

Viability of recycled fibres extracted from EoL composites

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Abstract. Recycling thermoset-based composites is a technological and economic challenge. The predominating recycling methods (mechanical recycling and pyrolysis) downgrade the fibre properties (length, strength) and are energy-intensive. To avoid downcycling and to ensure economic feasibility, technological solutions need to be developed to enable recycling of long reinforcing fibres that can also be used in demanding applications. Thermochemical recycling, where reinforcing fibres are extracted from the depolymerized matrix, can be considered as an advanced option for recovery of higher quality fibres from EoL composites. Additional steps are required to use these fibres in new composite structures. Thermochemical recycling removes the sizing from the fibre surface making it difficult to handle and resulting in poor fibre-matrix compatibility especially in the case of glass fibres. In this study, we discuss the re-sizing of recycled fibres. We have focused on fibres extracted from glass fibre reinforced composites (GFRPs) from EoL wind turbine blades and carbon fibre reinforced composites (CFRPs) collected from aeronautics industry. We demonstrate the steps for a batch re-sizing process and study the effects of the re-sizing on the properties of composites made of recycled fibres. The interfacial properties are analysed with micro-mechanical testing using the microbond method – both for thermoset and thermoplastic composites – and with tensile tests for thermoplastic composites. Re-sizing significantly eases the handling of the fibres, but the interfacial compatibility is improved only in the case of GFRPs as thermochemically recycled carbon fibres (rCF) exhibit very good interfacial performance even without sizing. This study highlights that with proper recovery and resizing processes, recycled fibres can be viable feedstocks for various applications and – with future improvements on the fibre recovery methods – even approach the applicability of virgin fibres.

1. Introduction

The volume of European composite production is the largest in short (< 2 mm) fibre reinforced composites, which have lower requirements for (mechanical) performance than long (2-5 mm) and endless (> 50 mm) fibre reinforced composites. With proper recycling, recycled composites can represent a valuable feedstock for short and long fibre reinforced composites with high part and production volumes and less restrictive performance requirements. Although this inherently includes some degree of down cycling, and cannot be therefore considered fully circular, much of the technical and economic added value of the material can be preserved.

One of the first hindrances of the circular economy trend is the poor consumer acceptance for the use of recycled composite feedstock. Mitigation strategies could include standardized verification processes and open availability of data, among others. Naturally, it also requires sufficient capacity of reliable and reproducible recycling technologies and reprocessing processes to ensure a steady supply of the material.



This ties into a lack of systemic value chains, actors within these value chains and circular economy business models, which slows down recycling in composite industry. The integration of different stakeholders into circular economy is poor partly due to their fragmented economic and geographical nature. This leads to poor communication between the stakeholders and to a less than unified front, making it more difficult to affect policymakers' decisions and to achieve rational changes in regulatory and legal frameworks. The circular economy business models are traditionally based on 'pushing' the recycled materials towards the demand side in the value chain. The FiberEU project [1] promoted the implementation of a model where the requirements and specifications of the materials to be recycled come from the demand side generating a 'pull' for the secondary raw materials. This model would better ensure the characteristics and functionalities of the recycled material are suitable for high added value products. This kind of demand-driven circular economy requires strong communication between the actors within the value chain and possible new innovations to support it, e.g., through novel ICT solutions. If a strong demand from the end-users (customers) for sustainable composites is realized, we believe that the probability of a demand-driven circular economy scenario might become reality as is gradually happening in the plastics industry. Here, the technological and commercial viability of fibres extracted from EoL composite structures is discussed based on both previous and on-going research efforts on the topic.

2. Composite waste streams and waste management

European composite industry is mostly producing glass fibre reinforced composites (GFRP, > 95%) the rest being mainly carbon and natural fibres reinforced composites (CFRP and NFRP, respectively). The volumes are divided roughly between short fibre reinforced thermoplastics (51%) and other thermoplastic and thermoset composites [2]. Thermoplastic composites are mostly used in the transport and electrical application sectors of industry. Thermoset composites, which are typically reinforced with long or endless fibres, cover a share of approx. 45% of the European market excluding the CFRPs [2] and are prominently used in construction, transportation and electrical sector. The CFRP composites production volume has increased from 36.000 tonnes in 2017 to 52.000 tonnes in 2022 [2] but still remains less than 2% of the total European composite production.

