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The Recycling of Carbon Components and the Reuse of Carbon Fibers for Concrete Reinforcements

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Abstract: Carbon fiber reinforced plastics are increasingly used in all areas of industry. With the increasing number of components and semi-finished products, more and more new carbon fibers will be produced. This also generates a greater number of end-of-life components. These end-of-life components can currently only be fed back, to a limited extent, for reuse, thus leading to a non-optimal, closed-material cycle of the carbon fiber. This article provides an overview of the recycling of carbon components, their further processing and their reuse in reinforcement elements made of carbon fibers. In addition, first results from recycled single fibers and yarn tensile tests from recycled carbon fibers (rCF) are presented. By demonstrating the reuse of carbon fibers in the construction sector, there is the potential to effectively close the carbon cycle. The utilization of carbon reinforcements also enables the reduction of concrete consumption, as the minimum concrete cover required to protect the reinforcement from corrosion is no longer needed.

Keywords: recycling; carbon; reinforcement; yarn; single fiber; tensile tests; rCF; pyrolysis; solvolysis

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1. Introduction

Carbon fiber reinforced polymers (CFRP) are being used in an increasing number of areas in recent years. Examples include the automotive and aircraft industries, the manufacture of wind turbines and space exploration [1]. The global demand for carbon fibers in 2022 was around 117 kt [1].

In addition to the use of carbon in the automotive and aerospace industries, carbon fibers are increasingly being used in the construction sector in the form of reinforcements. In the construction sector, concrete or reinforced concrete is the most widely used building material. According to [2], due to ongoing population growth, the construction industry is responsible for up to 70% of all land alterations (such as land sealing), up to 50% of resource consumption, and up to 40% of energy consumption. In 2018, 4.11 billion tons of cement were produced worldwide, resulting in 2.25 billion tons of CO_2 emissions. According to [3], a thick concrete cover layer is typically required to protect the steel beneath it against corrosion, depending on the component and other factors. In contrast, carbon fibers are corrosion-resistant. As a result, a large amount of concrete can be saved by replacing the reinforcement steel with carbon rods or carbon mats, since the concrete cover that protects the reinforcement steel from corrosion can be reduced. As the density of carbon fibers is four times lower compared to reinforced concrete and the tensile strength up to six times higher, up to 80% of the concrete can be saved compared to reinforced concrete while still having the same load-bearing capacity [4]. For example, the production of a reinforcement mat made out of virgin carbon fibers requires 12.8 t CO_2/t [4]. Global carbon fiber waste is projected to increase by 20 kt per year by 2025 [1]. Additionally, a large number of carbon components reach their end of life and must be recycled or disposed of. Therefore, a sustainable reuse of carbon fibers is essential. The use of recycled carbon fibers in construction represents a great potential area of application. However, the development of components from recycled fibers is still an incompletely explored research field.

This article provides an overview of the different methods of recycling carbon fibers and presents the production of carbon reinforcements from recycled carbon fibers. Moreover, initial tactile tests (single-fiber tensile tests and yarn tensile tests) with recycled carbon fibers are presented, and the resulting challenges for reinforcement development are outlined. The novel production of non-metallic reinforcements based on recycled carbon fibers aims to close the carbon cycle. This article provides an overview of the initial considerations and preliminary investigations regarding the production of reinforcements from recycled carbon fibers.

2. Methods

The recycling of carbon fibers or end-of-life components poses great challenges to research compared to steel. In particular, the separation of the individual carbon fibers from the matrix without damaging the actual fibers requires appropriate knowledge. According to [1], recycling can be divided into three main groups. The first option is mechanical processing. In this case, for example, end-of-life components are mechanically fragmented into their constituents. The second option is thermal recycling. In this process, high temperatures (pyrolysis, fluidizes bed method) are used to separate the matrix from the carbon. The third option is solvolysis. In this method, the matrix is extracted from the carbon fiber using a chemical solvent [1].

