CONTINUOUS MANUFACTURING OF HYBRID AND RECYCLABLE FIBER-REINFORCED THERMOPLASTIC PROFILES WITH THERMOPLASTIC TOP LAYER COATING

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ABSTRACT

Pultruded profiles are widely used as continuous fiber-reinforced semi-finished products in the building industry, for example in load-bearing parts of bridges, façade or window applications. To access new applications such as large-scale construction profiles, the pultruded profiles require adequate mechanical properties and excellent optical and haptic properties, which is challenging to provide with the fiber surface structure intrinsic to the pultruded material. In the present contribution, a process combination consisting of pultrusion and extrusion process is investigated to functionalize pultruded profiles by inline application of a thermoplastic coating layer. As resin for impregnating the fibers in the pultrusion process, an in-situ polymerizing acrylic resin is used. Based on challenges associated with the combination of two continuous processes involving different materials and processing conditions, a suitable process window has been identified and extensively studied. The hybrid profiles quality is assessed using mechanical testing methods and microscopic examinations of the profile cross-section area and surface. A particular focus is on studying the intrinsic bond between the pultruded profile and the cover layer. The new process hybridization opens the potential for one-step economic production of thermoplastic profiles with improved surface quality, impact strength and possible mechanical and chemical recycability.

INTRODUCTION

Pultruded profiles are widely used as continuous fiber-reinforced semi-finished products in the building industry [1]. The pultrusion process enables the cost-efficient manufacture of fiber-reinforced polymer (FRP) composite structures with constant cross-sections with comparably low energy intensity [2,3]. Therefore, the pultrusion process exhibits excellent potential for energy-efficient industrial series production, even for renewable materials [2]. However, to access new applications such as large-scale fully thermoplastic building profiles, the pultruded profiles require adequate mechanical properties and excellent optical and haptic properties, which are challenging to provide without subsequent finishing processes [4].

Although thermoset pultrusion market demonstrates significantly higher production volumes than those of the thermoplastic pultrusion, thermoplastic components exhibit many advantages compared to thermoset profiles [1]. Thermoplastic resins have high impact toughness [5], can be processed by welding [6], exhibit less environmental impact [3,7], and permit recycling of the material [3,8]. The increasing demand for recycling and improved manufacturing processes has led to a growing interest in thermoplastic resins [9]. During thermoplastic reaction injection pultrusion, the polymerization reaction occurs in the pultrusion die, opening up a process primarily reserved for thermosets to thermoplastic materials such as caprolactam resulting in Polyamide 6 (PA-6) and liquid reactive acrylic resin [9,10]. For thermoplastic reaction injection pultrusion, injection boxes are commonly used as certain resins' sensitive polymerization reaction needs to be isolated from environmental influences [3,11,12] and allow processing of highly-reactive resin systems [13].

Despite the advantages, pultruded profiles' surface quality is mostly not suitable for cosmetic applications without subsequent finishing [4]. Environmental influences may cause 'fibre-blooming' of uncovered fibers, preferably in exposed areas [14] and can ultimately result in the degradation of the composite structure [4]. Typically, resin-rich surfacing veilings are applied to enhance the weather resistance or application of inline powder coatings [4]. Still, for cosmetic applications, a subsequent cost-intensive finishing process is required to ensure 'class A' surfaces, which is accompanied by the emission of environmentally harmful volatile organic compounds [15,16]. The *Coaline* process introduced in [17] aims to reduce required finishing steps and emissions during the production of coated pultruded profiles by integrating forming, coating and finishing into the pultrusion die. Resin and gelcoat are separately injected into the die, while combined thermal and microwave curing allows reliable and fast processing [16]. To overcome the surface issue that pultruded profiles' surface quality is often unsuitable for cosmetic applications and add complementary protection from environmental impacts as well as weldability [4] of the profiles, a process combination consisting of a pultrusion and extrusion process (PulEx) has been developed in [18,19]. It has been demonstrated that pultruded glass FRP profiles with the polyurethane (PUR) matrix can be co-extruded in line with a thermoplastic polyethylene (PE) and PA coating layer. *Schneider* demonstrates by experimental investigations that the impact strength of the hybrid profiles is significantly affected by thermoplastic coating, and surface quality improvement is verified. Furthermore, excellent adhesion (in the range of the coating layer material strength) between the pultruded core and the PA coating has been shown [19].