Compared to other waste streams, the composite waste stream is relatively small. The composite waste stream is generally also quite unstable in composition and properties, unpredictable in volumes and geographically dispersed. Due to increasingly strict and varied regulations on waste collection and management, international composite waste collection does not seem practical. Instead, local cooperation of waste management companies could be a more realistic scenario to ensure higher collection volumes. The local collaboration and application of a demand driven circular economy approach could help in this attempt. A revision of the EU's End-of-Waste criteria to enable the easier use of secondary raw materials would also promote the implementation of robust composite recycling systems.

Current composite waste management practices vary considerably in Europe. Landfilling and incineration remain as the most typical routes while mechanical recycling is also used in smaller extent. Using the classification of European Waste Framework (EWF) directive, the use of composites in cement kilns is located somewhere between incineration and recycling. This 'recycling' method is compliant with EU's legislation and requires relatively small investments to incorporate into existing systems, thus offering a tempting choice for the composite waste management. For example, in Finland where the wind power infrastructure is fairly new and still increasing and where the local composite industry is small in production volume, the use of composite waste in cement industry was recently identified as the only feasible route for composite recycling [3]. In this process, the resin combusts providing energy for the cement manufacturing process while the reinforcing fibres are left to the cement as fillers. This can lead to some reductions in the CO₂ emissions of the final cement product. However, the use of composite waste in cement process fuel is technically restricted and composites containing E-glass are shown to have a negative effect on the cement properties. All in all, the technical added value of composites is fully lost in this scheme of recycling.

The composite waste can be divided roughly in three categories: small components that typically end up in municipal waste streams (e.g., electrical components such as switches or housings, sport equipment, etc.), large infrastructures – e.g., wind blades or boats that are dismantled in a controlled process – and industrial composite production waste. These waste streams have very different properties and potential for recycling. While the first group is a very heterogeneous fraction of the composite waste and has the least potential to be recycled without significant improvements to sorting and collection, it is also the smallest fraction. The large structures have better – if not always fully – known composition simplifying the recycling process. Considering the production volumes and the most typical composite applications, the volume of this fraction is large. It has estimated that the annual amount of End-of-Life (EoL) wind blades increases to 50.000 tons in Europe by 2030 [4] and the amount of disposed leisure boats is approx. 10.000 tons [5]. However, these composites have undergone ageing during their use and the residual material properties are poorly known. A composite components' lifetime is very long, typically 10 to 30 years, and they are typically used in environments where they are exposed to varying external stresses. Furthermore, the waste stream volume and geographic locations are somewhat unpredictable. The composition of industrial process waste is well-known, the material can be considered pristine, and its volume and location are the least problematic to predict. Further, the amount of the process waste is far from negligible, annually up to 50.000 tons [6]. Naturally, the reuse of in-house collected production waste should be promoted to reduce the amount of externally managed process waste to minimum. In cases where in-house use is not possible and established procedural framework should be established to avoid recycling gaps.

3. Composite recycling and reprocessing

The composite recycling technologies can be classified in three categories: mechanical, thermal and chemical recycling. Mechanical recycling (cutting, shredding, sieving) results in recyclates that have a size distribution dependent on the process parameters ranging from shredding powder into larger particles. The composition of individual particles resembles the original composition of the composite, although variation has been found to exist between different size fractions. The use of the particles and dust as fillers or reinforcement is somewhat limited due to low cost of virgin feedstock, processing issues (e.g., the increase in resin viscosity when dust is added) and decrease in the mechanical properties of the final composite parts (poor compatibility between the recyclates and the virgin matrix). However, for thermoplastic composites mechanical recycling is the most straightforward process as the recyclates can be used as such, together with virgin raw materials. The application of recycled feedstock is mostly in short fibre reinforced composites but especially the thermoplastic recyclates can be also used in long fibre reinforced ones. The smallest fractions are typically incinerated. Electrofragmentation can be classified as a mechanical recycling method and results in recyclates with higher fibre fraction and longer fibre length [7]. Electrofragmentation is actively studied in the RECREATE project [8].