2.1. Mechanical Recycling

The mechanical reprocessing is the most used principle for recycling carbon fibers. According to [5], slow-running cutting mills reduce end-of-life components into pieces that are from 50 mm to 100 mm in size. In comparison, fast-running mills produce fragments ranging in size from 50 µm to 10 mm for homogenous components. A classification of recycled materials can be made depending on the fiber or matrix. Often, mechanical comminution is preceded by thermal- or chemical-recycling processes. The use of carbon fibers in construction also requires a mechanical pre-treatment of end-of-life components. In particular, [2] this deals with the processing of broken components containing carbon fibers.

In the context of a research project, a first statement about the separation of concrete and carbon reinforcement should be made. For this purpose, a demonstrator building (Figure 1) made of carbon concrete was demolished with usual devices in the demolition process. The fragments were fed to the comminution and processing. By reducing the fragments down to a maximum grain size of 56 mm, the carbon reinforcement is completely separated from the concrete. The confirmation of a significant portion of carbon remaining in the concrete could not be established. With the help of various sorting devices (magnetic separation, camera-based single-grain sorting, etc.) a degree of separation greater than 99% could be achieved [6].

The results of [6] showed that the separation of carbon reinforcement and concrete from demolition debris is very feasible in construction practice.

2.2. Thermal Recycling

Thermal recycling of carbon fibers involves the separation of plastic-coated metals and similar mixed materials using high temperatures [7]. According to [5], there are at least two different thermal recycling methods. The two procedural options, the fluidized-bed process and the pyrolysis process, will be explained in the following sections.

2.2.1. Fluidized-Bed Process

The process chain consists of two steps. In the first step, a hot-air stream of 450 °C to 550 °C flows through a sand bed of silicate. This process dissolves the matrix from the carbon fiber and allows the matrix to be separated from the sand bed. The average grain size of the silicate is 0.85 mm. The speed of the heated air flow is between 0.4 m/s and 1.0 m/s. For optimum results, the composite components were broken down into 25 mm pieces

and individually added to the silicate bed. Finally, the fibers are separated from volatile compounds by means of cyclone or sieving technology. In the second step, the volatile compounds are oxidized at a temperature of 1000 °C [5]. Consequently, carbon fibers of lengths between 5.9 mm and 9.5 mm could be obtained according to [5]. Furthermore, the individual carbon fibers had only an 18.2% reduction in tensile strength, compared to the new fiber. In comparison to the production of new carbon fibers, the fluidized-bed method requires only 5–10% of the total energy [5].

Figure 1. Demonstrator building made out of carbon reinforced concrete; photo by Jan Kortmann and Florian Kopf.

2.2.2. Pyrolysis Process

In contrast to the fluidized-bed process, the carbon fiber components are heated via a pyrolysis process in the absence of oxygen. This causes the matrix to be released from the individual fibers and to decompose into a gaseous and liquid state. Depending on the fiber composite material, the pyrolysis process takes place between 450 °C and 550 °C. Since the temperature is below the decomposition temperature of carbon fibers (>600 °C), the pyrolysis method is very suitable for the separation of matrix and carbon fibers [7]. The disadvantage of pyrolysis can be that residues (such as oxidized matrix) remain on the individual carbon fibers [5]. Furthermore, the surface of the carbon fiber may be damaged despite the temperature difference [7]. To remove residues from the fibers, the fibers can be oxidized or washed [5]. The temperature can vary depending on the type of carbon fiber. However, carbon fibers can also be damaged at temperatures below 450 °C. Adjusting the temperatures is essential depending on the type of fiber. For different types of matrices, a lower temperature should be chosen for matrix dissolution. This reduces the likelihood of fiber damage. Exposing the fiber to excessively high temperatures can result in irreversible damage. According to [1], the tensile strength of the fibers after the pyrolysis process is between 50% and 85%, compared to a new fiber. The individual technical parameters, such as pyrolysis temperature, pyrolysis duration, and post-treatment, vary depending on the composite material. The advantage of this is that the technical requirements for the implementation of pyrolysis are rather low, thus offsetting the risk of fiber damage [7].