The profile coextrusion is based on combining the properties of the constituent layers (pultrude profile: high stiffness, impact; coating layer: optical and haptic appearance, fiber protection) in a single product while achieving good adhesion between the different layers [20]. For this, viscosity development during curing needs to be equal [18], and the exact temperature of the joining partners benefits the adhesion. Furthermore, from joining thermoplastics it is known that the joining process of two partners yields the best results when the viscosity ratio is close to 1 [21].

For target building applications (e.g., windows and doors element applications) where high stiffness and, at the same time, high environmental resistance and excellent surface properties are required, the PulEx process appears to provide a suitable manufacturing technology. PMMA, in particular, is suitable for this purpose as it provides good resistance to harsh UV radiation and weather conditions [22]. In addition to an energy-efficient production process, material selection plays a crucial role in realizing comprehensive recycling strategies of the hybrid profiles. FRP recycling is challenging because FRP are inherently a mixture of different materials that need to be separated so that recycling requires significant expenses [23]. Recycling routes, including remelting and subsequent forming processes, are reserved for thermoplastic matrix materials. During the curing of thermoset materials, chemical bonds are formed between the molecular chains, creating three-dimensional network structures that cannot be remelted or dissolved in solvents [24]. Concerning the PulEx process, a thermoplastic instead of a thermoset material seems a desirable core material to recover and reuse material and allow mechanical and chemical recycling approaches [25,26]. At the same time, applying a similar polymer material for a coating layer saves an additional separation step within the recycling process, as the core material does not need to be sorted from the coating layer. Furthermore, selecting the same polymers leads to sufficient adhesion strength between the core and coating layer [27,28].

In the present contribution, the PulEx process combination consisting of pultrusion and extrusion process is investigated to functionalize pultruded profiles by inline application of a thermoplastic coating layer. In contrast to previous studies with thermoset resin systems [18,19], as resin for impregnating the fibers in the pultrusion process, an in-situ polymerizing thermoplastic resin is used to meet the requirements for recyclability and material recovery, high impact strength, and reduced environmental impact [3]. Furthermore, by combining identical polymers in the pultrusion profile and the coating layer, the potential for mechanical and chemical recycling through depolymerization is to be increased. Within the contribution, a stable process window for the hybrid process is identified and extensively studied based on challenges associated with the combination of two continuous processes involving different processing conditions. Furthermore, mechanical testing methods and examinations of the profile cross-section area and surface quality assess the hybrid profile quality. A particular focus is on studying the intrinsic bond between the pultruded profile and the extrusion layer.

2 MATERIALS AND METHODS

This chapter first presents the material and experimental environment, then describes the analytical methods used to access the pultruded and hybrid profiles' quality.

2.1 Materials

For the pultrusion process, the thermoplastic acrylic Elium® C595 resin provided by Arkema S.A., France, is selected. According to the manufacturers' recommendation, three peroxides with 1 phr serve as initiators for the polymerization reaction [10]. Additionally, 2 phr internal release agent from Axel Plastics, USA, and 2 phr of a Norrish type I photoinitiator from Arkema Sartomer, France, are incorporated into the resin. Finally, extrusion-type impact-modified PMMA extrusion material (Röhm GmbH, Germany) is applied as top layer coating for the coextrusion process.
All trials use 4,800 tex rovings provided by Johns Manville Germany GmbH, Germany, resulting in a fiber volume content of 65 vol.-% (82 wt.-%) in the pultruded profile. According to the manufacturer, the applied silane sizing is suitable for processing the reactive acrylic thermoplastic resin system.

2.2 Experimental set-up of hybrid process

The experimental set-up is similar to [18,19] and depicted in Fig. 1. A Pultrex 500 pultrusion machine by Pultrex Ltd., England, with reciprocating pullers, serves as the basis for the hybrid process.