Thermal recycling, i.e. pyrolysis, fluidized-bed pyrolysis or microwave-assisted pyrolysis, can be positioned in the EWF directive somewhere between recycling and recovery, as the resin part is lost from the material circulation when it decomposes to volatile molecules during the process. However, the fibres are made available for further use. The process leaves char on the fibre surfaces that might negatively affect the final properties of a product made with the secondary feedstock. Thus, the char is typically removed in a subsequent process, which has the unwanted side effect of degrading the fibre properties. Typical reduction in fibre strength due to thermal recycling is 50-90% which is a significant feasibility challenge. With process optimization, CFRP pyrolysis can result in remarkably lower (only 5%) strength reduction in rCF compared to virgin [9, 10]. Thermal recycling is more energy intensive than mechanical recycling, further reducing its feasibility for glass fibres (GF). The volatilized resin can, however, be used as energy input to the system somewhat reducing the costs. Thermal recycling is equally applicable to thermoset and thermoplastic composites and the process is quite insensitive to material composition. The purified fibres can be potentially used in long or endless fibre reinforced composites with higher added value, as also demonstrated in the FibreEUse project. However, the

process is more effective if the density of the recycled material is low, i.e., the composite has been shredded to small particles, which is even a technical requirement in microwave-assisted pyrolysis.

Chemical recycling is based on solvolysis, i.e., degrading the resin into monomers using a solvent, most typically water, a catalyst and increased temperature lower than in thermal methods. No char is formed or left on the fibres. The reactors are expensive, and the process is more sensitive to the composition of the recycled material than in other methods. Thus, chemical recycling is yet to become a larger scale possibility for composite recycling. However, if it is combined with a thermal recycling process, additional value can be gained. Successful pilots for these thermochemical processes are already patented (EP 3114191, 2018 by Korec srl) and implemented in industry. In Korec's process, a controlled CO₂ atmosphere is used to enable the collection of both clean fibres and the resin as a liquid fraction applicable to further use as a thermoset feedstock, for which the method has been originally developed. This pilot system was also applied in the FiberEUse project.

After shredding the composite into smaller particles or extracting the fibres for further use, the secondary raw materials must be reprocessed. When it comes to mechanically recycled materials, the reprocessing might not differ significantly from the use of virgin feedstock, i.e., compounding thermoplastic granulates or mixing fillers and thermoset resins. However, the relatively clean fibres from thermal or chemical recycling are not applicable as such. The recycling process removes the original surface treatment, sizing, that is applied by the fibre manufacturer to ensure the processability and compatibility of the fibres with the matrix. This part of the value chain is absent from the value chain of traditional (virgin) reinforcements and a natural actor for the part is missing. The resizing (i.e., sizing the recycled fibres) falls between the actor specialized in the recycling technology and the actor compounding the recycled fibres with a polymer matrix. Neither of these actors generally has the infrastructure or expertise to apply the sizing of the fibres, both of which exist practically exclusively with fibre manufacturers.

4. Resizing recycled fibres

In many composite recycling studies, where extracted fibres are discussed, the surface treatments of the fibres are mentioned being highly important for the properties of the final composite, but the topic is not addressed further [11]. In the FiberEUse project, the aspects of the resizing of recycled fibres were studied for different kind of waste streams (GFRPs from EoL wind turbine blades and CFRPs collected from aeronautical industry) and output compositions (short fibre reinforced thermoplastic composites and long fibre reinforced thermoset composites, for the latter the results available in [12]). In this chapter, the resizing process, and the reprocessing of resized fibres for the FiberEUse demo cases are discussed and the results for the use case validation are reported. This is intended as a practical example of the multitude of scientific and process related aspects of resizing, and, perhaps more importantly, of functional products from the recovered fibres.

4.1. *Materials and methods*

The composites were shredded, recycled by the thermochemical process patented by Korec, and resized in a batch process with a chemistry compatible with the matrix material. The resulting fibres were clean, and their mechanical properties are reported in [10].

In general, sizing is a water solution comprising mainly film former and a coupling agent, the latter of which is not used for carbon fibres. These represent the main components for improving interfacial properties. Other possible sizing components are lubricants, antistatic agents, wetting agents and cross-linking agents, which are often included in a commercial sizing to improve, for example, storage properties, handling, and resin flow [13]. A batch process for the resizing where fibres are immersed in the sizing solution was considered the most realistic for the resizing at laboratory scale. The case is very different from continuous virgin fibres treated in very fast roll-to-roll processes. Here, the preceding thermal recycling processes as well as the following compounding the resized fibres with a thermoplastic matrix are also batch processes. However, in other cases, such as reinforcement non-woven preparation, or a fibre spray-up process, the resizing could be incorporated into the process together with a possible

binder or as the binder. This would enable more industrial scale options for the use of recovered fibres in thermoset applications and such options are currently explored in the RECREATE project.