2.3. Chemical Recycling

In chemical recycling, the composite material is split into its individual components by dissolving it in chemical solutions, such as acids, bases and solvents. Depending on the type of composite material, a different treatment may be necessary. To obtain a better separation between fibers and matrix, the end-of-life components are mechanically shredded

beforehand. This increases the surface area which can react with the chemical solutions. Generally, chemical recycling can be divided into two categories. The first category uses solvents to remove the matrix from the fibers (solvolysis). The second category uses water to remove the matrix from the fibers (hydrolysis). Solvolysis offers a large number of possibilities for the processing of the composite material, due to the availability of different solvents. By combining temperature, pressure and catalysts, the matrix of the fibers can be skillfully solved [8]. The advantage of chemical recycling is that long fibers can be recovered [5]. Furthermore, the recycled fibers showed a tensile strength close to that of new fibers [8]. The solvolysis process can be divided into supercritical, subcritical and near-critical (ambient) processes [1].

2.3.1. Supercritical/Subcritical Solvolyse

The background for the supercritical state is that solvents possess a higher diffusion capability when reaching critical temperatures or pressures. Typical liquids are water and alcohol [5]. According to [5], up to 99% of the resin can be removed from the carbon fibers using alcohol-based solvents. The effect of the solvent on the tensile strength of individual fibers is, according to [5], at least 85% of the tensile strength compared to newly produced fibers.

2.3.2. Ambient Solvolyse

The recycling of composites under mild conditions (lower temperature and pressure) is also possible. According to [5], decomposition of the resin also occurs at temperatures below 100 °C. Using the swelling method according to [9], the surface of the end-of-life components can be enlarged. This method can achieve an epoxy degradation of up to 90% and a tensile strength, compared to the new fiber, of more than 90% [5].

3. Experimental Investigation

In this chapter, the materials used for the experiments will be presented. For the yarn tensile tests, a yarn made of recycled carbon fibers (rCF) was used. The yarn was produced at the institute of textile machinery and high-performance material technology (ITM) of TU Dresden as a prototype, and should illustrate the operation of the production. To produce yarn from rCF, a nonwoven must first be produced from the rCF (carding process). This nonwoven can be produced by the dry-laid method or the wet-laid method [10]. Both methods will be briefly presented here. After the carding process, the nonwoven fabric is stretched and then spun into yarn. Figure 2 represents the simplified process chain for the production of staple fiber yarns.

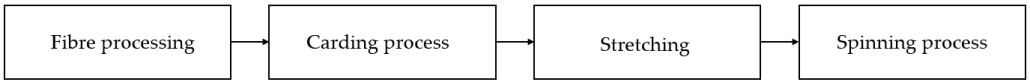

Figure 2. Process chain for the production of fiber yarns.

3.1. Fleece Process

To produce yarns from recycled carbon fibers, the individual short fibers must be processed back into a yarn. One possibility is to process the fibers into nonwovens. Generally, the production of nonwovens can be divided into dry processes, wet processes, and extrusion processes according to [10]. The individual processes can be applied depending on the starting materials. For the production of yarns from recycled fibers, the dry or wet process is best suited. Both processes use short fibers as the starting material. The extrusion process uses granulate as a base and is, therefore, not considered in this article [10]. Figure 3 systematically illustrates the wet and dry fleece process.

