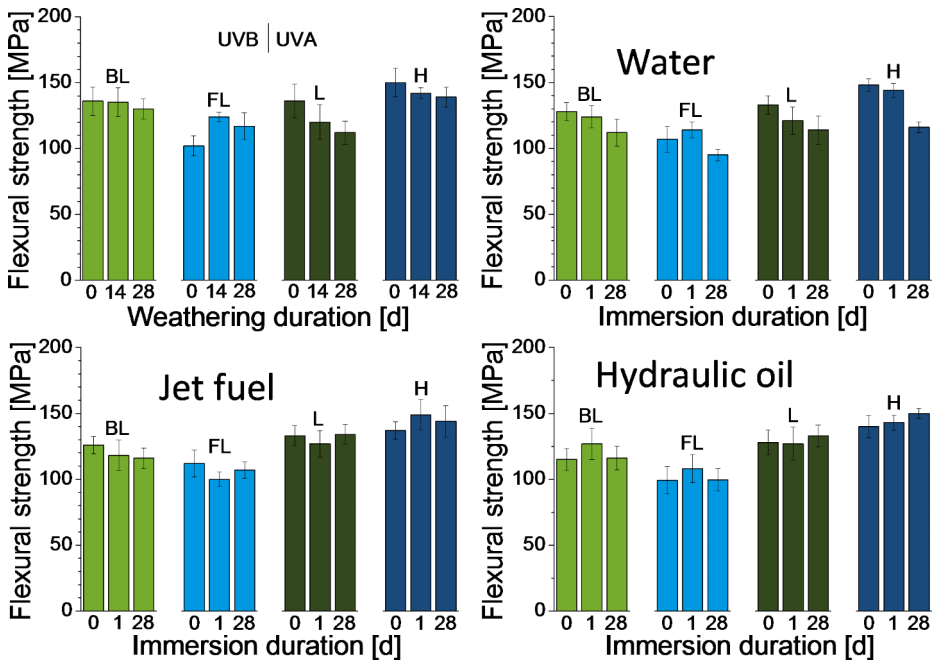


Fig. 9 Flexural strength of uncoated (F and L) and coated (BL and FL) samples exposed to water, jet fuel, hydraulic oil and weathering conditions

water spraying on flax fiber-reinforced polypropylene composites. They found only a slight deterioration in Young's modulus. However, depending on the type of polypropylene used, the tensile strength decreased by 13% and 23% respectively when exposed to 504 h of UV radiation without water spraying. Sit et al. [54] obtained similar results with flax fiber-reinforced polylactic acid, which was also exposed to UV light for 500 h in a dry environment. The flexural modulus decreased by only 6%, while the flexural strength decreased by 15%. These results are consistent with the investigations of Nasri et al. [51], who were able to show that the stiffness was maintained for 230 h under UV radiation in a dry environment, while the strength had already decreased significantly by this time. However, they also showed that moist condensation accelerates the loss of stiffness and strength. In this study, moist condensation was possible in the times in which the UV lamp was turned off. The degradation due to UV weathering observed shows a decrease in both the flexural modulus and flexural strength. The difference to the degradation behavior reported by Alam et al. and Sit et al. is possibly due to the fact that the condensate water was able to penetrate into the composite specimens and thus accelerate the deterioration of the flexural modulus. Another possibility is that the choice of matrix, in this study epoxy resin, could be responsible for the differences. Alam et al. [53] already saw differences in degradation behavior for different polypropylene matrices. As described in Sect. 3.2, epoxy resin is particularly susceptible to UV light. This could also lead to increased deterioration of the mechanical properties.

The highest reference values are achieved by the uncoated composite with the higher fiber content. This behavior is also found in literature. For example, Andersons et al. [31] showed that the tensile strength of unidirectional flax fiber-reinforced epoxy composites

increased from approx. 200 MPa at 21% v/v to approx. 280 MPa at 42% v/v. A further increase in the fiber volume fraction did not lead to an improvement in tensile strength. For twill weave flax fiber-reinforced epoxy composites, Alipour et al. [32] found an increase in tensile strength from 98 MPa to 126 MPa with a change from 33% v/v to 44% v/v. The stiffness increased from approx. 5 GPa to approx. 6 GPa. They indicate that if there is not enough polymer to laminate the fibers when the fiber content is too high, failure and a deterioration of the mechanical properties will occur due to insufficient load transfer into the fibers.

