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Review

A Review on the Recycling Technologies of Fibre-Reinforced Plastic (FRP) Materials Used in Industrial Fields

Dario De Fazio, Luca Boccarusso, Antonio Formisano, Antonio Viscusi * and Massimo Durante

Department of Chemical, Materials and Production Engineering (DICMaPI), University of Naples Federico II, P.le V. Tecchio, 80, 80125 Naples, Italy; dario.defazio@unina.it (D.D.F.); luca.boccarusso@unina.it (L.B.); aformisa@unina.it (A.F.); mdurante@unina.it (M.D.)
* Correspondence: antonio.viscusi@unina.it

Abstract: Fibre-reinforced plastic (FRP) materials are attracting growing interest because of their high specific mechanical properties. These characteristics, in addition to a high level of tailorability and design of freedom, make them attractive for marine, aerospace, automotive, sports and energy applications. However, the large use of this class of material dramatically increases the amount of waste that derives from end-of-life products and offcuts generated during the manufacturing processes. In this context, especially when thermosetting matrices are considered, the need to deeply study the recycling process of FRPs is an open topic both in academic and industrial research. This review aims to present the current state of the art of the most affirmed recycling technologies used for polymeric composites commonly used in industrial applications, such as carbon and glass FRPs. Each recycling method (i.e., chemical, thermal and mechanical) was analysed in terms of technological solutions and process parameters required for matrix dissolution and fibre recovery, showing their advantages, drawbacks, applications and properties of the recycled composites. Therefore, the aim of this review is to offer an extensive overview of the recycling process of polymeric composite materials, which is useful to academic and industrial researchers that work on this topic.

Keywords: recycling; composite materials; waste management; chemical recycling; thermal recycling; mechanical recycling

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1. Introduction

Carbon fibre-reinforced polymers (CFRPs) and glass fibre-reinforced plastics (GFRPs) are attracting significant interest in our life because of their interesting mechanical properties and low weight. The mechanical properties that characterise this category of materials, in addition to a high level of tailorability and design freedom, make them very attractive in various application fields, such as aerospace, automotive, marine and energy [1–3]. Among all composite materials, CFRP composites play a key role as carbon fibres (CFs) are stronger and lighter than other fibres, and their overall composite structure is characterised by additional advantages, such as the capability to maintain their mechanical properties at high temperatures and show high durability in aggressive and corrosive environments [2,4–7].

All these aspects have raised the demand for composite materials over the last 40 years in existing and emerging industries, and during these years, despite the COVID pandemic, the demand for CFRPs is growing at a current rate of 11% per year because of the increasing need for lightweight materials. It was estimated that CF demand was around 120 kTons during the present year [8–10]. Composite material demand is also rising over time due to new targets for a reduction of CO_2 emissions and the need for structural components that conciliate both light weight and high strength. It is possible to observe from Figure 1 that the global demand for CFRP materials increased from almost 68 kTons in 2010 to around 170 kTons in 2020 [11–13]. A further increase is expected in the near future as it is estimated that the demand for CFRP composites will reach almost 190 kTons in 2050 [11–13].

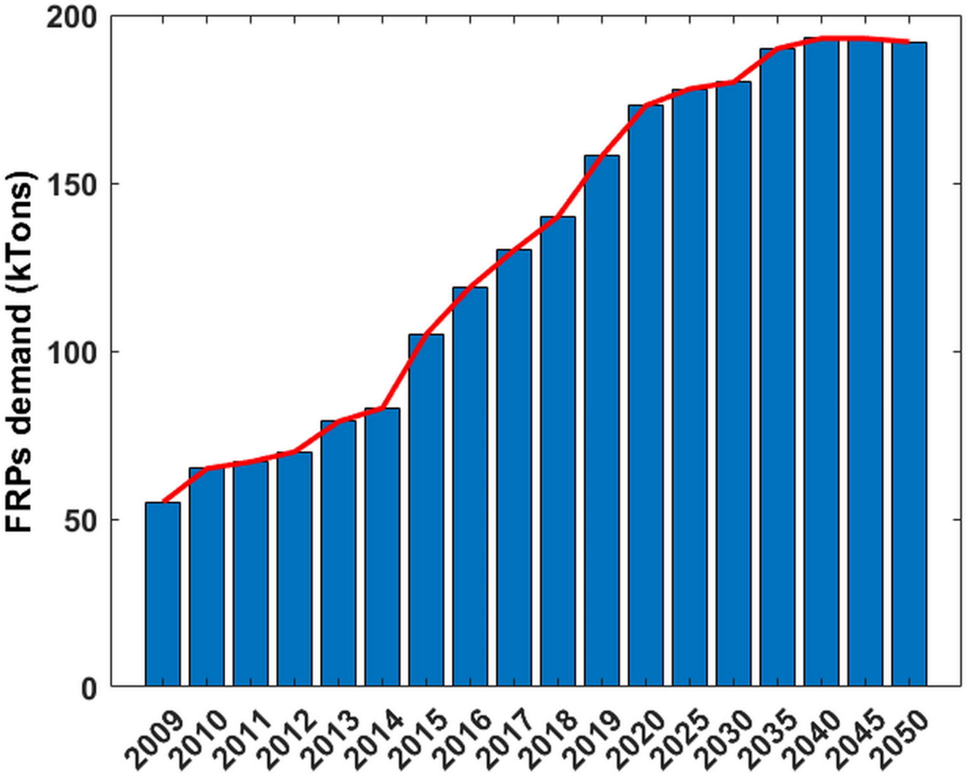


Figure 1. CFRP demand over the years [11,12].

The demand for composite materials is interestingly growing in wind turbine blades and marine applications, testifying a volume utilisation in wind blades of almost 34 kTons in comparison with use in aircraft applications of almost 33 kTons [11,14,15]. The volume utilisation of CF composite materials in marine applications is still reduced (around 3 kTons in 2018) when compared to wind and aeronautic sectors, but it is estimated to grow in the imminent future [11,14,15].

As well as increasing demand for CFRPs in some industrial sectors, further growing demand for GFRP composite materials is estimated as they represent almost the total portion of FRP composites used in the industrial world. According to the European Composite Industry Association (EuCia), despite the COVID-19 pandemic that affected industrial production in 2020, an annual raise in GFRPs of almost 6% is expected as well as CFRP materials leading the production of GF composites compared to the pre-pandemic period [16–18].

However, if, on the one hand, composite materials are largely used in industrial sectors, on the other hand, they will dramatically increase the amount of waste. Part of this waste derives from offcuts generated during the production phases of composite materials, but a large part derives from end-of-life products. It is easy to presume that a defined waste management and recycling policy is required as an annual increase of CFRP waste of around 20 kTons by 2025 is estimated, and around 6000–8000 commercial aircraft will reach their end-of-life by 2030 [8,19–21]. Based on these considerations, considering the amount of energy required for the production of this fibre's typology, it is possible to assert that carbon fibres are a very energy-expensive reinforcement [22]. It has been calculated by a life cycle assessment (LCA) analysis that the amount of energy required for the production of vCFs is almost 198–595 MJ/kg [16]. Similar considerations can be made about the production of virgin glass fibres (vGFs) that, as well as CFs, are one of the most used synthetic fibres; for a vGF, an amount of almost 13–32 MJ/kg is required. Then, from LCA analyses, it appeared that vCFs required an amount of energy that is around 10 times higher than that required for the production of vGFs [23]. Based on these considerations, if a recovery and recycling plan is activated, a sensible reduction in the environmental impact can be obtained. The production of recycled CFs (rCFs) requires an amount of energy that is much less than that of vCFs, even if it depends on the recycling method used. According to the literature, an amount of almost 38 MJ/kg is required to recycle CFRPs using a chemical method, which is around 20–30% of the energy required for the production of vCFs [24]. Therefore, these interesting results in terms of reduced costs and energy and environmental sustainability move the research and industry worlds towards a circular economy based on the reusing, remanufacturing and recycling of offcuts and end-of-life composite materials.

Based on a primary analysis of the diffusion of composite materials in the industrial world and the rising demand in terms of their use, it is possible to conclude that the amount of waste materials will inevitably increase. Therefore, based on the prevision of waste production per industrial sector by 2025 (Figure 2), there is a strong need to know and study appropriate recycling strategies for this category of materials [17].

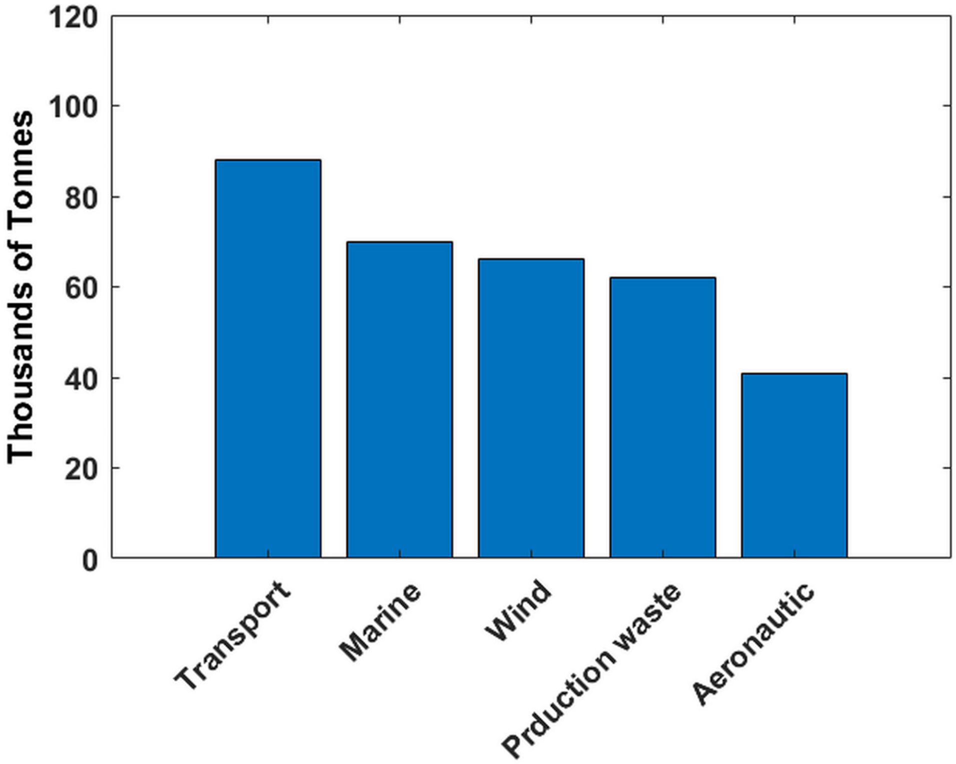


Figure 2. Prevision of waste production per sector in thousands of tons by 2025 [17].