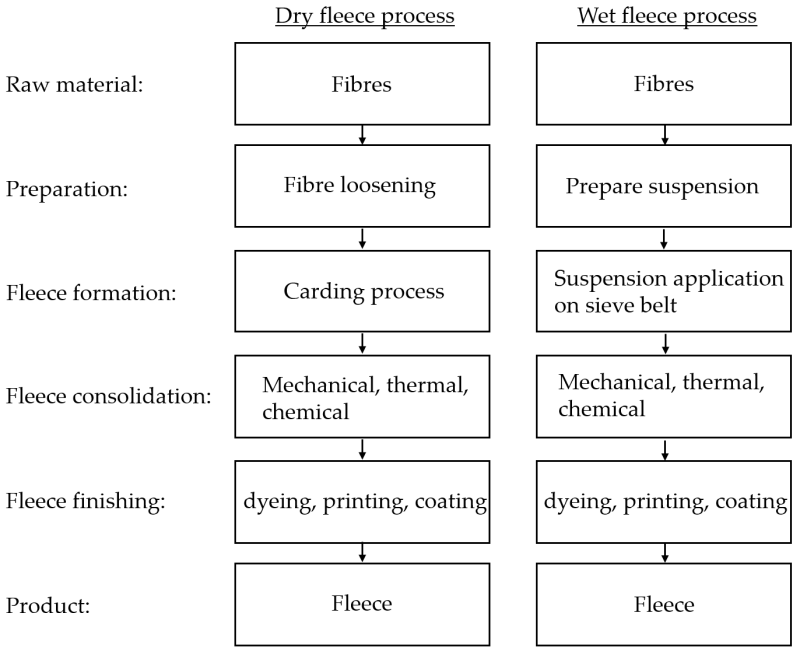

Figure 3. Illustration of the wet and dry fleece process, adapted with permission from Ref. [10]. Copyright 2000, Albrecht Wilhelm, Wiley-VCH GmbH.

3.1.1. Dry Fleece Process

For the production of dry non-woven fabrics, carding is used. This carding creates a fiber-flock or single-fiber fabric. The basic elements of a carding/carding machine consist of a main drum with a cover or a main drum with worker and reversing rolls. The main task of a carding machine is to arrange the randomly presented single-fiber mass into an ordered and desired fiber layer. Furthermore, a carding machine must present the fiber flock per unit of time in terms of length and width. In addition, the fibers are cleaned through the carding process and foreign parts are separated [10]. The main roller is used to process the fibers into a fleece. To form a nonwoven, the individual fiber fleeces are layered one on top of each other. The individual nonwoven formations can refer to [10].

3.1.2. Wet Fleece Process

The advantage of the wet-laid process is that all fibers dispersible in liquids can be deposited in a nonwoven. Characteristic of the wet-laid process is a very good homogeneity of the products and a high level of production performance. The basic production of wet-laids takes place in three steps. In the first step, the fibers are dispersed in water. Through continuous application on a screen belt, the nonwoven is created. The water is filtered simultaneously with the nonwoven formation through the screen belt. In the second step, the nonwoven is dried and rolled up for further processing [10]. Finally, the tapes/nonwovens are combined into a web-band using nozzles or funnels and is stored [11].

3.2. Staple Yarn Production

The produced fiber nonwoven is fed through a stretching process into a stretched web. Several creel webs are combined and stretched into a stretched web. The essential task of the stretching process is to align the fibers in the longitudinal direction. Furthermore, the fluctuations in the diameter of the individual webs are compensated by combining several webs (doubling). By using different feed speeds of the stretched webs, thick and thin spots in the web can be compensated. Another advantage of the stretching process is that the fibers are mixed and the individual webs are dusted [11]. After the stretching process, the final spinning process takes place. In this process, the stretched tape is solidified and stretched to the final yarn fineness using various spinning processes (similar to cotton

yarn production). The solidification of the yarn is achieved by twisting the fiber tape. Another dust removal of the fiber tape also takes place [11].

3.3. Impregnation of the Staple Yarns

The yarns, made of recycled carbon fibers, must be impregnated with a matrix before they are used in construction. The matrix creates an internal bond between the yarns, and thus ensures an ideal power transmission. Three different impregnation processes were selected for the initial test runs. The first impregnation is a duromer, the second one is a thermoplastic and the third matrix a geopolymer.

The duromer was an epoxy resin from Gremolith, based on an epoxy-bisphenol-A-vinylester resin dissolved in styrene. Due to the properties of the resin, this could only be applied manually to the rCF yarn. Mechanical impregnation was not possible as part of the tests. After impregnation, the surface was cured with UV light. The final strength of the impregnated yarn was achieved in the drying oven at 140 °C for 24 h.

The second wetting was performed using a polymer called TECOSIT CC 1000 from CHT Germany GmbH. To wet the individual yarns reproducibly, a wetting machine developed at ITM was used [12]. The machine shown in Figure 4 is used for the production of profiled yarns [13,14]. The wetting, shaping and curing are carried out in a continuous process. In the case of the first wetting experiments and for better comparability, the yarn was wetted and cured in the laboratory unit.