In FiberEUse the focus was primarily on improving short fibre reinforced thermoplastic compounds with a simple sizing formulation. Detailed formulations are given in [14] together with the process parameters. The following aspects were studied when optimizing the sizing composition and process: the total solid content of the sizing solution and rinsing after the immersion in the sizing solution. In general, a thin sizing layer and lack of large fibre clusters are preferable for mechanical performance, whereas a higher packing density and fibre agglomeration are advantageous for the compounding process. The resizing process was optimized against these aspects. The resized fibres were analysed with Fourier transformation infrared spectroscopy (FTIR), thermogravimetric analysis (TGA) and scanning electron microscopy (SEM). The resized fibres were compounded with copo-polypropylene (copo-PP) and polyamide 6 (PA, only rCF) thermoplastic matrices into tensile test specimens and tested (ISO 1172 (method A) standard). The fibre fraction of the compounds was 20 wt% and verified by the burning-off method (ISO 1172 (method A)). Reference compounds were prepared with recycled fibres without resizing.

After the selection of the most optimal resizing process, larger batches of thermoplastic composites for the use case demonstrators of the FiberEUse project were produced. The same copo-PP and PA matrices were used but in addition, a homo-PP was used with rCFs. The compounding process was carried out with a twin-screw extruder. For more details about the compounding, see [12]. The final fibre fractions of the compounds were verified to be within ± 2 wt% from the intended value (20 wt% or 30 wt%) via TGA. The mechanical properties of the compounds were characterized by tensile tests to compare the influence of sizing (fibres with and without sizing) and the influence of the origin of the fibre (recycling process). The comparison was done to material specifications used for component design in the automotive sector. After tensile testing, composite fracture surfaces were studied with SEM. Finally, the use case demonstrators were manufactured, and the components were tested with use case specific validation procedures.

4.2. Optimizing the sizing concentration

Based on the FTIR analysis of the resized rGF, the surfaces contained both the film former and the coupling agent. FTIR and TGA results both indicated that higher total solid content (concentration) in the solution also increased the amount of sizing deposited on the fibres. Too high silane concentrations led to self-polymerization of the silane – instead of interaction with the fibre surface – and the silane was mostly removed by the rinsing. The SEM analysis revealed a slightly uneven sizing layer but differences between different concentrations were not observed. In the case of recycled carbon fibres (rCF), the sizing layer was more homogeneous, smoothing out also the original surface grooves of the fibres. TGA revealed that the average sizing layer thickness followed the resizing solution concentration if the fibres were rinsed after the immersion to remove excess sizing. Without rinsing the amount of sizing in the fibres was higher and random. More detailed characterization of the resized fibres can be found from [12, 14]. Table 1 presents collected key results from microscale interfacial testing (microbond) performed using these sizing formulations. A detailed description of the experimental setup and analysis of the results can be found in [15].

The mechanical properties of the rGF reinforced PP composites were improved after resizing and SEM analysis revealed better fibre-matrix contact for the resized rGF. As expected, no further improvement was observed with increasing solution concentration and the maximum values were reached at a range of 0.5 wt% (tensile modulus) to 2 wt% (tensile strength). For the rCF reinforced PP and PA composites, no significant difference was observed in the tensile properties as a function of sizing concentration indicating that after thermochemical recycling the surface of recycled carbon fibres is also by itself compatible with thermoplastic matrices. However, the fractography revealed that the distribution of the fibres was improved with resizing. Further, the binding and packing of the resized fibres are highly important for the handleability and industrially feasible processing of the recycled fibres. The optimal resizing for rGF was concluded to be 1 wt% total solid content without rinsing and

for rCF 5 wt% total solid content without rinsing. In the FiberEUse project, this aspect was more critical than the exact mechanical properties, and therefore a relatively high sizing concentration was chosen as ‘optimal’.

Table 1. Influence of the sizing formulations on the interfacial strength of various fibre-matrix combinations. Data partially from optimisation trials and partially collected from [16,17].

Fibre (+sizing)	Matrix	IFSS [MPa]	stdev [MPa]
rGF/GF		35.2	8.9
rGF (1 w%, m-PP FF [†])	Epoxy	31.7	2.1
GF (1 w%, EP FF [‡])		58.2	3.2
rGF (5 w%, m-PP FF)		28.9	2.0
rCF		43.6	2.5
rCF (1 w%, m-PP FF)		25.0	2.2
CF	PP	8.4	4.0
rCF		20.1	9.9
rCF (1 w%, m-PP FF)		17.3	6.1
rCF (5 w%, m-PP FF)		17.3	5.0
rCF	PA6	66.5	6.0

[†] maleated polypropylene film former

[‡] epoxy film former

4.3. Compounding secondary raw materials for use cases

For the compounding experiments and the FiberEUse project use case demonstrators, larger batches of resized fibres were prepared. The fibre fraction of the compounds was also different from the resizing process optimization step. Thus, the results of the resizing process optimization study and the compounding study are not directly comparable.