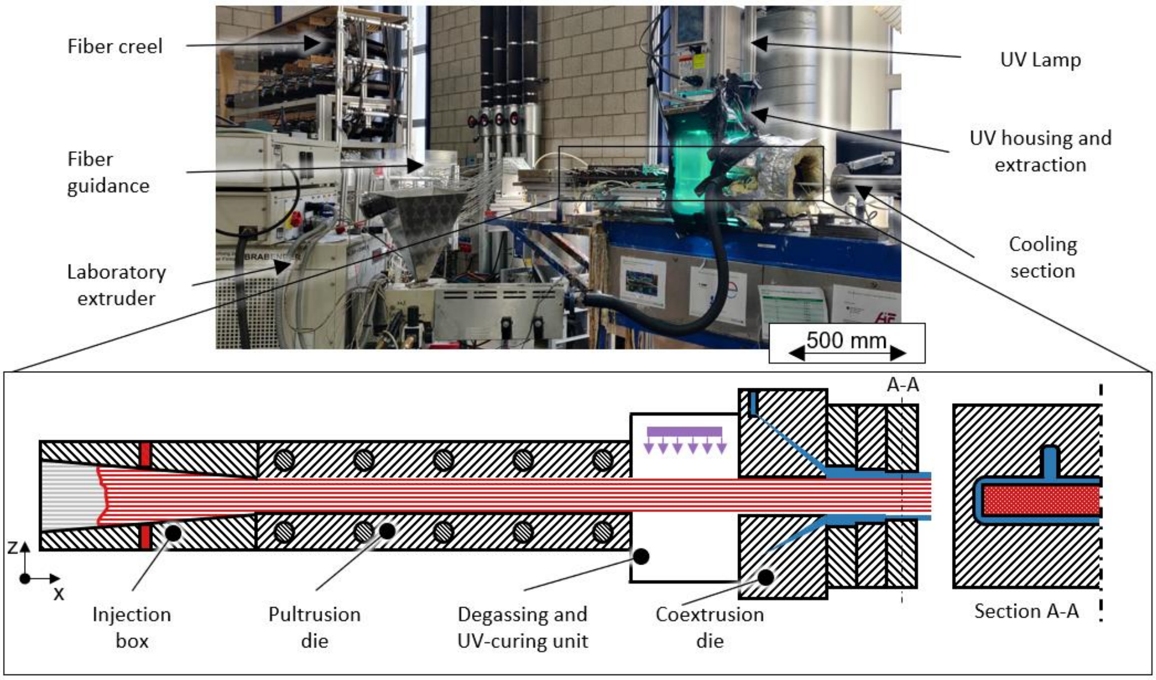

Figure 1: Process layout for the PulEx process (top) and sectional view of the tooling (bottom).

An aluminum open injection box, a pultrusion die with a rectangular cross-section (4 x 35 mm²), and the coextrusion die introduced in [18]. In contrast to [18], a pultrusion die with a length of 700 mm was used to provide more time for the polymerization reaction. The joining of pultruded profile and coextruded layer occurs immediately after the feed profile leaves the centre melt guidance and is exposed to the thermoplastic melt. The thermoplastic top layer shape is formed in a conventional extrusion die set-up, which features transition and parallel zones. These different zones, which are realized by three nozzle plates, gradually form the top layer of the profile to the final shape (Fig. 3). Both dies are mounted on a floating sled connected to a force sensor (C9C by Hottinger Baldwin Messtechnik GmbH, Germany) with a maximum force of 5 kN. An additional section for UV post-curing of the pultrusion profiles is integrated between the pultrusion and the coextrusion die to increase the maximum haul-off speed limited by the polymerization reaction. A microwave-powered electrodeless lamp F300S by Heraeus GmbH, Germany is selected as UV and additional heating source. Optionally, a heated air blower can be integrated in the UV section. For the pultrusions' material supply, a peristaltic pump Ismatec MCP Process by Cole Palmer GmbH, Germany, is utilized for volumetrically dosing the reactive mixture into the injection box. The thermoplastic melt for the coating layer is fed into the die using a single-screw laboratory extruder of the type Plasti-Corder from Brabender, Germany, with a screw diameter of 19 mm and L/D ratio of 44. To evaluate the process frame of the PulEx process haul-off forces, haul-off speed and die temperatures are continuously monitored at a rate of 2 Hz. Immediately after the hybrid profile exits the coextrusion die, convective cooling ensures sufficient dimensional stability for the pullers.