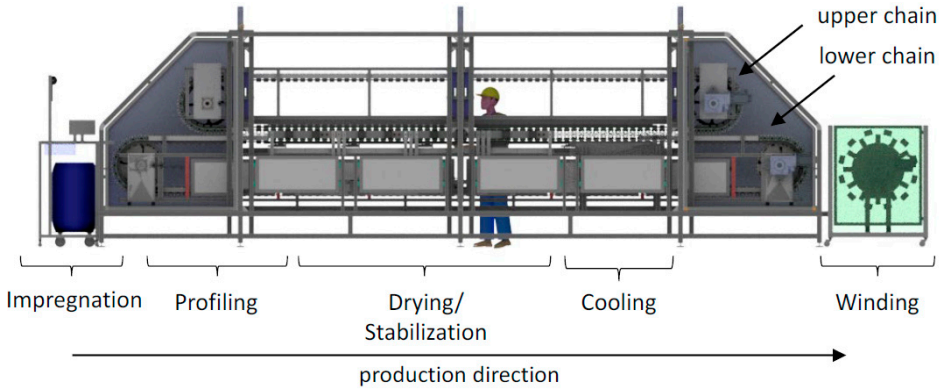

Figure 4. Schematic illustration of the laboratory profiling unit, reprinted from Ref. [13]. Copyright 2022, ITM.

The third impregnation was developed at the Institute of Building Materials (IfB) at the Technical University of Dresden [15].This geopolymer impregnation is based on a mixture of silicon and aluminum oxide. The impregnation of the yarn was also carried out in a laboratory unit specially developed for the continuous process [15]. The yarn is deflected over several rollers to achieve optimal impregnation. Subsequently, the still wet yarn is stretched on a rotating frame and dried for at least 24 h. To accelerate the drying speed, the drying temperature can be increased.

4. Results and Discussion

For the determination of the strengths, single-fiber tensile tests and yarn tensile tests were carried out. The yarn to be examined was produced by the described dry-fleece process.

4.1. Single-Fiber Tensile Tests

The single-fiber tensile tests were conducted using a fiber tensile testing machine, following the ASTM D3822 standard [16]. Fibers were taken directly from the yarn for sample preparation and inserted into the testing machine. Subsequently, the individual 25 mm long fibers were loaded until failure. The loading speed was set to 1 mm/min. A total of 20 individual fibers were examined. In addition to the breaking force, other

material properties such as diameter and Young's modulus of the fiber were determined. The average fiber diameter was 5.41 µm, and the average Young's modulus was 229.54 GPa. The stress–strain curves are presented in Figure 5.

The average fracture stress is 3.34 GPa of 20 samples. The stress–strain diagram also shows that the fiber's tensile stresses increase linearly. The failure of the fibers occurred suddenly and without prior notice. The strain of the fibers studied ranges from 0.87% to 2.02%. The average maximum strain of the fibers is 1.39%. All results are shown in Table 1. The exact material-specific properties (such as tensile strength) of the fibers, before the recycling process, could not be definitively determined. However, due to their similar diameter, it is assumed that they belong to the same type of fibers. In comparison, virgin carbon fibers with a diameter of 5 µm have a tensile strength ranging from 5.9 GPa to 6.0 Gpa, and a Young's modulus between 280 Gpa and 290 Gpa. For a fiber diameter of 7.0 µm, the tensile strength ranges from 4.1 Gpa to 5.1 Gpa [17]. Therefore, this would result in a strength loss of up to 10%, compared to a new fiber. According to [18], the recycled fibers achieved a tensile strength between 72% and 94% compared to the new fibers. In the experiments conducted by [19], the tensile strengths of the recycled fibers were found to be similar to those of the new fibers.

From the diagram in Figure 5, it is evident that there is a large fluctuation in the individual breaking loads of the single-fiber test. The average fiber rupture stress is 3.34 GPa. However, the arithmetic mean is of limited use due to the variation of the 20 individual values. The correlation coefficient of Figure 5 is 0.99. The standard deviations of the conducted experiments are listed in the following Table 2.