To date, it is suspected that several tons of composite materials never reach the appropriate recycling channel due to the lack of proper waste management policies and environmental legislation [25]. Therefore, in this context, the life cycle of composite material can be resumed in a linear process that starts from the raw material and finishes with offcuts and end-of-life composite materials stocked in landfill as it is the cheapest method of disposal. Then, aimed at improving environmental sustainability, more mature and restrictive legislations are required regarding end-of-life materials and the use of landfill for composite materials. These regulations have to be applied not only to end-of-life materials but also to prevent and reduce the use of this category of material [26–28]. Therefore, the idea is to work toward a circular economy aiming at using recycled materials produced with renewable energy, reducing at the same time the production of waste materials and toxic chemical agents [29].

If, on the one hand, the idea of the circular economy is the production of new products with recycled materials and zero waste, on the other, composite materials are still largely produced using virgin fibres. The use of conventional prepreg materials and lay-up processes are still widely used for the manufacturing of composite materials in the aeronautic, sports and wind sectors. All these materials when decommissioned at the end of their lives are usually disposed of in landfills or incinerated in order to generate energy from the combustion, but it is estimated (in accordance with the review of composite waste in UK supply chains) that composite material wastes are usually just buried in landfill sites [30–32]. Focusing the attention on composite incineration, it is important to note that even if this process demonstrates the potential to produce almost 30 MJ/kg from CFRP waste combustion [33], it is not considered, similar to landfill disposal, an appropriate recycling method. As landfill and incineration do not consist of waste material recovery, they do not reintroduce recycled material into new industrial products according to the circular economy philosophy. Based on these considerations, the European Union implemented some directives adding tax rates on waste materials and banning landfill disposal in some European countries. Therefore, the above-mentioned 2008/98/EC directive suggests a waste administration should indicate a defined recycling channel and establish at the same time that an amount of almost 70% in weight of waste material must be recycled in order to reduce both landfill and the environmental impact [34]. As well as the 2008/98/EC, the 2000/53/EC directive requires that almost 85% of the weight of the end-of-life vehicles must be recovered and recycled.

All these aspects lead to the conclusion that the recycling of synthetic fibres is an open challenge for both industry and research groups to study a suitable way to recover fibres from the polymeric matrix in order to remanufacture a composite material using the recycled fibres [35,36]. The fibre recovery methods depend on both the fibre typology and the nature of the polymeric matrix (thermoplastic or thermoset). Between thermoset and thermoplastic resins, the recycling process of thermoset composites is more complex due to the crosslinks that characterise the matrix structure, making this class of composite material non-remelting, non-remoulding and, therefore, hard to recycle [37].

Currently, thermoset composite materials are widely used for structural applications; it was estimated that they are almost 80% of the overall used FRP composites [38,39]. The large employment of thermoset polymers for the production of composite materials can be attributed to high chemical and thermal stability, dimensional stability and mechanical properties that characterise this matrix typology.

To date, it is estimated that CFRP materials are widely used in the aeronautic field (almost 26 kTons in 2020), where a large part of the total demand is predominately in commercial aircraft production [11,14,15]. CFRP materials are also ordinarily used in automotive and sports, where, in this case, the total demand during 2020 was almost 15 kTons. On the other hand, even if CFRP materials are affirmed in the automotive, sports and aeronautic sectors, an interesting demand for composite materials is occurring in the energy and marine fields [11,14,15,40,41].

Over recent decades, the wind energy sector has been considered one of the most promising sources of renewable energy. Numerous plants are annually installed, and numerous wind turbines have been manufactured. However, some factors related to noise, landscape alteration or the eventual impact on flora and fauna have limited the installation of these structures in the proximity of cities. These aspects promoted a growing interest in offshore plants that are, at the same time, exposed to constant and higher wind flows and subjected to reduced restrictions in terms of environmental impacts [42]. Therefore, the growing interest in the wind energy sector, in addition to the knowledge of the manufacturing processes that permit the production of larger wind turbines, promotes a larger use of CFs and GFs to produce wind blade structures. It is expected that around 66 kTons of composite materials will be employed for wind blade production by 2025, and looking to the imminent future, the production of almost 40 million tons of composite wind blades as a consequence of the intensive installation of wind turbines by 2050 is expected [17,43,44].

As mentioned above, CF and GF composites are attracting particular attention in the marine field due to their high durability in aggressive and corrosive environments as well as for their high mechanical properties. In fact, CFRP materials are being increasingly used for the manufacturing of offshore platforms for petroleum extraction. It is known that steel is widely used in pipelines and primary and secondary structures. However, these parts are subjected to severe environmental conditions, which lead to corrosion phenomena that can be responsible for stopping the system. In this scenario, composite materials are considered in the production of primary parts (i.e., risers or anchor devices), secondary parts (i.e., helicopter bridges and stairs) or pipelines [42]. CF and GF composites demand is rising in the production of parts of ship hulls and structural parts in racing boats or luxury yachts. It is known that competitions require high-performance and lightweight materials; therefore, the overall structure of a racing boat is produced with CFRP materials (i.e., America's Cup boats or H1 unlimited boats). Focusing on luxury yachts, the trend is moving from wood- and steel-based boats to composite-based ones; indeed GFRP materials are largely used for the production of the hull, structural parts or propellers (i.e., Azimut or Fiart boats). Another example of a luxury yacht is the "Tecnomar for Lamborghini 63", which is the only yacht totally manufactured with CFRP materials. The use of composite materials in pleasure yachts offers some advantages when compared to the traditional ones as they are lighter and characterised by reduced maintenance costs thanks to their better resistance to marine organisms and improved corrosion resistance [42]. All these aspects led to an increase in CF utilisation from 800 Tons in 2010 to around 3 kTons in 2020 [11,14,15]. Based on these considerations, it is clear that the quantity of composite materials will inexorably increase over time along with the amount of waste materials; therefore, some technologies considered green and eco-friendly will become unsustainable. Therefore, based on the rising interest in the use of composite materials in the marine field and on the concrete issue of waste management and more restrictive legislations in terms of material recycling, a defined change in how waste is generated within the supply chain and the composite manufacturing process is required, and a more efficient recycling process of composite materials is mandatory.

Currently, the marine field is moving toward the large use of composite materials, and a relevant waste management issue is expected in the imminent future. Therefore, in the absence of concrete cases of marine composite recycling, the present review is focused on the evaluation of the state of the art of the most affirmed recycling technologies of CFRP and GFRP composite materials, with a focus on the physical and chemical properties of the recycled fibres at the end of each recovery method. The scope of the present review is to compare the energy consumption of each method and the mechanical properties of the recycled composite materials to find the most suitable recycling conditions with a view to environmental sustainability.

2. Recycling Processes of FRP Materials

The above-mentioned 2008/98/EC directive on the recyclability of composite materials defines a waste material hierarchy (Figure 3), suggesting to the state members the appropriate recycling route in order to reduce landfill disposal and environmental issues [34]. The use of an appropriate recycling method reduces not only the environmental impacts but also the production of virgin raw materials and the overall cost of new recycled industrial products. For example, it is estimated that, even if some recycling methods are still expensive in terms of energy requirements, the production of recycled glass fibres (rGFs) and resin requires, respectively, almost 25–30 MJ/kg and 20–90 MJ/kg in comparison with 13–45 MJ/kg and 76–137 MJ/kg required to produce virgin materials. In addition, recycled materials do not require the same amount of fossil fuel consumption used to produce virgin ones. All these aspects make recycled materials more competitive, allowing the use of these materials in a closed loop in accordance with the circular economy philosophy [45–48].

To date, even if the circular economy philosophy promotes the principles of material prevention, reuse and repurposing, the main strategies to manage waste are recycling, incineration and, at least, landfill disposal; despite the last one being not considered an eco-friendly approach because it does not allow for energy recovery and leads to severe environmental impacts, it is the most common and cheapest method used to manage waste materials [49,50].

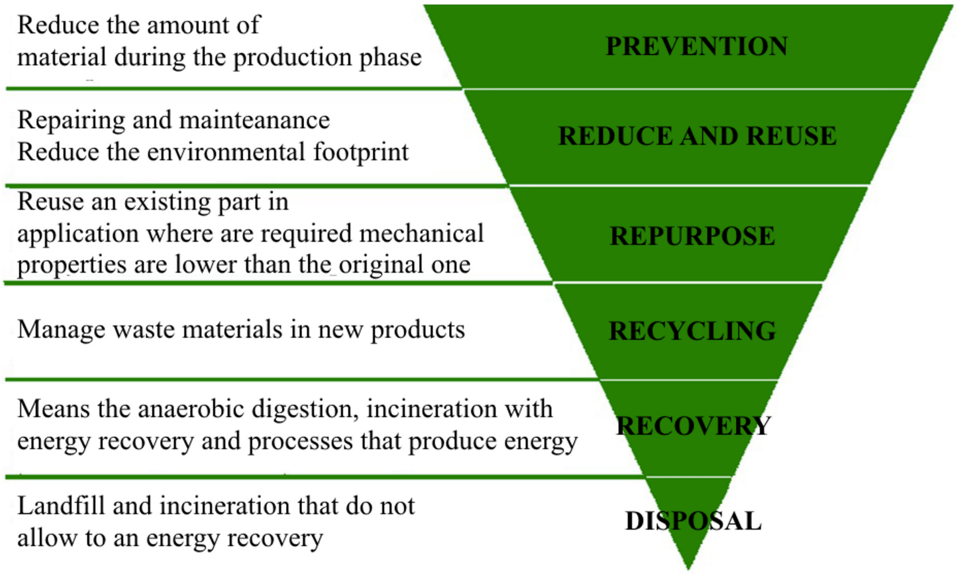


Figure 3. 2008/98/EC directive waste management hierarchy.

Incineration is usually employed to recover the embodied energy from composite materials. However, it is not considered a good alternative for material recycling as it does not reintroduce waste material into new recycled industrial products and releases large amounts of hazardous substances into the environment, making this solution non-sustainable and dangerous for human health [51].

Therefore, the recycling process of composite materials has been largely considered to recover embodied energy from FRP materials and recover fibres from the polymeric matrix to reuse them in recycled composite material. There are three main recycling strategies, such as chemical, thermal and mechanical methods. The first one includes a composite treatment with acid, solvolysis or supercritical fluid solvolysis. The thermal recycling method is characterised by processes such as pyrolysis and fluidised bed pyrolysis. The mechanical process is mainly characterised by material fragmentation into small pieces using grinding, shredding or milling. Each of these recycling methodologies produces recycled fibres with different characteristics in terms of length, mechanical properties and surface quality. Furthermore, it is highlighted that each of these techniques gives back different results if employed to recycle CFRP or GFRP composite materials. These aspects will be discussed in the following sections.

2.1. Chemical Recycling

The chemical recycling process, usually known as solvolysis, consists of matrix depolymerisation with fibre liberation. The polymeric matrix is dissolved by means of a chemical solution into the basic monomers, oils and gasses that can be used as feedstock to manufacture new polymer materials or as fuel in other industrial processes [52,53]. Depending on the solvent typology, the chemical recycling process can be called hydrolysis if a water-based solution is used, glycolysis if substances such as methanol, ethanol or acetone are used for the chemical solution and acid digestion in the case of solutions based on acids [22,54]. A schematisation of a generic chemical recycling process is reported in Figure 4.