2.3 Experimental methods

By systematic variation of the resin formulation, die temperatures, and haul-off speeds, a robust process window is identified by microscopic examinations of the profiles' cross-section area and dimensional stability. Ten hybrid profile samples are investigated every 3 m of produced profile to provide a basis for evaluating the process stability. Micrographs of the profile's cross-section area are recorded by a digital microscope VHX5000 of Keyence AG, Japan, to verify dimensional stability. Subsequently, stylus profilometry quantifies the influence of the extrusion layer on the hybrid profile's surface properties. Three equidistant lines with a length of 17.5 mm and spacing of 3 mm are investigated in the center of the hybrid profile. Measurements are conducted in the y-direction (perpendicular to the main orientation direction of the fibers). After measuring the primary surface profiles, the measured profiles are filtered to calculate roughness characteristics. According to DIN EN ISO 4288, a Gaussian filter (DIN EN ISO 11562) with cut-off wavelengths of $\lambda_c = 0.25$ mm is selected to remove the waviness profile and non-relevant wavelength. The arithmetic average of the profile height (R_a) and maximum peak to valley height of the profile (R_z) are employed to evaluate the surface characteristics. Further, a portable CM-25cG spectrometer from Konica Minolta, Japan is utilized to conduct 60° gloss measurements (DIN EN ISO 2813).

The endless pultruded and hybrid profiles are cut to the specimen sizes for adhesion and bond strength testing utilizing a circular saw with a diamond blade. Reference specimens for the coextrusion material are injection molded utilizing a ergotech 80/420- 310 from Demag Plastics Machinery GmbH, Germany, with a tool temperature of 60 °C. The adhesion or bond strength between the PMMA coating and the pultruded profile is determined following ISO 4624 and DIN EN 15870 utilizing the adhesion analyzer LUMiFrac 200 (L.U.M. GmbH, Germany). A copper test stamp (Type TSU-M-V2A from L.U.M) with a test area defined by 10 mm diameter is glued to the extrusion layer using cyanoacrylate-based Loctite 406 adhesive by Henkel AG & Co. KGaA, Germany. A centrifuge accelerates the copper stamp by increasing the centrifugal force by 5 Ns^{-1}. When the specimen's fracture was detected, the bonding force was calculated using the rotation speed at this point. The adhesion is analyzed by relating the bonding force to the fracture area.

The impact strength of the pultruded and hybrid profiles is determined according to the test method ISO 179-1/1eUb with a pendulum impact tester of type S101 from ZwickRoell, Germany. For this, 80 x 10 mm^2 unnotched specimens are prepared from the center of the pultruded profile and tested on the narrow side with a support distance of 62 mm. A 25 J impact hammer is used. Additionally, 3-point bending testing of the pultruded and coextruded profiles follow DIN EN ISO 14125 by utilizing a universal testing machine Z150 by ZwickRoell, Germany. The testing machines' software records the test specimens' force and displacement and provides the basis for the mechanical evaluation. The universal testing machine records the time, transverse displacement, and standard force during the measurement. Temperature and humidity are continuously recorded during mechanical testing and amount to 22.8 ± 0.3 °C and 30 ± 1 % humidity.

3 RESULTS AND DISCUSSION

In this chapter, the process development of the hybrid process is described first. Subsequently, the pultruded and hybrid profiles' profile properties (dimensional stability, surface quality, adhesion and mechanics) are successively compared.

3.1 Process development

First, a suitable processing window is identified by systematically adjusting the pull speed (v_{pull}), extrusion and pultrusion die temperature and melt feed. Depending on the process point, the additional inline coextrusion process increases the pull-off force by up to 23 %. The melt is assumed to form a sliding film on the pultrusion profile surface, thus reducing solid-solid (pultrusion profile surface - die surface) friction. At the lower end of the processing window of the extrusion material (T < 230 °C) and the increase in melt viscosity, a significant increase in the pull-off force by up to 100 % accompanied by a less robust process is observed. The limiting factor for the total throughput is v_{pull}, since on the one hand, the polymerization must run entirely, and on the other hand, the evaporation of residual monomer methylmethacrylate of the elium resin must be prevented. The residual monomer evaporates through the significantly warmer coextrusion die and becomes visible as gas entrapment at the boundary layer between the pultruded profile and coating (Fig. 2), thus leading to poor visual and haptic and haptic appearance.