The large fluctuation in the breaking loads could be attributed to the manufacturing process, during which the yarns are subjected to different levels of stress during the dry-laid process, stretching and spinning. This caused defects in the individual fibers; therefore, they failed at different load levels. Since all the diameters of the fiber range between 5.14 µm and 5.82 µm, all individual values can be compared. The yarns used in these experiments were produced in a prototype manner. The individual fibers were extracted from the yarn and examined. Due to the manufacturing process using the dry-web method, the fibers experienced varying degrees of mechanical stress. Therefore, the fluctuations in the tensile strengths of the individual fibers can be attributed to this mechanical strain.

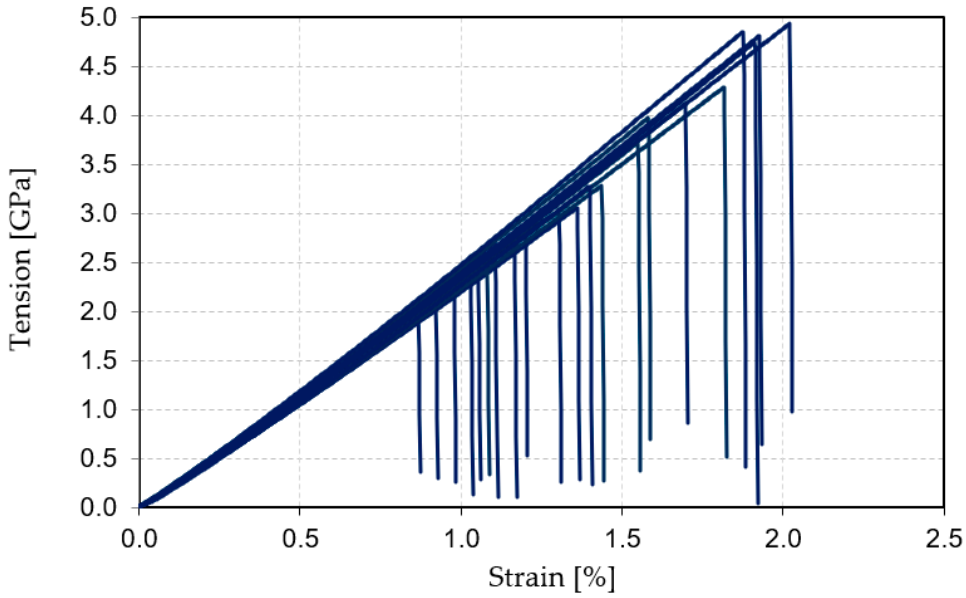

Figure 5. Tension–strain diagram of single-fiber tensile tests.

Table 1. Results of the single-fiber tensile tests.

Number	Breaking Stress [cN]	Fiber Diameter [µm]	Tension [GPa]	Strain [%]	Young's Modulus [GPa]
1	7.5	5.82	2.82	1.20	228.21
2	7.07	5.47	3.01	1.30	226.90
3	6.08	5.35	2.71	1.11	238.94
4	4.46	5.23	2.07	0.87	236.20
5	7.46	5.57	3.06	1.36	215.68
6	5.09	5.32	2.29	0.98	230.36
7	6.52	5.48	2.76	1.17	231.08
8	5.64	5.37	2.49	1.03	236.80
9	5.59	5.52	2.34	1.05	217.04
10	5.88	5.47	2.50	1.08	225.64
11	12.25	5.62	4.94	2.02	229.31
12	8.52	5.40	3.73	1.55	228.97
13	7.39	5.35	3.28	1.44	218.67
14	8.26	5.15	3.97	1.58	238.33
15	10.07	5.47	4.29	1.82	223.47
16	10.09	5.58	4.12	1.70	230.46
17	7.00	5.21	3.28	1.40	224.39
18	10.32	5.20	4.85	1.88	243.65
19	10.60	5.30	4.81	1.93	235.01
20	9.86	5.14	4.75	1.91	232.03

Table 2. Standard deviation of the single-fibre tensile tests.

	Breaking Stress [cN]	Fiber Diameter [µm]	Tension [GPa]	Strain [%]	Young's Modulus [GPa]
Standard deviation	2.14	0.17	0.95	0.36	7.46

4.2. Yarn Tensile Tests

The tensile tests were carried out in accordance with [20,21]. The individual yarns were cut to a length of 400 mm. A total of five yarns were tested for each type of wetting. For testing, the yarns were glued to aluminum sheets. The aluminum sheets served to introduce the load. According to [20], the free length of the yarns must not be less than 200 mm. The loading speed of the yarns during the tensile test was set to 3 mm/min. The measurement of the strain was carried out with a MultiXtens extensometer. The results of the yarn tests are presented in Figure 6.