As aggressive solutions and severe processing conditions are employed in chemical recycling techniques, an appropriate laboratory apparatus is required; therefore, usually, stainless-steel pressure vessels are used as a reactor in order to avoid corrosion from the chemical reactions [53]. As mentioned above, severe processing conditions in terms of high pressure and temperature are used for a long time to process the composite material.

This aspect leads to an energy demand that is in the range of almost 20 MJ/kg up to 60–90 MJ/kg. Therefore, even if the chemical process can be used for both CFs and GFs, it is more suitable for CFs as GFs are characterised by a lower commercial value and are sensitive to degradation because of their fragility when exposed to high temperatures and corrosive conditions [55,56].

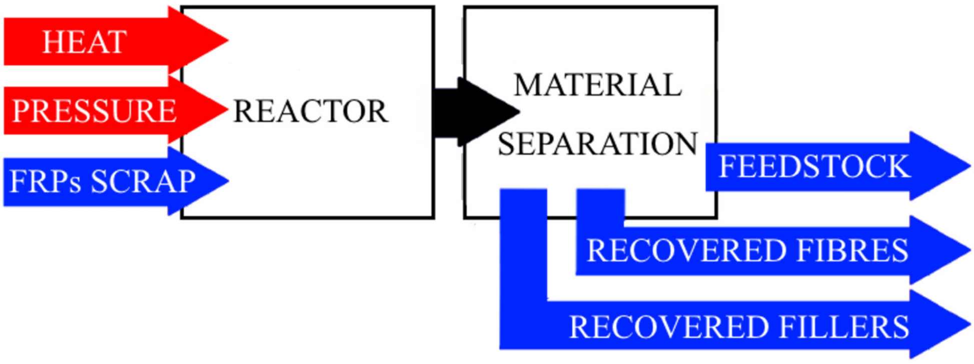


Figure 4. Schematisation of a generic chemical recycle.

Based on the chemical solvents and the process parameters, it is possible to distinguish supercritical and subcritical solvolysis, the electrochemical recycling method and acid digestion. Each chemical recycling process will be discussed in detail.

2.1.1. Supercritical and Subcritical Solvolysis

This chemical method is usually based on the use of a heated solution to recover synthetic fibres by means of matrix decomposition. Different results can be obtained thanks to the availability of different solution typologies and process parameter combinations. Then, two main categories of chemical recycling processes can be identified, supercritical and subcritical solvolysis, that differ from each other by the solution conditions in terms of pressure and temperature. Subcritical solvolysis is usually carried out at atmospheric pressure and generally uses a solution at a temperature below 200 °C; consequently, some catalyst and swelling additives are used to accelerate the recycling process and guarantee satisfying fibre recovery [56]. On the other hand, in supercritical solvolysis, high temperatures and pressure in the range of 350–450 °C and 4–27 MPa, respectively, are used [56,57]. These working conditions make the fluid very efficient in terms of diffusion and transport coefficients, allowing good penetration into the laminate, followed by satisfying resin removal [56]. The overall matrix removal from the fibres is achieved as a consequence of the chemical solution diffusion on the impregnated fibres, the reaction of the fluid with the surface of the fibres and the dissolution of the matrix and monomer asportation [40,53,57]. All these aspects lead to some advantages as it is possible to obtain clean fibres with competitive mechanical properties; in the case of rCFs, they maintain the original length [58]. On the other hand, this type of solvolysis is expensive because it requires reactor vessels that are able to resist corrosive conditions and a large amount of energy [56].

Among different types of fluids used to prepare the chemical solutions, water is the most employed thanks to its environmentally friendly nature [59]. It was found in the literature that further solutions are prepared using methanol, ethanol and acetone in addition to some catalysts, such as KOH or NaOH [57,60]. It has been demonstrated that even if these solutions preserved the mechanical properties of the recycled fibres, they caused an oxygen concentration on the fibre's surface involving a reduced adhesion with the new matrix, followed by a reduced interfacial shear strength of the recycled fibres [60].

Several studies have focused on composite material recycling through supercritical and subcritical chemical solutions, especially on the recycling of CFs. In contrast, few works have focused on GF recycling by means of solvolysis because of the GF fragility at high temperatures and corrosive agents. Oliveux et al. [53] studied the recycling process of

GFs in a polyester resin using a subcritical water-based solution. The effect of the process parameters on the efficiency of the process and the quality of the recovered fibres was evaluated. Then, the GFRP composite material was treated with a solution in a temperature range of 200–374 °C and a pressure of almost 22 MPa, concluding that, at the end of the recycling process, GFs present a reduction in mechanical strength of almost 50–60% in comparison with the virgin ones. Another study was carried out by Oliveux et al. [56] in accordance with the EURECOMP report [61] on polyester resin reinforced with GFs recycled through a water solution solvolysis with a temperature of 300 °C and a pressure of almost 25 MPa. At the end of the treatment, fluid diffusion problems were detected in the central region of the laminate, and additional observations were carried out on supercritical fluid, concluding that its saturation during the process led to a reduction in the recycling efficiency.

On the other hand, Zabihi et al. [62] studied the recycling process of GFRP material using a solution of H_2O_2 with assisted microwave radiation. The recycling material was placed in the solution bath at a temperature of almost 70 °C for a period of treatment of 60 min. Using this recycling process, it was possible to obtain a good epoxy matrix decomposition with appealing residual mechanical properties of the recycled fibres. The rGFs revealed a reduction in tensile strength of almost 7% in comparison with the vGFs. This result is expected as the recycling process does not involve high processing temperatures, resulting in the preserved mechanical properties of the recycled fibres.

Kuang et al. [63] recycled a thermoset composite material characterised by an epoxy resin reinforced with GF. They used a solvent mixture at a relatively low temperature (170 °C) at atmospheric pressure. The authors concluded that a period of treatment of almost 28 min leads to 50% of resin depolymerisation, meanwhile a period of 70 min to 95%, with the rCFs characterised by a reduction in tensile strength of almost 10% in comparison with the virgin ones.

Different results can be instead achieved considering CFRPs. For example, Pinero-Hernanz et al. [57] analysed the recycling process of CFRP materials using supercritical and near-supercritical solutions using a temperature of almost 250–400 °C with a pressure variation of 4–28 MPa. The solution was H_2O_2 based with the addition of a KOH catalyst. At the end of the experimental campaign, they concluded that the presence of the KOH catalyst improved recycling efficiency by up to 95.4%, with satisfying epoxy matrix removal and a reduction in the mechanical properties of the rCFs by 2–10% in comparison with the vCFs.

Liu et al. [64] found an optimal chemical solution based on an optimal ratio of KOH mass, phenol mass and water volume (1 g KOH, 10 g phenol, and 100 mL water) and obtained the highest epoxy matrix removal efficiency whilst preserving the mechanical properties of CFs at the same time. The main result obtained is that treatment at a temperature of 315 °C and 325 °C for 30 min led to a fibre recovery of almost 95.2 and 100%, respectively. Focusing on the mechanical properties of the rCFs, it was asserted that these fibres are comparable with virgin ones.

Kim et al. [59] studied the recycling of CFRP material using a supercritical water-based solution, obtaining an epoxy resin removal rate of almost 99.5% after a period of treatment of almost 120 min. The recycling process was carried out at a temperature of almost 400 °C and a pressure of around 28 MPa. At the end of the recycling process, a reduction in the fibre tensile strength in a range of 18–36% in comparison with vCFs was observed. A summary of the specific process conditions and results in terms of matrix depolymerisation and the residual properties of the recovered fibres through supercritical and subcritical solvolysis is reported in Table 1.

An overview of the chemical recycling method using subcritical and supercritical solvolysis leads to the conclusion that, although the nature of the chemical solution changes, it is possible to obtain a recovered fibre surface that does not present any trace of old matrix. Then, as shown in Table 1, this method is characterised by an overall efficiency that is, in all cases, over 95%. On the other hand, in the case of GFs, the solvolysis leads to a severe reduction in the mechanical properties of the recovered fibres that, in some cases, are around 50% when compared with the mechanical properties of virgin fibres.

Table 1. Main results deriving from the solvolysis using supercritical and subcritical fluids.

Material	Solution	Process Parameters	Process Efficiency	Mechanical Characteristics	Ref.
GF—Polyester	Subcritical water-based	200–374 °C 22 MPa	Good matrix removal	Mechanical strength 50–60% reduction	Oliveux et al. [53]
GF—Epoxy	Subcritical H_2O_2 solution, microwave-assisted	70 °C	Good matrix removal	Tensile strength −7%	Zabihi et al. [62]
GF—Epoxy	Subcritical mixture of solvents, EG/NMP 10% EG	170 °C atmospheric pressure	95% matrix removal	Tensile strength −10%	Kuang et al. [63]
CF—Epoxy	Supercritical H_2O_2 solution with KOH catalyst	250–400 °C 2–28 MPa	Up to 95.4% matrix removal	Mechanical properties 2–10% reduction	Pinero-Hernanz et al. [57]
CF—Epoxy	Supercritical water-based with KOH and phenol	315–325 °C	Fibre recovery 95.2–100%	Comparable with virgin fibres	Liu et al. [64]
CF—Epoxy	Supercritical water-based	400 °C 28 MPa	99.5% matrix removal	Tensile strength 18–36% reduction	Kim et al. [59]

2.1.2. Electrochemical Recycling Process

The electrochemical recycling process consists of matrix removal using high electrical currents. In Figure 5, a schematisation of this recycling method is represented. Through the electrochemical method, it is possible to obtain matrix fragmentation by means of electrical currents on the composite material; however, in some cases, the FRP material can be used as electrodes in the electrochemical cells. Meanwhile, in other circumstances, high electrical pulses are generated between the electrodes and are transferred to the material placed in a water solution [65]. This recycling process permits long and clean recycled fibres to be obtained, but, on the other hand, it is characterised by high energy consumption [66,67].

Therefore, the electrochemical recycling method does not represent an appealing strategy to recycle GFRPs because the amount of energy required for fibre recovery, in some cases, is higher [68] than the energy required for the production of virgin ones [23].

Otherwise, for CFRPs, some applications were found in the literature. For instance, Zhu et al. [69] studied the electrochemical recycling method on CFRP materials using an additional chemical solution based on NaCl and KOH catalysts. The research group examined the effects of chemical solution concentration and current intensity on polymer fragmentation, concluding that the combination of the effects of the electrical currents and the dissolution from the chemical solution led to a total epoxy resin decomposition, followed by fibre recovery. At the end of the experimental campaign, it was found that the recycling process in addition to the suitable process parameters allows rCFs to be obtained that possess almost 90% of virgin fibre tensile strength and improved interfacial shear strength (+20% in comparison with vCFs).