The modulus of the rGF-PP composite was improved by 38% and the tensile strength by 58% via resizing and better fibre-matrix adhesion was evident from fracture surfaces. However, the properties were lower than the corresponding automotive reference specifications and the main issue was assumed to be the cohesive properties of the recycled glass fibres [18].

The resizing increased the modulus of the rCF-PA compounds by 100% and the tensile strength by 45%. An even distribution and good fibre-matrix contact was seen in SEM images. The resized composites achieved almost the required level for the mechanical properties, the strength being only 8% below the automotive reference material.

In homo-PP matrix, the resizing of rCF increased the modulus by 37% and the tensile strength by 40% and, similarly to rCF-PA compounds, resizing improved the compatibility of the fibres in the matrix. However, the dispersion of the fibres was good even without the sizing. Compared with automotive reference, the material was stiffer (23% higher modulus) but less strong (13% lower tensile strength). The results were very similar with copo-PP matrix. Based on the results and the experience gathered during the compounding trials, it was clear that the resizing improves especially the processability of the rCF compounds having simultaneously positive effect on the mechanical properties.

Based on the compounding trials, small changes were made to the formulation of the materials, namely the fibre fraction, and the design of the use case demonstrators. However, the required changes were relatively small and no new design concepts or other significant changes were needed.

4.4. Verification of the demonstrator properties

Three different automotive use case demonstrators were produced from the recycled fibres. Details of these demonstrators can be found from [19]. A **cowl top** is a stiff and strong component that should break after absorbing a certain amount of energy at dynamic conditions and is produced by injection moulding. The current design is produced with short GF (30 wt%) reinforced PP and the new design with rGF was based on similar composition. In the validation phase, the demonstrator must withstand specific impact loads without breaks and cracks, weathering tests without deformations and peeling off and prove sufficient thermal stability. All criteria were passed.

The second demonstrator was a **pedal bracket**, which is a security part with high structural requirements both in stiffness and strength. The reference structure is manufactured by injection moulding with short GF reinforced PA (fibre fraction 40 wt%) and the secondary raw material design was based on PA and 20 wt% rCF. In the validation test, the part was subjected to varying loads applied to different areas of the brake and gas pedals. To pass the test, the applied load must induce deformations less than a vehicle manufacturer defined requirement. Also, the component must withstand loads up to 3000 N without any break at extreme temperatures (80 °C and -35 °C). The new bracket manufactured with recycled fibres fulfilled the requirements.

A **front-end carrier**, which was the third demonstrator, must support load and prevent torsions. The reference structure is manufactured by injection moulding with long GF reinforced PP (fibre fraction 40 wt%) and the secondary raw material design was based on PP and 20 wt% rCF. The prototype must fulfil static load cases with combined compression and a traction load up to 2300 N loading without any breaks and achieving a specific stiffness. The front-end carrier produced with recycled feedstock met the requirements and was lighter than the original version.

5. Conclusions

To ensure that circular economy will be viable scheme for composite materials, the secondary composite feedstock produced from recycled materials must be able to compete with virgin raw materials in environmental, economic, and technical aspects. Further, each step of the recycling and reprocessing value chain must be covered by an industrial actor with suitable expertise. Currently, this is not yet the case for thermal recycling of composites, where the reinforcing fibres are extracted for further use. Especially, resizing (sizing of recycled fibres) is shown to be a necessary step of the reprocessing route to achieve recycled fibres that can be processed into high performance composites, but currently the industry is lacking actors that could combine it either with fibre extraction, compounding (thermoplastic composites) or thermoset composite manufacturing. Possible options for fibre recovery incorporated resizing include reinforcement mats, non-wovens, recycled fibre prepregs or other comparable preforms. At least for thermochemically recovered carbon fibres with high mechanical properties, a polymer binder can replace majority of the functions of the sizing enabling straightforward, cost-effective, and safe approaches with few process steps from composite waste to new feedstock. Similar approaches could be available for extracted glass fibres with the added step of resizing. These aspects must be studied further, as is done in RECREATE [8] and DeremCo [20] projects.

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