To increase the pull speed, an additional dual (thermal and UV) curing process is integrated to improve the product quality simultaneously. Fig. 2 shows the temperature profiles in the injection box, the pultrusion, and the CoEx die recorded for the process set-up with and without dual curing. During the pultrusion process, the pultruded profiles core temperature rises to 125 °C at the pultrusion dies exit and 238 °C in the PulEx die. As reported in [29], MMA evaporation (101 °C at standard pressure) significantly influences PMMA/GF adhesion quality. However, as the pressure in the injection box and pultrusion die considerably increases [30,31], the boiling shifts to higher temperatures. As the final glass transition temperature $T_{g,\infty}$ of the resin systems amounts to 119 °C [10], during the CoEx process the resin material is permanently in the rubbery state. Integrating the dual curing section increases the temperature in the pultrusion die caused by radiant heating. As a result, the temperature at the die exit is approximately 70 °C higher than without dual curing. Afterward, temperature decreases, indicated by the suction before the profile enters the PulEx die, and temperature sharply rises.

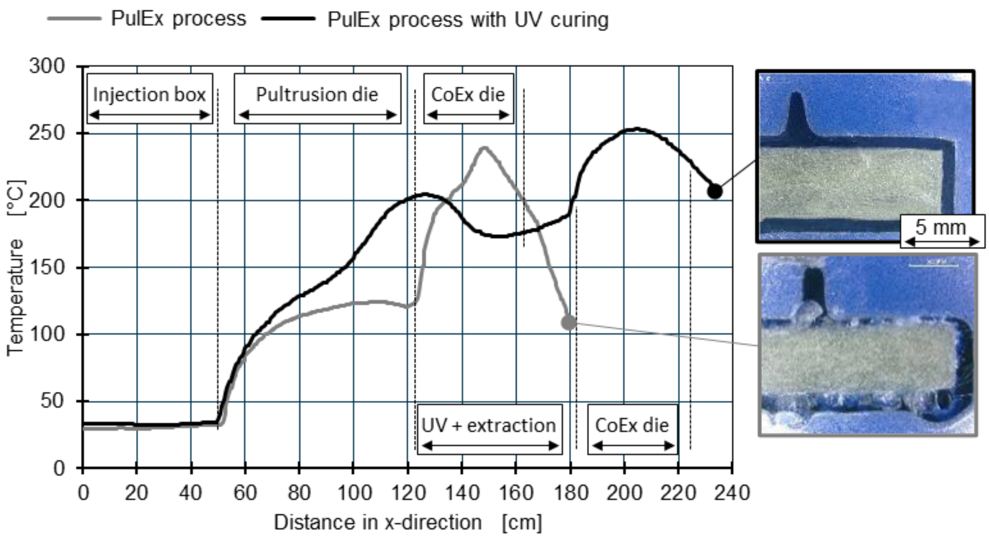

Figure 2: Temperature profiles in the center of the pultrusion profile for the PulEx process with ($v_{pull} = 0.5\ m/min$) and without ($v_{pull} = 0.25\ m/min$) dual curing. Cross-section of the PulEx profiles.

By integration of the dual curing section v_{pull} can be increased from 0.25 m/min to 0.5 m/min by simultaneously increasing the profile quality and eliminating gas entrapments (Fig. 2) at the interface of pultruded profile and the extrusion layer.

3.2 Dimensional stability of the hybrid profile

A cross-section of the profile is given in Figure 3. Both the extrusion film and the rib structure are well-formed.

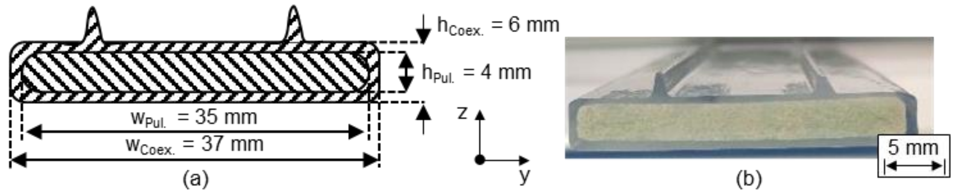

Figure 3: Cross-section area of the hybrid profile (a) and (b) photographic image.

The shape tolerances (thickness: ± 0.4, width: ± 0.8) for extruded parts according to DIN 16941 are met for both the pultrusion (3.92 ± 0.05 mm) and the coated profile with 5.84 ± 0.04 mm in thickness and width direction (34.72 ± 0.14 and 36.62 ± 0.04 mm). The alignment of the pultrusion profile in the mold represents a particular challenge. Although the overall tolerances are met, unequal cover thicknesses are measured, especially in the height direction. These can amount to up to 0.34 mm in the profile range investigated.