A different approach was used by Oshima et al. in their research, as the CFRP material constitutes part of the electrochemical cell [70,71]. In their first experimental campaign, Oshima et al. [70] studied the effect of the combination of high voltage and electrolyte solution on matrix removal efficiency. The experimental campaign considered the use of a carbon/epoxy composite material as an anode in an aqueous solution of NaCl, KCl, NaOH, KOH and Na_2CO_3 with a concentration between 0.01 and 1.0 mol L^{-1}. At the end of the recycling process, some matrix cracks and delamination that moved from the external side to the inner part of the laminate were observed as a consequence of the high voltage application, and further composite weight loss was detected as a consequence of the motion of the electrons from the anodic to the cathodic regions. A further phenomenon responsible for the weight loss is the matrix peeling off due to the formation of gas on the laminate surface. This condition, in conjunction with localised electric fields, influenced fibre continuity as some CF fragmentation was detected.

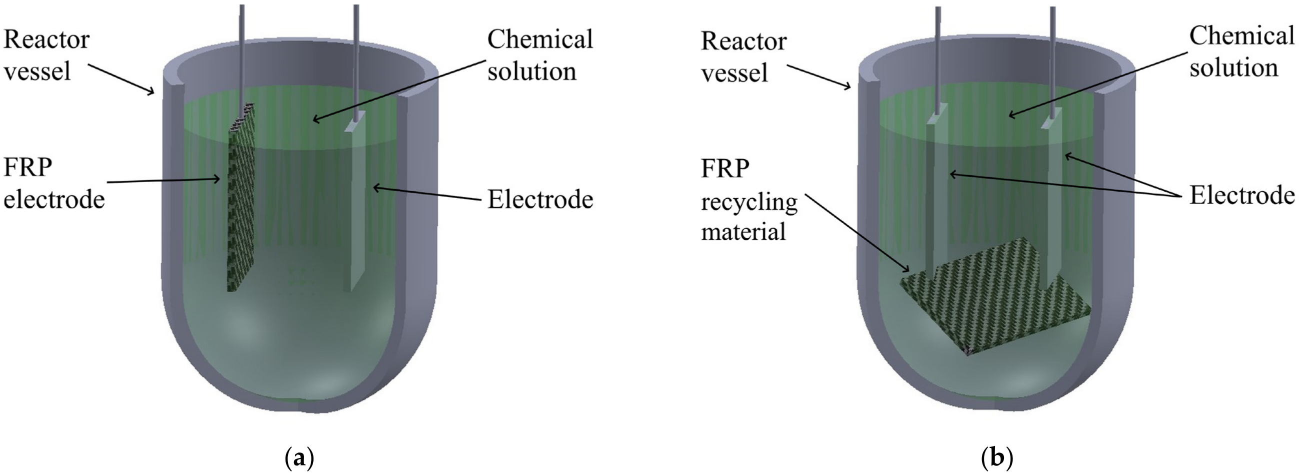


Figure 5. Schematisation of the electrochemical recycling process: FRP material used as electrode (**a**); FRP recycling material in electrochemical solution (**b**).

In a further research activity, Oshima et al. [71] used the same experimental setup as their previous work [70], varying only the electrochemical solution that consists of phosphoric acid as the electrolyte. At the end of the experimental campaign, the research group concluded that matrix removal does not depend on the nature of the electrochemical solution, and it is only influenced by the gas formation that mechanically removes the epoxy matrix from the fibre surface. The authors further observed that the matrix peeling off started from some superficial and internal voids generated during the anodic treatment where the electric conduction led to matrix heating. Similar to the previous work, the recovered CFs were characterised by some cracks that influenced fibre continuity and residual mechanical properties. The CFRP recycling conditions and the results in terms of mechanical characteristics and matrix removal through the electrochemical recycling method are shown in Table 2.

The results shown in Table 2 revealed that the electrochemical recycling method is often responsible for cracks and delamination that propagate into the recycling composite material. These cracks that are generated by the formation of gases during the recycling process cause the fragmentation of the fibre bundles, consequently reducing the mechanical properties of the recovered fibres. Therefore, this aspect, in addition to the reduced volume of the processed material and high energy demand, makes the electrochemical process unsuitable for fibre recovery.

2.1.3. Acid Digestion

The acid digestion process differs from the traditional supercritical and subcritical recycling process by the temperature and pressure at which the matrix dissolution occurs. Indeed, this recycling process takes place at atmospheric pressure and requires expensive apparatus. Usually, this recycling process is based on a pre-treatment with acids or other solutions with catalysts aiming at a composite swelling that permits a better penetration of the chemical solution into the laminate and then a deeper matrix depolymerisation [72,73]. It is demonstrated that the acid pre-treatment and the use of catalysts improve the composite swelling; among the catalysts, acetic acid is responsible for the higher composite swelling ratio, which is almost 50% in comparison with ethanol, which allows a swelling ratio of around 20% [74]. In Figure 6, the swelling ratio of the most used solvents and acetic acid is reported.

Table 2. Main results deriving from the electrochemical recycling method.

Material	**Solution**	**Process Efficiency**	**Mechanical Characteristics**	**Ref.**
CF—Epoxy	Chemical solution with NaCl with KOH catalyst	Total matrix removal	Tensile strength −10% and interfacial shear strength +20%	Zhou et al. [69]
CF—Epoxy	Water-based solution with NaCl, KCl, NaOH, KOH and Na_2CO_3	Delamination, matrix cracks and pieces of matrix that peel off from the laminate	Fibre fragmentation	Oshima et al. [70]
CF—Epoxy	Phosphoric acid	Pieces of matrix that peel off from the laminate	Fibre fragmentation	Oshima et al. [71]

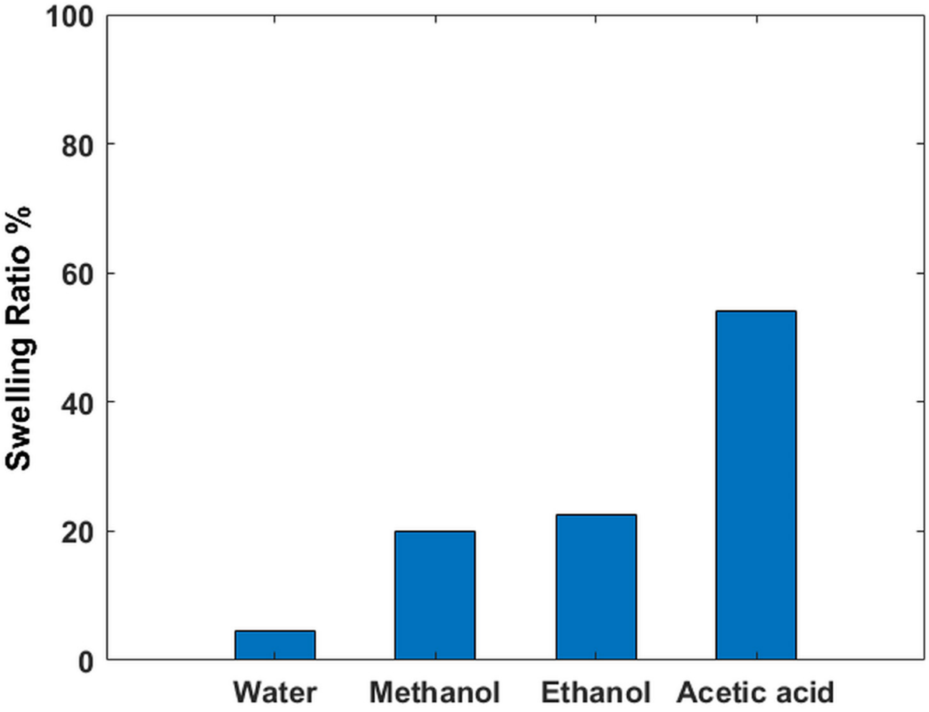


Figure 6. Swelling ratio of the different solvents on composite materials [74].

Some studies found in the literature are based on this recycling process. Rani et al. [75] examined the treatment of a glass/epoxy composite material with a microwave-assisted chemical solution based on H_2O_2 and acetic acid. During the experimental campaign, the GFRP material was treated with different chemical solutions heated by means of microwave exposure for a period of 180 s. The authors pointed out that the optimal recycling conditions can be reached with a chemical solution-to-solvent combination of almost 30%, allowing a resin decomposition rate of around 97.2%. The tensile tests carried out on the rGFs revealed a reduction in the tensile strength and Young's modulus of almost 2.4 and 8.6%, respectively, in comparison with virgin fibres. The research group performed further SEM analyses on the recovered fibres to study their surface morphology, confirming that the surfaces of the fibres were perfectly clear without any presence of the old resin. Xu et al. [76] studied the pre-treatment process of CFRP material with acid acetic, obtaining satisfying composite swelling. Then, the pre-treated composite material was treated with an H_2O_2 chemical solution and placed into a hermetic autoclave at 80–150 °C for a period that varied from 5 to 120 min. The result is that, with well-defined process parameters, it is possible to obtain quite good epoxy matrix dissolution; indeed, an efficiency of 99.1% was obtained with a temperature of 90 °C and a period of treatment of 30 min. The residual rCF mechanical properties were estimated to be around 98 and 90% in comparison with the vCFs, if the composite material was treated at 100 or 120 °C, respectively.

Zabihi et al. [77] studied a combination of the pre-treatment with tartaric acid and epoxy matrix dissolution in an H_2O_2 chemical solution with assisted microwave radiation. The experimental campaign was carried out with different ratios of tartaric acid in a chemical solution with a defined period of microwave radiation that varied from 1 to 3 min. At the end of the experimental campaign, the research group concluded that this process led to a matrix dissolution of 95%, with a reduction of almost 8% in tensile strength for the rCFs, in comparison with virgin ones. The main results in terms of process efficiency and mechanical properties of the recovered fibres by means of acid digestion are reported in Table 3.

Table 3. Main results deriving from acid digestion.

Material	Solution	Process Efficiency	Mechanical Characteristics	Ref.
GF—Epoxy	Microwave-assisted H_2O_2 with 30% acetic acid	97.2% matrix removal	Tensile strength −2.4% and Young's modulus −8.6%	Rani et al. [75]
CF—Epoxy	H_2O_2 solution with acetic acid	99.1% matrix removal	Mechanical properties reduction 2–10%	Xu et al. [76]
CF—Epoxy	H_2O_2 solution with pre-treatment with tartaric acid	95% matrix removal	Tensile strength −8%	Zabihi et al. [77]

Looking at the results in Table 3, it is possible to assert that, similar to solvolysis with subcritical and supercritical fluids, acid digestion, thanks to the addition of a catalyst that allows composite swelling and then an improved penetration of the solution into the laminate, leads to a high matrix dissolution (in all cases over 95%) and then to clean recovered fibres. However, this process does not require a high processing temperature as in solvolysis; therefore, the recycling of the fibres is not subjected to thermal alteration, and their residual mechanical properties are largely preserved.