It can be seen that the flexural strength of the uncoated sample with the lower fiber content L decreases significantly when exposed to UV weathering. However, the effect is not significant for the sample with the higher fiber content H. One possible explanation for this could be that the epoxy resin matrix is particularly affected by UV irradiation and, as already described above in the discussion of Fig. 4, material is removed until the fibers are exposed. Although the fibers are ultimately also damaged by the UV radiation, as the color fading shows, they still shield the rest of the composite. Therefore, the composite with the higher fiber content may suffer less from epoxy degradation at the surface. Nonetheless, it must be noted that the mean value of the flexural modulus of H decreases more than the stiffness of L during weathering, which does not fit as both stiffness and strength should benefit from a possible protective effect. In addition, moist condensation is allowed during weathering, which should damage the sample with the higher fiber content H more strongly as observed during exposure in water. The standard deviations are high overall, so that the behavior cannot be conclusively evaluated.

There are no significant decreases in flexural strength for the coated samples. It can therefore be concluded that the coating provides effective protection of the composite against weathering so that the flexural strength is maintained.

The uncoated samples both show a decrease in flexural strength due to immersion in water. This deterioration is more severe for the uncoated sample with the higher fiber content. There is a strong reduction in strength. This is probably due to the fact that this sample also absorbed more water, which might have led to increased damage. For immersion in water, a deterioration in flexural strength can be observed for the coated samples in the case of the fossil coating (FL) between 1 day and 28 days of immersion. For the partially bio-based coating, the values are still within the standard deviation, so that no clear statement can be made here. It can be concluded that the fossil coating does not provide sufficient protection against prolonged contact of FFRCs with water.

Deterioration in strength due to exposure to jet fuel and hydraulic oil was not found. Neither jet fuel nor hydraulic oil appeared to significantly damage the fibers or contribute to delamination from the matrix during the tested time period. In contrast, in the study by Krzyżak et al. [20], a greater deterioration in tensile strength was found for FFRC exposed to jet fuel than for FFRC exposed to water. This result could not be reproduced in this study. Instead, comparing to three aviation fluids, water had the only significantly negative effect on the strength of the composites.

Similar to the findings in this study, Hurdelbrink [18] showed that classical materials for light weight design like glass fiber-reinforced composites (GFRP) are also quite stable against immersion in hydraulic oil. Stiffness reduction for most process parameters was less than 5% after 2 years of immersion. The flexural strength did not decrease by more than 15% and was dependent on the amount of hydraulic oil absorbed that varied for different

processing parameters [18]. For water immersion of GFRC, it was shown that the fiber orientation in the composite as well as the water temperature were important factors. Water at RT had hardly any effect on Young's modulus and tensile strength. This behavior differs substantially from the FFRC investigated in this study, which are very sensitive to immersion in water. However, water at 90 °C was absorbed much better by the composite and increased the damage. It was also shown that the mechanical properties for $-45^{\circ}/45^{\circ}$ laminates both at RT and at 90 °C water led to significantly increased degradation compared to $0^{\circ}/90^{\circ}$ laminates [55]. Similar behavior was observed for carbon fiber-reinforced composites (CFRC) in water. Sala et al. [17] showed that immersion in water for 30 days reduced Young's modulus of CFRC by only 2.4% for $0^{\circ}/90^{\circ}$ laminates. In contrast, the reduction in Young's modulus for $+45^{\circ}/-45^{\circ}$ laminates was 49%. It must therefore be considered that although the classic lightweight materials appear to be less susceptible to water, their use in contact with aviation fluids is not entirely straight-forward.

4 Conclusion

In this study, the effects of UV weathering and immersion in the aviation fluids water, jet fuel and hydraulic oil on the optical and mechanical properties of flax fiber-reinforced epoxy composites were comparatively evaluated. Uncoated samples as well as samples coated with a partially biobased automotive interior coating and an aircraft coating were compared. It was found that UV weathering and contact with water lead to deterioration in both the optical and mechanical properties (flexural modulus, flexural strength) of uncoated samples. Composites with a high fiber content are more susceptible to water degradation, as they absorb higher amounts of water and are therefore likely to swell which leads to fiber-matrix delamination. The effect of hydraulic oil and jet fuel is negligible compared to the damage caused by UV weathering and water. No degradation of the optical and mechanical properties was observed over an exposure period of 28 days. This is probably due to the fact that the main damage mechanism of water is the swelling of the fibers. However, hydraulic oil and jet fuel were hardly absorbed by the fibers and therefore did not damage the composite. Both the partially biobased automotive interior coating and the fossil-based aircraft coating were able to protect against UV weathering and delay the absorption of water. However, after 28 days of immersion in water, the mechanical properties of the FFRC covered with the fossil aircraft coating showed decreased flexural modulus and strength. Coating adhesion and hardness were barely affected by the environmental factors. It can therefore be summarized regarding stability towards environmental factors, that flax fiber-reinforced composites could very well be suitable for exterior components in the aviation sector when protected with a suitable coating. Prolonged direct water contact should nevertheless be avoided.

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Data Availability Data is provided within the manuscript and supplementary information. Raw data files are available from S. Schulte upon reasonable request.

Declarations

Competing Interests The authors declare no competing interests.

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