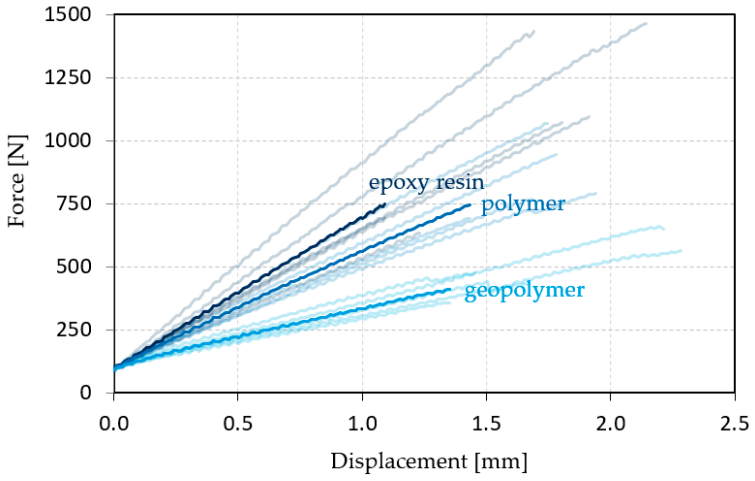

Figure 6. Force-displacement diagram of the fiber tensile tests.

The rupture loads, as well as fracture behavior, vary depending on the impregnation. The average rupture load of the epoxy resin-impregnated yarn is 1.5 kN. For the polymer-impregnated yarns, failure occurred at 1.1 kN and for the geopolymer-impregnated yarn at 0.65 kN. The individual test curves are shown in the diagram. All results were shown in Table 3. All yarns failed between the load application points within the free span. Therefore, the tests are valid according to [20] and can be assigned to the type of failure I. In all tests, the yarn failed at the point with the smallest diameter. Direct determination of the stress–strain curve is not derivable. The background is that due to the manufacturing process of the yarn, the diameter is not constant. This has the consequence that an exact yarn tension cannot be determined. The average manually measured yarn diameter is approximately 1.5 mm. Since all yarns are made from a continuous yarn, a first comparison of the values is nevertheless possible.

Table 3. Results of the yarn tensile tests.

Number	Impregnation [-]	Force [N]	Displacement [mm]
1	duromer	649.17	1.09
2	duromer	1433.65	1.69
3	duromer	1463.64	2.14
4	duromer	1093.33	1.91
5	duromer	1073.05	1.80
6	polymer	1069.04	1.74
7	polymer	943.72	1.78
8	polymer	769.52	1.68
9	polymer	792.07	1.94
10	polymer	691.51	1.43
11	geopolymer	430.39	1.66
12	geopolymer	356.55	1.35
13	geopolymer	649.55	2.21
14	geopolymer	563.99	2.28
15	geopolymer	477.57	1.45

As was already the case with the single-fiber tests, fluctuations in the individual breaking loads could also be observed in the fiber tests. In the case of yarns impregnated with an epoxy resin, the tensile forces range between 649.17 N and 1463.64 N. The displacement ranges were between 1.09 mm and 2.14 mm. The yarns impregnated with a polymer were able to withstand forces between 691.51 N and 1069.04 N before failure occurred. The measured displacement ranged from 1.43 mm to 1.94 mm for the yarns impregnated with geopolymer. The breaking load for these yarns ranged between 430.39 N and 649.55 N. The displacement varied from 1.35 mm to 2.28 mm. The correlation coefficient of Figure 6 is 0.34. The standard deviation of the conducted experiments is listed in the following Table 4.

Table 4. Standard deviation of the yarn tensile tests.

	Force [N]	Displacement [mm]
Standard deviation	72.03	0.06

The fluctuations can also be attributed to the production of the yarn at this point. Due to varying diameters, the yarns failed under different loads. The curves shown in the diagram also reveal that the yarns impregnated with epoxy resin have the least amount of displacement. Thus, the elongation of the yarns with epoxy resin, of the three examined, is the lowest. This leads to the result that the epoxy resin-impregnated yarns have the highest stiffness of the examined impregnations.