2.2. Thermal Recycling

In thermal recycling methods, GFs and CFs are released from the matrix using high temperatures that deteriorate the polymeric matrix. These recycling methods are usually classified in pyrolysis and fluidised beds.

2.2.1. Pyrolysis

Pyrolysis is a destructive method where synthetic fibres are recovered through matrix decomposition into a mixture of solids, oils, gases and char substances. Polymeric matrix disintegration is performed in an inert atmosphere where the thermosetting composite material is heated to a high temperature in the range of 400–1000 °C, depending on the nature of the composite matrix [16,78–80]. A schematisation of a typical pyrolysis method is represented in Figure 7.

The absence of oxygen during matrix disintegration leads to the formation of some char materials on the fibre surface. Therefore, an additional post-pyrolysis procedure in a furnace is required to remove the residues of char materials with the aim of obtaining cleaner recycled fibres [19,81]. This recycling process is used for both GFRPs and CFRPs, with some differences below discussed.

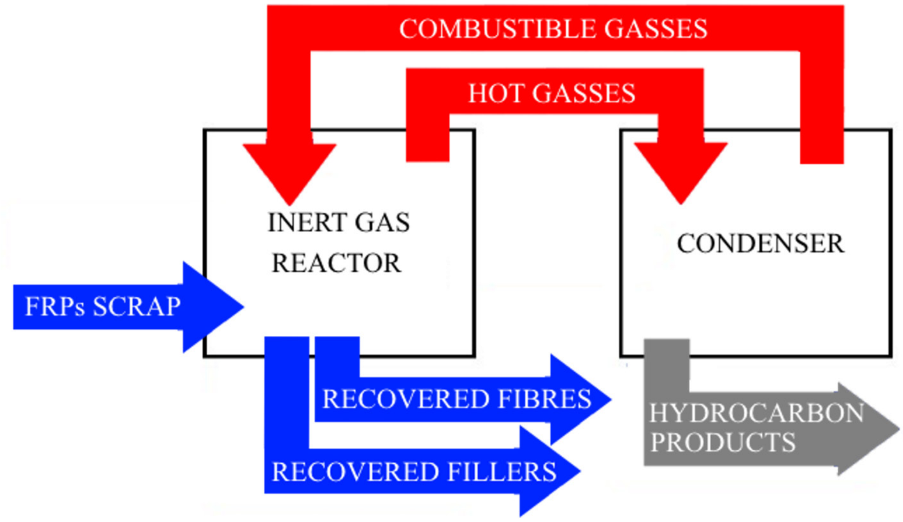


Figure 7. Schematisation of a typical pyrolysis process for thermoset composite materials.

An example of recovered GFs is represented in Figure 8, where it is possible to distinguish rGFs before and after post-pyrolysis treatment [81]. During the process, the recycling composite material is placed into a reactor and subjected to a temperature of around 450 °C for a period of 270 min. The pyrolyzed fibres (Figure 8a) are characterised by a black aspect because of the deposition of materials that derive from the matrix carbonisation, preserving, at the same time, the typical aspect and structure of the composite material [81]. On the other hand, considering rGFs after the post-pyrolysis process (Figure 8b), it is possible to assert that the fibres do not present the tendency to agglomerate as well as in Figure 8a; therefore, it is possible to conclude that all char materials have been removed from the additional process [81].

Based on the two recycling steps required for fibre release, it is possible to assert that the pyrolysis method requires a large amount of energy estimated to be around 20–30 MJ/kg [55]. However, as some oils and gases are produced from matrix dissolution, it is possible to cover part of the overall energy demand using those materials as fuels [55,82].

It is estimated that GFs suffer thermal processes reducing their mechanical properties to around 50% when the recycling temperature is already around 400 °C. Therefore, pyrolyzed GFs are often used as reinforcements in non-structural applications, such as sheet moulding compounds (SMCs) and bulk moulding compounds (BMCs) [41,83]. Based on these results, de Marco et al. [84] studied the effect of pyrolysis on GFs under a nitrogen atmosphere with temperatures in the range of 300–700 °C and a period of treatment of 30 min. The research group studied the sustainability of the thermal process for recycling SMC materials aiming to use recycled GFs as reinforcement in BMCs. The mechanical properties of the recycled BMC materials were compared with those of virgin BMCs, and it was found that the most suitable recycling temperatures are in a range of 400–500 °C. Under these conditions, the recycled BMC composites revealed a reduction in flexural strength of almost 19% when compared with that of virgin BMC materials.

Similar conclusions in terms of the reduction of the mechanical properties of the recycled fibres can be drawn in the research work carried out by Nahil and Williams [85]. They studied the pyrolysis process on CFRP material with a temperature variation in the range of 300–500 °C and a further process at 700 °C for a period of 60 min. Additional post-pyrolysis treatment was carried out at 500 and 700 °C, aiming to remove any trace of char materials on the recycled fibres. The research group pointed out that the best recycling conditions can be obtained through pyrolysis and post-pyrolysis with a processing temperature of 500 °C, as the best residual mechanical properties can be maintained at around 93 and 96% for tensile strength and Young's modulus, respectively, in comparison with vCFs. Further post-pyrolysis process was carried out at a temperature of 700 °C to reduce the processing time; however, it was observed that the mechanical properties decrease by almost 65% in comparison with virgin fibres.

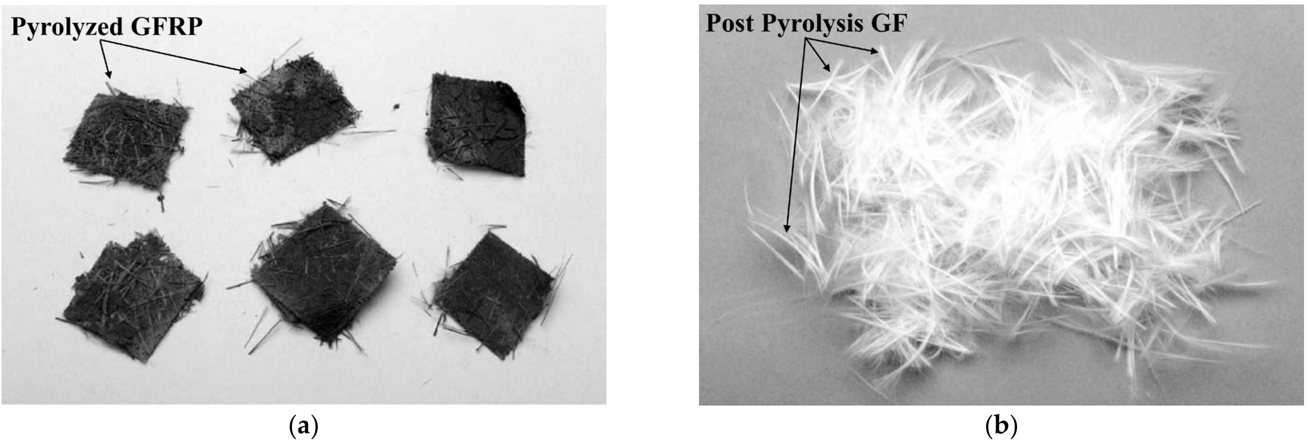


Figure 8. Example of pyrolyzed material before (**a**) and after (**b**) the post-pyrolysis process [81].

Therefore, an overview of the pyrolysis of GFs and CFs leads to the conclusion that the two steps of pyrolysis and post-pyrolysis, high temperatures and the processing time at high temperatures required to produce rCFs and rGFs have a relevant impact on fibre surface quality and mechanical properties. The processing parameters are able to modify the structure and the surface chemistry of the recycled fibres, leading to a fibre reinforcement that cannot be suggested as an appropriate substitute for virgin fibres [86,87].

A study on the effect of the temperature and duration of the post-pyrolysis process on the surface chemistry of rCFs was carried out by Mazzocchetti et al. [87]. They studied this effect on both vCFs and rCFs, concluding that vCFs are subjected more to surface degradation. During the experimental campaign, it was observed that both rCFs and vCFs are subjected to diameter reduction, but when vCFs are heated, the fibre surface is directly exposed to the oxidation agents resulting in diameter reduction and surface chemistry modification. In contrast, pyrolyzed CFs present a char layer that acts as a sacrificial layer that protects the recycled fibres from damage. The research group applied a post-pyrolysis process with a temperature of 500 °C initially for 20 min, then for 60 min on both vCFs and rCFs to analyse the diameter reduction evolution over time. An acceptable reduction of the section of the vCFs was achieved with a treating duration of 20 min (−2.8% in diameter), as a larger heating period leads to a diameter reduction of almost 10%. The research group pointed out that the same treating parameters are not so effective in completely removing the char layer from the recycled fibres; therefore, more severe conditions are required. In particular, an increase in the treatment duration (60 min) led to further diameter reduction comparable with the vCFs after a period of treatment of 20 min; in these conditions, the complete burning out of the char layer was reached. Therefore, at the end of the experimental campaign, it was pointed out that an appropriate balance between temperature and time of exposure leads to an appreciable rCF surface purification, while, at the same time, preserving the mechanical properties of the recycled fibres. Further tensile tests carried out on both virgin and recycled samples highlight that the sample typology produced with recycled CFs possesses mechanical properties comparable with that of the sample produced with virgin fibres. The recycled composite revealed an improvement in tensile strength and Young's modulus of almost 6.4 and 5%, respectively, when compared with the sample produced with virgin fibres. This result can be attributed to the post-pyrolysis treatment that promotes fibre–matrix adhesion. However, some studies in the literature focus on modified pyrolysis methods aiming to optimise the recycling process and control, at the same time, the quality of the recycled fibres. The modified pyrolysis methods are referred to in terms of the variation of oxygen gas concentration, the use of superheated steam and microwave irradiation.

The first one was implemented with the aim of managing fibre surface chemistry and its residual mechanical properties. Yang et al. [88] studied the combined effect of temperature, duration and oxygen concentration during the pyrolysis process of CF-reinforced epoxy matrix composites. The authors performed the experimental activities by varying the amount of oxygen, starting from an inert atmosphere with 0% of oxygen, then 5, 10 and 100% of air. The recycling process was carried out with a temperature variation in the range of 550–650 °C for a period of 15–60 min. They observed that, by increasing the amount of oxygen and the temperature and process duration, a significative chemical alteration on the surface of the fibres and a mass loss occurs. They concluded that the optimal pyrolysis process conditions are a temperature of 650 °C for a period of 45 min and an amount of oxygen of 5%. This balance between the process parameters led to a residual tensile strength of the rCFs of almost 80% in comparison with vCFs. In addition, the pyrolysis process with oxygen gas regulation allows oxygen content control on the fibre surface, hence the presence of functional groups that improve the interface adhesion between the recycled fibres and matrix in recycled composite materials.