3.3 Surface quality

Fig. 4 compares the surface quality of the pultruded and the hybrid profile. The surface quality can be significantly increased by applying the thermoplastic extrusion layer. R_a decreased from 12 ± 3 µm to 0.25 ± 0.1 µm and R_z (which indicates surface defects) decreases by factor 9. The considerable improvement in surface quality due to the top layer application can also be observed in the surface profiles recorded by LCM (Fig. 4 (b)).

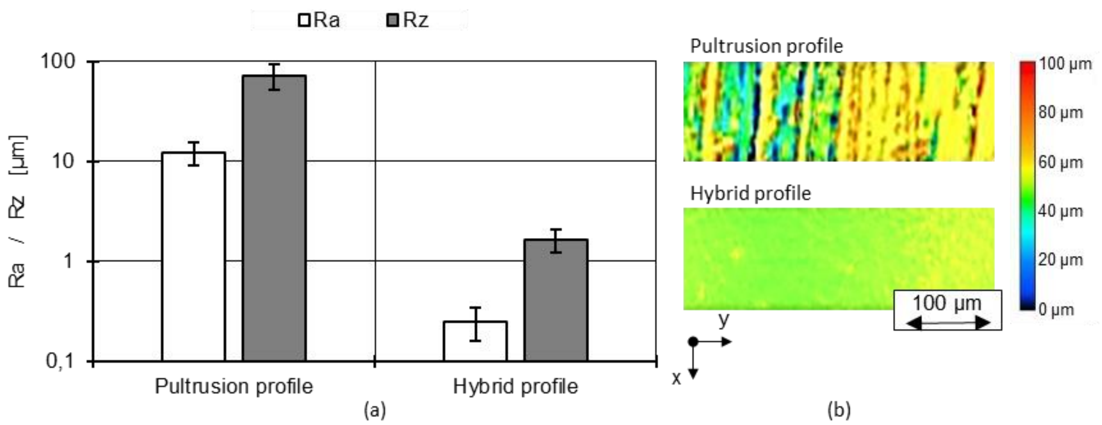

Figure 4: Average roughness of the pultruded profiles compared to hybrid PulEx profiles (a). Surface profiles recorded by LCM (b).

At the pultrusion profiles' surface, the individual surface-near fibers are visible as groves running in the x-direction, while in the hybrid process, a significantly smoother surface is formed. As the Fiber print through (FPT) effect represents a significant challenge during FRP manufacturing, inline coating application is an attractive opportunity to improve surface quality right from the mold. The FPT emerges from different coefficients of thermal expansion of the fiber and resin constituents and the polymerization shrinkage of the resin material. Since fiber-rich and resin-rich areas coexist at the surface, the FPT becomes visible during polymerization and cooling [32,33]. The gloss the uncoated and coated surface are determined to 12 and 69 GU. By applying the extrusion layer, the surface gloss can be significantly increased and initially improved from an eggshell-like finish (10 - 25 GU) to a semi-gloss (35 - 70 GU) or even gloss finish (70 - 85 GU) [34].

Therefore, the inline application of the extrusion layer can significantly influence the surface quality of the components and meets the requirements of 15-20 GU and a homogeneous, stripe-free surface. Further extrusion optimization of the profile surface, e.g. through a calibration unit might increase the surface quality.

3.3 Adhesion of the extrusion layer

Fig. 5 presents the adhesion testing results conducted by the LUMiFrac 200 adhesion analyzer. An adhesion strength of 1.79 MPa is reached for the C595 material and increases to 2.29 MPa by the additional dual curing section. After adding convective heating, the adhesion strength slightly increases by 5 % up to 2.40 MPa. The coextrusion of the coating layer might be compared to the extrusions welding and is characterized by similar materials, heating the joining partners to welding temperature, and a pressurized joining process [35]. In addition, the following aspects are decisive for the adhesion achieved between the pultruded profile and the surface coating:

- Equal flow velocity of pultrusion profile and melt in the welding zone of the coextrusion die [36].
- Similar temperatures and a viscosity ratio of approximately 1 for both joining partners [18,21].
- For hot plate welding of PMMA, temperatures of 230 to 280 °C and pressures of 0.2 to 0.6 MPa are recommended [28]. At higher welding temperatures, the weld strength tends to increase as long as no material degradation occurs [27]. In addition, chemical bonding and molecular mobility for the interdiffusion process are enhanced by elevated temperatures [37].