The tensile strength of the yarns is directly proportional to the saturation of the yarn. The better the saturation of the yarns, the better the occurring tensile forces can be dissipated over the individual fibers. As can be seen in Figure 7 (left), the non-fully saturated yarn is stretched. The yarn saturated with geopolymer also has the lowest tensile strength and the highest machine travel. Based on the behavior during the tests (core extraction) and the measured values, it can be inferred that the impregnation of the yarn was not complete. The larger machine path compared to the epoxy-impregnated yarn can be explained by the yarn itself. In the case of an unimpregnated core, the yarn is stretched again. This increases the elongation or machine path. As the tensile forces are only absorbed by the outer impregnated yarn area, a complete activation of the yarn is not possible.

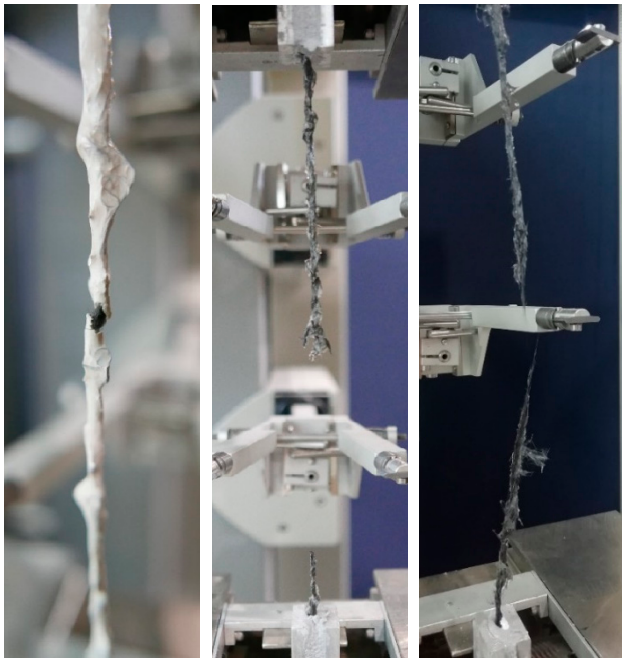

Figure 7. Failed yarn impregnated with geopolymer (**left**), thermoplastic (**center**) and epoxy resin (**right**).

The yarn impregnated with the polymer matrix achieves a similarly high-tensile load compared to the yarn impregnated with epoxy resin. Only machine path or elongation is larger in direct comparison to the yarn impregnated with epoxy resin. A direct comparison with other yarns made from recycled carbon fibers is only partially possible. Recent studies have shown that the processability of yarns made from recycled carbon fibers and thermoplastic fibers yielded better results [22]. In [23], a yarn made from recycled carbon and thermoplastic fibers was produced and examined. According to [23], the tensile strength of the hybrid yarn is approximately 1.4 GPa. Both the hybrid yarn and the yarn examined in the experiments were produced in a similar manner at ITM. With an average yarn diameter of 1.5 mm, this results in a tensile strength of 0.43 GPa.

5. Conclusions

The information presented in this article is intended to provide an initial overview of the recycling of carbon fibers and their reuse as reinforcement. Furthermore, initial experiments with recycled fibers were carried out. The influence on the tensile strength of the yarns in relation to the impregnation or the matrix could also be demonstrated. For further investigations, the use of an epoxy resin or a vinyl-ester resin is recommended. However, it is important to differentiate the use of carbon reinforcements based on their intended applications. When using epoxy resin, subsequent deformation of the yarn is no longer possible once the resin has fully cured. However, yarns impregnated with polymers allow for post-curing thermal reshaping even after the matrix has fully hardened.

Furthermore, the fluctuation of the tensile strengths of the individual fibers is a factor to be taken into consideration during the recycling process. The production of yarns from recycled carbon fibers is technically feasible. The impregnation process can be carried out differently depending on the type of impregnation. This can be explained with different physical properties. In the next step, rebar and yarns were produced from the recycled fibers and examined.

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