The use of superheated steam in the pyrolysis process leads to the formation of matrix disintegration products characterised by a higher molecular weight instead of more volatile materials, such as carbon dioxide. Therefore, this variation in the recycling process leads to a more sustainable pyrolysis method, making it more sensitive to environmental issues. Kim et al. [89] studied the use of superheated steam to dissolve the epoxy matrix and recover CFs by exposing the CFRP material to water steam at a temperature of 550 °C for 30 min. A further post-pyrolysis process was applied at a temperature of 550 °C for a period in the range of 30–75 min. The research group concluded that for a complete surface purification of rCFs, a period of treatment of almost 75 min is required, observing, at the same time, that the exposition of rCFs to the post-pyrolysis parameters over time does not significantly affect the mechanical properties of the recycled fibres. They also found that a period of post-pyrolysis of almost 60 min leads to a tensile strength reduction of almost 10% in comparison with vCFs. Furthermore, similar to oxygen-assisted pyrolysis, the use of superheated steam leads to the formation of functional groups on the fibre surface that improve the adhesion capability of the recycled fibres with new resin.

Among the modified pyrolysis methods, microwave-assisted is the most recent and is considered more sustainable in comparison with traditional methods. Some research in the literature has focused on the microwave-assisted pyrolysis of GFs and CFs. For instance, Akesson et al. [90] studied the possibility of recycling wind turbine blade structures by recovering GFs from the epoxy matrix. The research group performed microwave-assisted pyrolysis at a temperature in the range of 300–600 °C for a period of 90 min, observing a mass reduction in the recycled fibres of almost 70% and a fibre surface still coated with residuals of char materials. At the end of the experimental campaign, they concluded that the rGF-reinforced composites demonstrate a reduction in flexural strength and modulus of almost 68 and 39%, respectively, in comparison with vGF-reinforced composites. The reduction in the mechanical properties of the recycled fibres can be attributed to the surface chemistry variation that leads to poor fibre–matrix adhesion and then to a fibre pull-out mechanism (Figure 9) when a solicitation is applied.

Jiang et al. [91] used microwave-assisted pyrolysis in an inert atmosphere with a temperature variation in the range of 400–600 °C for a period of treatment of 30 min to recycle CFs.

The experimental campaign was based on CFs recovering from an epoxy resin and on the possibility to apply these fibres as reinforcement in a thermoplastic matrix. Therefore, aiming to evaluate the contribution of the recycled fibres to the mechanical properties of the composite material, a comparison with vCFs in the same matrix typology was carried out. At the end of the experimental campaign, the research group pointed out that the rCFs demonstrate a reduction in the flexural strength and modulus of almost 14 and 25%, respectively, when compared with vCFs. Similar to the study carried out by Akesson et al. [90], the reduction of the mechanical properties can be attributed to the recycling process. Jiang et al. observed that a complete surface cleaning of the recycled fibres was not achieved; furthermore, the recycling process left some defects and cavities on the surface of the recycled material. The main results of the research where pyrolysis was used as the recycling method of FRPs are listed in Table 4.

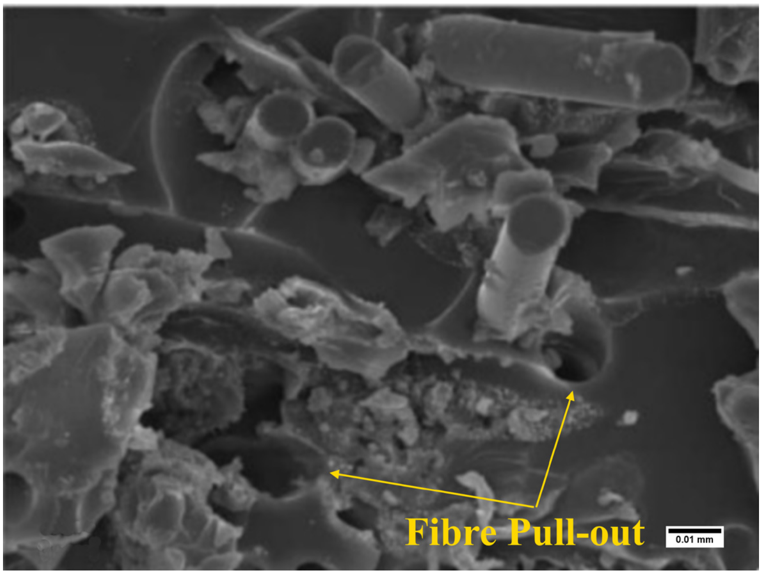


Figure 9. SEM micrography where the pull-out mechanism of rGFs is evidenced [90].

Looking at the Table 4, it is possible to observe that the pyrolysis recycling process leads to satisfying matrix decomposition; however, in some cases, especially when performed in combination with other processes, such as microwave irradiation, it does not allow total matrix removal. Therefore, the surface of the recovered fibres presents residues of the old matrix that result in poor adhesion at the fibre–matrix interface, followed by a reduction in the mechanical properties of the recycled composite materials.

2.2.2. Fluidised Bed

The fluidised bed is a recycling method where thermoset composite materials are placed into a bath characterised by silica sand. This recycling method cannot process waste composite materials of high dimensions; therefore, a previous shredding procedure is required to reduce the overall dimension of the recycling material [69]. The fluidised bed is a thermal recycling method, and matrix dissolution can be obtained by applying high temperatures. An additional hot airflow with a temperature in the range of 450–550 °C blows into the silica sand bath aiming to transform it into a fluid state that allows matrix dissolution and, consequently, the removal of the residues of the polymerised matrix [92]. Gases and other char materials deriving from matrix dissolutions are removed from the fluidised bath thanks to the hot airflow. At the end of the recycling process, all inserts or fasteners embedded in the recycled composite materials are recovered by means of the sand regrading process, meanwhile, the recycled fibres are reclaimed through a sieving process in an appropriate device [35,93]. In Figure 10, a generic schematisation of the fluidised bed recycling method is depicted.

During the fluidised bed process, the reaction temperature is selected in order to guarantee matrix dissolution without severe fibre degradation; however, even if a severe thermal effect on recycling fibres is avoided, other factors, such as sand abrasion in the fluidised bath, lead to severe degradation of the mechanical properties of the fibres. Pickering et al. [93] studied the fluidised bed recycling process to recover GFs from an unsaturated polyester matrix. The recovering process was carried out at a temperature of 450 °C and a fluidising velocity of hot airflow of almost 1.3 m/s. At the end of the experimental campaign, the research group pointed out that the rGFs are affected by a reduction in mechanical properties because of both the process temperature and the mechanical abrasion. Therefore, they observed that rGFs, even if they do not present a significative tensile modulus variation, are affected by a severe tensile strength reduction of almost 50% in comparison with vGFs.

Table 4. Main results deriving from the thermal recycling process of pyrolysis.

Material	Process Parameters	Process Efficiency	Mechanical Characteristics	Ref.
GF—Epoxy	400–500 °C 30 min	Total matrix removal	Flexural strength −19%	de Marco et al. [84]
CF—Epoxy	500 °C and post-pyrolysis 500 °C 60 min	Total matrix removal	Tensile strength −7% and Young's modulus −4%	Nahil and Williams [85]
CF—Epoxy	500 °C 60 min	Total matrix removal	Tensile strength +6.4% and Young's modulus +5%	Mazzocchetti et al. [87]
CF—Epoxy	650 °C 45 min 5% oxygen	Total matrix removal	Tensile strength −20%	Yang et al. [88]
CF—Epoxy	Water stream 550 °C 30 min Post-pyrolysis 550 °C 60 min	Almost total matrix removal	Tensile strength −10%	Kim et al. [89]
GF—Epoxy	Microwave-assisted pyrolysis 300–600 °C 90 min	Residues of matrix on the treated fibres	Flexural strength −68% and flexural modulus −39%	Akesson et al. [90]
CF—Epoxy	Microwave-assisted pyrolysis 400–600 °C 30 min	Residues of matrix on the treated fibres	Flexural strength −14% and flexural modulus −25%	Jiang et al. [91]

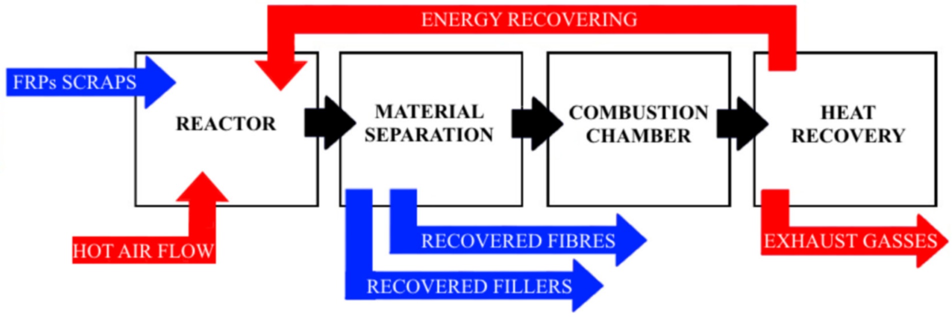


Figure 10. Schematisation of a generic fluidised bed recycling process.

The same conclusion can be drawn from the research carried out by Kennerley et al. [94]. They recovered GFs from polyester resin using a fluidised bed method at 450 °C and airflow in the range of 1.3–1.7 m/s, pointing out that the fluidising velocity does not significantly affect the recycled fibre strength. At the end of the recycling process, the authors tested the recycled composite materials, concluding that no appreciable variation in the flexural modulus was detected, in agreement with Pickering et al. [93]. However, a severe reduction in flexural strength was found, with a reduction of almost 50% in comparison with vGFs.

In another research work, Pickering et al. [95] performed a fluidised bed method on CFRP material with hot airflow at a temperature of 500 °C. At the end of the recycling process, they tested the vCFs and the recycled ones to compare the residual properties of the rCFs. They found that the recycled fibres are characterised by good quality and can be recovered with no significant reduction in mechanical properties. In summary, rCFs demonstrate a reduction in tensile stress of almost 18% in comparison with virgin fibres, with no significant differences in terms of tensile modulus.

The main results from research on the recovery of CFs and GFs by means of fluidised beds are listed in Table 5.

Table 5. Main results deriving from the thermal recycling process of fluidised beds.

Material	Process Parameters	Process Efficiency	Mechanical Characteristics	Ref.
GF—Polyester	450 °C Airflow 1.3 m/s	Good matrix removal	Flexural strength -50%	Pickering et al. [93]
CF—Polyester	450 °C Airflow 1.3–1.7 m/s	Good matrix removal	Flexural strength -50%; no appreciable variation in flexural modulus	Kennerley et al. [94]
CF—Epoxy	500 °C	Good matrix removal	Tensile strength -18%	Pickering et al. [95]

The results obtained through the fluidised bed process highlight that even if it allows for appreciable matrix removal from the surface of the recovered fibres, it is responsible for a severe reduction in the mechanical properties that, in some cases, halves the results in comparison with virgin fibres.