The welding strength increased through additional heating of the pultruded profile and, thus, a smaller temperature gradient between both (pultruded profile, melt feed) joining partners. The adhesion decreases significantly without the dual curing section and the respective radiation heat exposure (Fig. 2) before entering the welding zone.

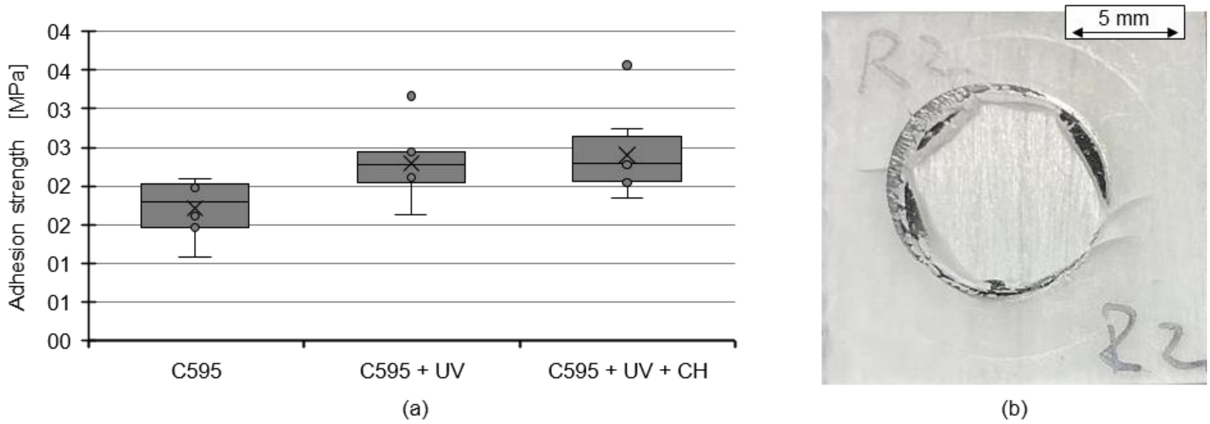

Figure 5: Adhesion strength of the surface coating and pultruded profile determined by LumiFrac measurements (a). In addition to the dual curing, convective heating (CH) by a heated air blower is performed. (b) shows the fractured surface. Black edges mark the remains of the adhesive.

In general, excellent adhesion of the layers is found, which is in the range of the bonding strength of PECVD-coated polypropylene surfaces, which amounts to 2.0 ± 0.5 MPa [38] using the equal evaluation method. While the formation of gas entrapments increases at elevated temperatures, a sharp increase in the pull-off force at lower die temperatures makes slight variation possible for the coextrusion die temperature range.

3.4 Mechanical testing of the hybrid profile

Charpy impact strength of 478 ± 20 kJ/m² is determined for the pultruded profile, which is located above an epoxy-GF (405 kJ/m²) and vinyl ester-GF (380 kJ/m²) profile (405 kJ/m²) produced in [39] on a similar machine set-up. The coextruded profile's impact strength amounts 218 ± 29 kJ/m² with dual curing. Charpy impact strength a_{cU} is evaluated according to equation 1:

$$a_{cU} = \frac{W_C}{h \cdot b}, \tag{1}$$

where h and b represent the thickness and width of the specimens and W_C the required energy to break the specimen. Appling role of mixture and $a_{cU,zk6hf}$ = 75 kJ/m² [40], the overall $a_{cU,hybrid}$ can be estimated to 343 kJ/m² which above the experimentally determined values. A change in the failure criterion of the test specimens is observed (Fig. 6). In the case of the pultruded profile, buckling or compression failure is observed. The hybrid test specimens show a shear or multiple shear failures.

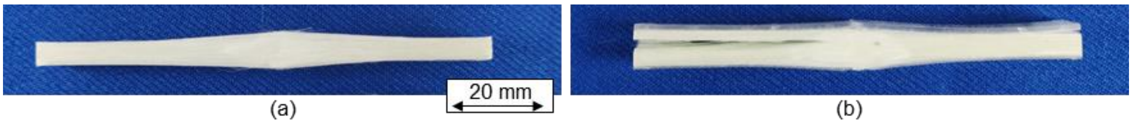

Figure 6: Pultruded (a) and hybrid (b) test specimens. Failure modulus changes from buckling or compression failure for the pultruded profiles to shear failure for the hybrid profiles.