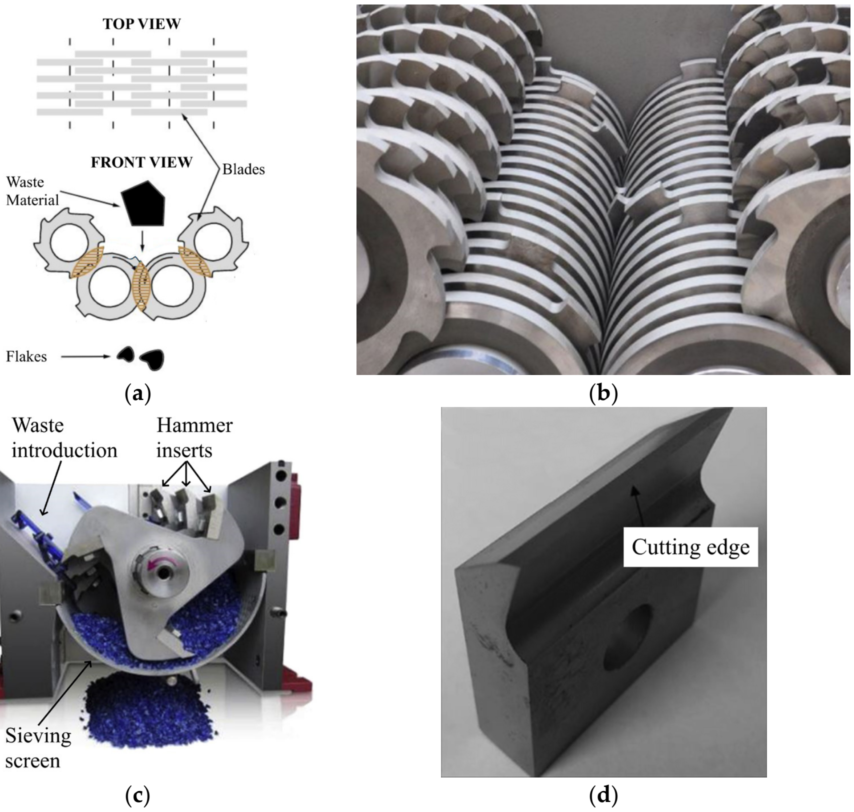
However, this thermal method, as well as pyrolysis, permits the formation of functional groups on the recycled fibre surface that improves the adhesion at the interface between fibres and the matrix. Despite all the advantages, the fluidised bed method permits the recycling of shredded composite materials and then the recovery of short fibres. All these aspects make this method limited to a lab-scale experiment.

2.3. Mechanical Recycling

Mechanical recycling is the most promising method to recycle synthetic reinforcements as high temperatures and chemical agents are not required for thermoset matrix depolymerisation. Therefore, contrary to what was observed in the chemical and thermal recycling processes, the mechanical one does not involve thermal and chemical fibre alterations. The mechanical method is usually used to indicate broader process categories, such as shredding, hammer milling, milling, and grinding, that, in some cases, can be combined to obtain recycled materials with the desired dimensions. Each process leads to composite fragmentation into small pieces, which can be further reduced to powder dimensions [96].

The shredding process (Figure 11a,b) consists of one or more couples of counter-rotating shafts equipped with blades, where several teeth are able to engage the waste composite material and force it through the overlapping blades. Usually, the shredding rotors move with a low rotary speed to improve process efficiency by avoiding the material rebound on the blades and create, at the same time, a complex stress state characterised by shear, bending and torsion; these conditions can be furtherly modified by the distance between the blades, their width and the geometry of the teeth [97,98]. A defined combination of these process parameters is able to produce large and uniform flakes of composite materials, removing at the same time all inserts and fastenings usually embedded into the material [96]. The dimension of the shredded material is in the range of 50–100 mm, and it is usually in the form of flakes; therefore, further machining operations are required to obtain recovered fibres in the form of bundles. Based on these considerations, the shredded material is milled and ground, producing, respectively, fine fibrous recycled material with a dimension of almost 5 mm and powder material [16,97,99,100].

The hammer milling recycling process, along with shredding, are the most diffused methods for the mechanical recycling of composite materials. Compared to the shredding machine where the recycling material is crushed in small flakes, the hammer milling machine (Figure 11c,d) consists of a closed chamber where a rigid shaft is usually equipped with high-speed steel (HSS) cutting inserts that act as hammers on the recycling material [100,101]. Similar to the shredding process, hammer milling is characterised by a low rotary speed to avoid the rebound of the processing material and improve the efficiency of the process. The FRP waste is introduced into the chamber by hand, and it is milled until its dimensions are small enough to pass through a sieving plate localised on the bottom of the chamber [100,101]. At the end of the milling method, it is possible to obtain a recovered material that consists of resin-rich powder and fine fibre-rich recycled materials [100,101].



(a) (b) (c) (d)

Figure 11. Schematisation of the shredding process (**a**), example of multiple blade shredding (**b**), schematisation of hammer milling process (**c**), example of hammer milling inserts (**d**) [98,100].

A further satisfying separation of these categories of recovered materials can be obtained using a sieving process where interchangeable sieving grids that equip the milling and grinding machines allow for collection according to the dimension of the recycled material [41,83].

Therefore, at the end of each mechanical recycling method and the further sieving process, it is possible to obtain (i) a mixture of fine fibres with a length in the range of 2–20 mm, (ii) powders of composite material and (iii) additional coarse fibres with a length of almost 50 mm [102,103]. An appreciable separation in different dimensions of these recycled materials can be obtained using a sieving process. In detail, the selection by dimension is usually carried out using a rotary sieving cylinder or a section sieving shaking column assisted by airflow that allows fine fibre and powder separation [41,104]. Then, at the end of the sieving process, it is possible to distinguish recovered materials with different dimensions, such as resin-rich powders and fibre-rich recycled material. A typical schematisation of a mechanical recycling process is shown in Figure 12.

An overview of the mechanical recycling process leads to the conclusion that this method produces short, recovered fibres. Therefore, the geometrical characteristics of the recycled fibres, with the addition of other production factors, such as fibre concentration in the polymeric matrix and their orientation in the recycled composite, severely affect the overall mechanical properties of the recycled composite material [105]. All these aspects compromise the use of mechanically recycled composite as load-bearing structures, limiting their use sometimes as fillers in low-property composite materials such as SMCs and BMCs.

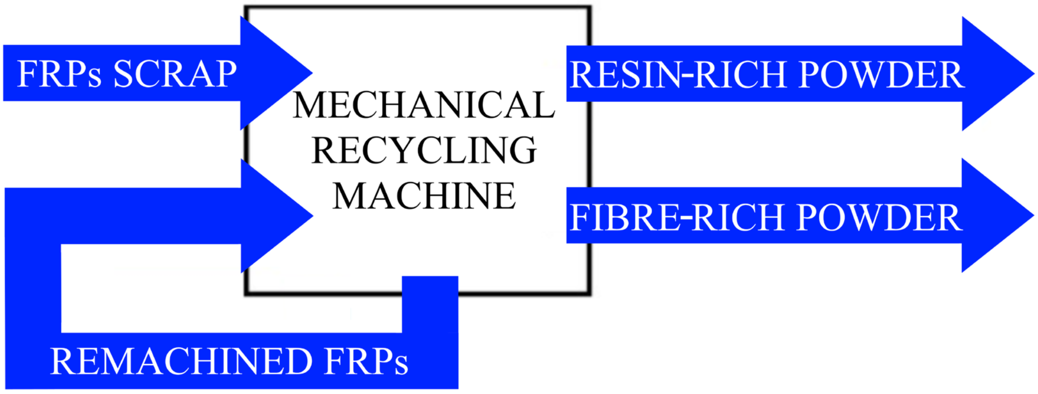


Figure 12. Schematisation of a typical mechanical recycling process.

However, the mechanical recycling process presents numerous advantages in terms of industrial scalability, economic process sustainability (especially referring to the energy demand that is estimated at around 0.1–4.8 MJ/kg) and environmental sustainability [55]. It is estimated that the mechanical method does not use any typologies of chemical substances and does not produce any kind of environmental pollution [41]. On the other hand, this recycling method is characterised by some disadvantages. First of all, it does not allow recycled fibre dimension preservation leading to recycled composite materials reinforced with short fibres; furthermore, it produces recovered fibres that contain matrix residues on the surface [52]. Therefore, this method leads to a recycled composite material characterised by reduced mechanical properties because of the low length-to-fibre diameter ratio and poor adhesion at the fibre–matrix interface as the recycled reinforcement is not completely cleaned [40,55,81].

If, on the one hand, the mechanical recycling method results are very attractive thanks to all the sustainability, environmental and economical perspectives, on the other hand, some aspects, such as the optimisation of the process parameters, the effects of machining on the residual properties of the recovered fibres and their efficiency in polymeric matrix removal require further investigations. Based on these considerations, some research works are focused on the mechanical recycling process of CFRPs and GFRPs and the use of recovered fibre in new composite products. For instance, Pietroluongo et al. [106] investigated the mechanical recycling process of end-of-life GF-reinforced PA66 thermoplastic matrix derived from automotive applications. The research group studied the effect of multiple mechanical recycling processes using the hammer milling method on the fibre dimension and mechanical properties in terms of tensile and flexural strength and modulus. All recycled samples used for both the tensile and the flexural tests were produced using an injection moulding technique with a temperature of almost 290 °C and a pressure of around 10 MPa. Starting from a short GF composite material, a mean fibre length reduction of almost 31% was observed after the first recycling process, then a mean dimension reduction of almost 15% in comparison with the first recycled composite, both in the case of the second and third recycling processes. At the end of the experimental campaign, Pietroluongo et al. observed that the first recycled composite material demonstrates a reduction in the tensile strength and modulus of almost 29 and 23%, respectively. A reduction in the flexural strength and modulus of 28 and 24% were found, respectively, in comparison with the virgin material. Then, there was a progressive reduction of the tensile strength and modulus of almost 5 and 10% and flexural strength and modulus of around 6 and 8% after the second and third recycling cycles. In addition to the mechanical tests, during the experimental campaign, further tests were carried out on the injected samples to evaluate the fibre length and distribution. The result is that during the composite extrusion, the fibre orientation along the injection direction can occur because of the viscosity of the matrix. However, despite the fibre orientation phenomenon, the research group pointed out that with the reduction in the fibre dimensions, the load transferring efficiency between fibres and the polymeric matrix falls; therefore, when the aspect ratio decreases, the reinforcement is no longer able to contribute to matrix stiffening.