Beneath the Charpy impact strength and adhesion, the mechanical characterizations of the hybrid profiles cover three-point flexural testing. Fig. 7 shows the stress-strain curve of the pultruded and the hybrid profiles, and Tab. 1 gives an overview of the characteristic values.

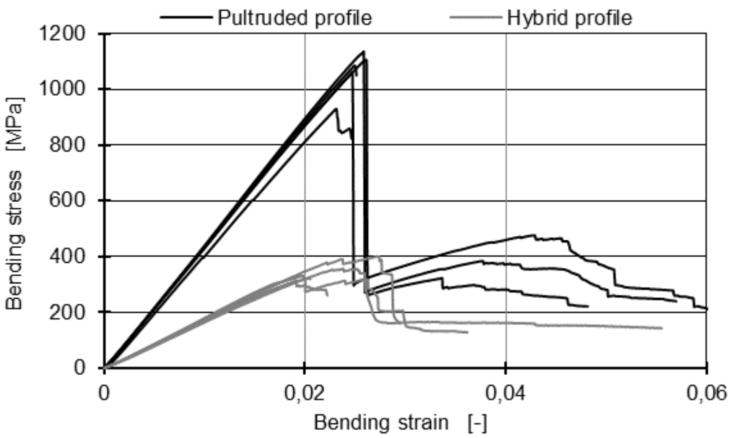

Figure 7: Stress-strain curve of pultruded and hybrid profile in a three-point bending flexural test.

	Flexural strength [MPa]	Flexural modulus [GPa]	Elongation at break [%]
Pultruded profile	1063 ± 42	43.8 ± 0.9	2.5 ± 0.1
Hybrid profile	330 ± 35	17.9 ± 0.8	2.1 ± 0.4
zk6hf extrusion material	63 ± 3	1.5 ± 0.1	6.7 ± 0.2

Table 1: Mechanical characteristics values for the pultruded, hybrid profiles and the neat coextrusion material (zk6hf) obtained by three-point bending test.

The determined flexural modulus of the pultruded profile agrees with the literature data published in [10]. Hybridization of the pultruded profile significantly reduces the flexural modulus by 68 % and flexural strength by 59 %. Since the surface layer is targeted in the tests and the profiles' thickness increases by 50 % from 4 to 6, a substantial decrease is to be expected. As a result of the hybridization, the elongation at break decreases from 2.5 % to 2.1 % and scatters significantly more than in the fully reinforced pultruded profiles. However, the failure occurs less suddenly, and the decay of the stress-strain curve indicates the fracture. The hybrid profiles' experimental determined flexural strength is significantly below the pultursion profiles' flexural strength. It is, therefore, reasonable to assume that the high temperatures caused by the dual cure process in combination with the Norrish type I photoinitiator influence the polymerization reaction behavior so that the properties of the resulting neat polymer are negatively affected.

The hybrid profiles' Charpy impact and bending properties were determined and compared with the pultruded profiles' results. Applying an unreinforced extrusion layer reduces Charpy impact strength and bending properties. However, the continuous fiber reinforcement of the core still has an effect, and significantly higher values are obtained than for the neat coating layer material.

4 CONCLUSION

In the present contribution, thermoplastic pultrusion and the extrusion process are inline combined to a hybrid PulEx process. Hybrid profiles were manufactured and characterized in terms of dimensional stability, surface and mechanical properties. Integrating an additional dual curing section increases the hybrid process's haul-off speed by up to 50 % while improving the profile properties. The process meets the dimensional tolerances for profile extrusion according to DIN 16941. In addition, the surface quality can significantly be increased by the inline application of a thermoplastic cover layer. Further, an excellent adhesion strength of up to 2.4 MPa between pultruded profile and the extrusion layer is demonstrated.

The new process hybridization opens the potential for one-step economic production of thermoplastic profiles with improved surface quality, impact strength, and additional weldability. Future analyses will focus on the transferability of the hybrid process to more complex profile geometries, detailed analysis of the dual cure polymerization and extensive studies on mechanical and chemical recycling approaches for the hybrid profile.

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