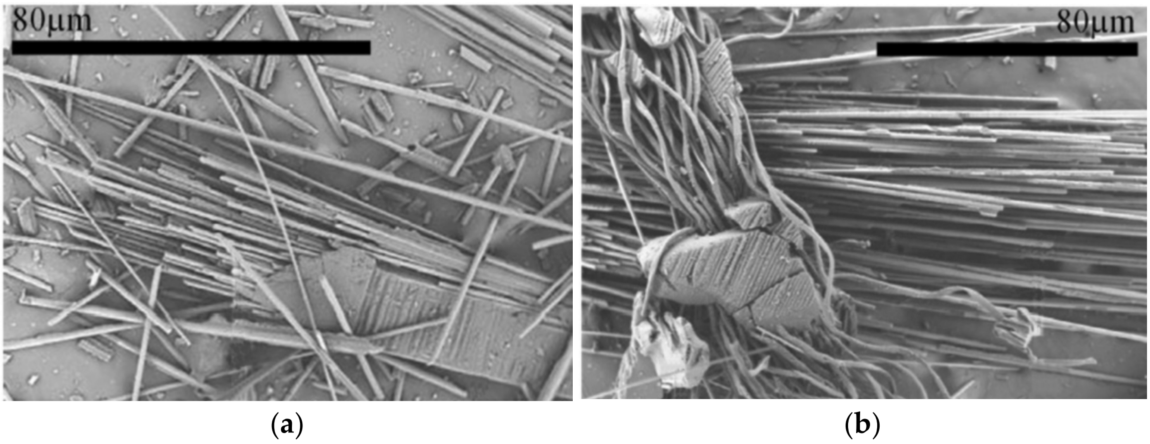
As well as Pietroluongo et al., Beauson et al. [103] investigated mechanical recycling of GFs derived from end-of-life of wind turbine blade structures. In their study, the fibrous reinforcement was shredded and then sieved in different dimensions aiming to understand the mechanical properties of rGFs reinforcing polyester resin. The choice of the specific polymeric resin was carried out to avoid any kind of alteration of the mechanical properties due to the different nature of the employed resins. In this experimental campaign, different sample typologies were studied by varying the amount of fibre weight fraction; in detail, 10, 20 and 30% of recycled fibres were used to manufacture recycled laminates by means of a special vacuum infusion technique. Before the impregnation process, the research group investigated the morphology of the rGFs and pointed out that the shredded material presents some old polymeric matrix residues on the fibre surface (Figure 13).

Tensile tests were carried out on the recycled composites, concluding that all recycled samples are characterised by a lower tensile strength when compared with virgin polyester resin. The reduction in the tensile strength varied from around 41 to 69% depending on the amount of recycled reinforcement, which is far from the expected improvement in the range of 65 and 140%. Based on these results, Beauson et al. investigated the tensile fracture surface of recycled composites (Figure 14).

From an inspection of the fracture surface, the research group pointed out that in the occurrence of the failure of the specimen, debonding and pull-out phenomena at the interface between fibres and the matrix influence the mechanical performances, leading to the premature failure of the specimen.

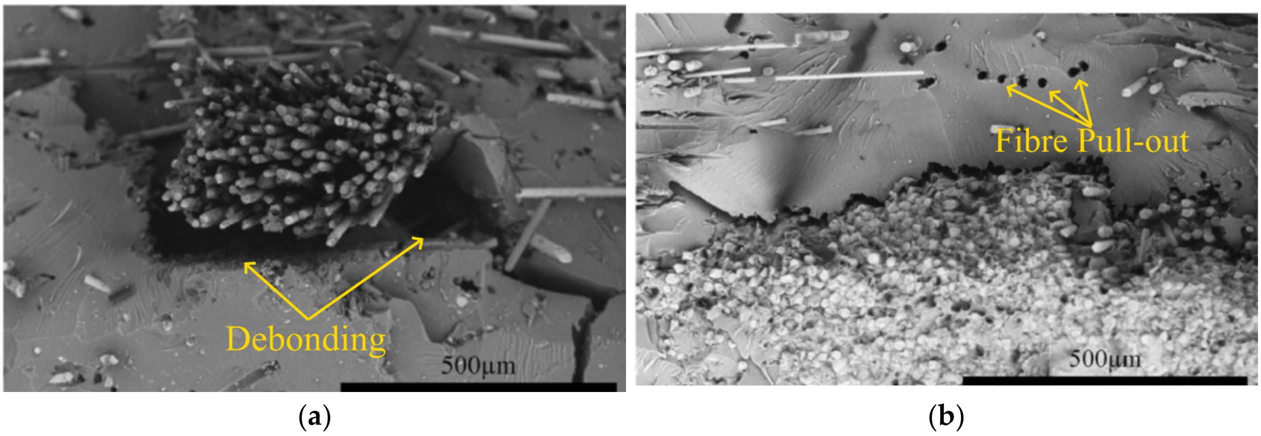
Based on these observations, the research group concluded that the premature failure of the recycled material is linked to the poor adhesion between the shredded material and the new polymeric resin; therefore, alternatives to improve the interface efficiency are physical or chemical treatments to activate the surface of the recycled materials.

Palmer et al. [107] investigated the possibility to produce SMC composite materials using rCFs in place of GFs. A rotating hammer milling process was used to produce rCFs, and then they were classified into four different dimensions using a "zig-zag" vibration sieving apparatus. At the end of the recycling process, the research group obtained recovered materials with different dimensions, such as resin-rich powders with recovered fibres 0.5 mm long, fibre-rich material with dimensions in the range of 5–10 mm and coarse fibres with a length greater than 10 mm. rCFs, 5–10 mm in length, were used by the research group for the comparison of recycled SMC materials with the reference one. For the experimental phase, four different sample typologies were used including (i) a traditional SMC composite material as a reference, (ii) an SMC with rGFs of 20% by weight, (iii) an SMC panel reinforced with rCFs of 20% by weight (6.5% by volume) and (iv) an SMC sample reinforced with rCFs of 4.6% by volume. The amount of reinforcement used in the last typology was evaluated by replacing the same amount of virgin fibres in the SMC reference with rCFs; however, these fibres are characterised by a reduced specific weight when compared with GFs. Hence, the volume fraction of the recycled fibres used to reinforce the SMC panel is lower. Flexural and Charpy impact tests were performed on all samples, concluding that the SMC material with 20% by weight of rCFs is characterised by a reduction in impact strength of around 20%, a reduction in flexural strength of almost 14% and an improvement of 15% in the flexural modulus. On the other hand, the SMC sample with 4.6% by volume of rCFs revealed mechanical properties comparable with the reference, with a reduction of around 3 and 9% of the flexural modulus and flexural strength, respectively. An improvement in the impact strength of 7% was also detected. The difference in the mechanical properties of the SMC samples with rCFs can be attributed to the amount of reinforcement; indeed, when 20% by weight of recycled CFs is used, an effective fibre impregnation cannot be reached due to the poor amount of polymeric matrix. This condition, in addition to the reduced fibre–matrix adhesion efficiency that characterises mechanical recycled fibres, leads to the overall mechanical property reduction. However, at the end of the experimental campaign, the research group concluded that the SMC material reinforced with rCFs possesses mechanical properties that are comparable with the sample used as a reference, and it could be used as its substitute.



(**a**) (**b**)

Figure 13. SEM images of shredded rGFs: presence of old polymeric matrix on the fibre surface (**a**); matrix particle on the fibre bundle (**b**) [103].



(**a**) (**b**)

Figure 14. SEM image of the tensile fracture surface of rGF-reinforced polyester resin composite: debonding phenomenon (**a**); fibre pull-out (**b**) [103].

Thomas et al. [108] studied the possibility of using carbon powder waste in epoxy resin, aiming to increase its mechanical properties. The rCFs derived from mechanical cutting processes on composite laminates were subjected to a grinding method and then classified using a sieving process. The research group defined a maximum fibre dimension of almost 1.25 mm; therefore, all fibres greater in dimension were excluded from this research work. Two main sample typologies were produced by adding 10 and 20% by weight of recycled fibres, and then the mechanical properties of these samples were compared with the reference sample produced with the same epoxy matrix. At the end of the experimental campaign, Thomas et al. pointed out that the epoxy sample reinforced with 20% of recycled material possessed improved mechanical properties in terms of compressive and flexural strength. The 20% reinforced epoxy matrix demonstrated an improvement of around 20 and 30% of compressive and flexural strength, respectively, in comparison with the pure resin sample, pointing out that the presence of almost 20% of rCFs is able to generate a better load transfer mechanism within the epoxy matrix.

Durante et al. [109] investigated the recycling of CFRP materials using a peripheral down-milling process by adopting different cutting parameters aiming to understand how the process parameters influence the specific cutting energy and dimension of the recovered fibres. At the end of the recycling process, rCFs of different dimensions were obtained, but only fine fibres (with a size smaller than 0.3 mm) and coarse fibres (with a size bigger than 0.3 mm) were used to produce recycled composite materials using an epoxy resin as the matrix. The authors pointed out that the lowest value of specific cutting energy is reached when the worst cutting conditions are adopted and then when the higher feed per tooth is used. The flexural tests revealed that with respect to the pure resin, both the sample typologies were characterised by an increase in the flexural modulus of almost 80 and 160% in the case of fine and coarse fibres, respectively. However, focusing on flexural strength, it was observed that only samples produced with coarse fibres revealed an improvement with respect to the epoxy matrix of almost 45%.

The research group attributed the reduced mechanical properties that characterise the fine fibre-reinforced sample to the geometrical properties of the recycled material; in detail, the short fibres that are almost in the form of particles, in addition to the presence of physical porosities, act as points where internal cracks start to nucleate and propagate leading to premature sample failure. However, the reduced mechanical properties were attributed not only to the fibre dimension but also to the well-known poor adhesion efficiency at the interface caused by the presence of residues of the old matrix on the recovered fibre surfaces.

The results of the most used mechanical recycling processes in terms of fibre dimension and mechanical properties of the recycled composite materials are shown in Table 6.

Table 6. Main results deriving from mechanical recycling methods.

Material	Recycling Method	Fibre Dimension	Mechanical Characteristics	Ref.
GF—PA66	Hammer milling	Short fibres–length reduction of 31% after the first recycle, reduction of 15% after further recycles	Tensile strength −29% and Young's modulus −23% Flexural strength 28% and flexural modulus 24% reduction after the first recycle	Pietroluongo et al. [106]
GF—Polyester	Shredding	Coarse fibres Fine fibres Powder	Flexural strength −41% and −69% as function of the fibre percentage	Beauson et al. [103]
CF—Epoxy	Hammer milling	Coarse fibres Fine fibres Powder	Flexural strength −9%, flexural modulus −3% and impact strength +7%	Palmer et al. [107]
CF—Epoxy	Grinding	Powder	Flexural strength 30% and compression strength 20% improvement in comparison with pure resin	Thomas et al. [108]
CF—Epoxy	Peripheral down-milling	Coarse fibres Fine fibres Powder	Flexural strength −15% (fine fibres), +45% (coarse fibres) flexural modulus +80% and +160% (fine and coarse fibres, respectively)	Durante et al. [109]

Looking at Table 6, it is possible to conclude that in all cases, the recycled composite materials are characterised by a severe reduction in mechanical properties when compared with the materials used as references. Therefore, if, on the one hand, this aspect is first related to the reduced dimension of the recovered fibres due to the mechanical milling, on the other hand, it is related to poor adhesion at the interface between the recovered fibre and the fresh resin.

However, in contrast with the chemical and thermal recycling methods, the mechanical one does not require high processing temperatures as it is performed at room temperature. This aspect makes it a suitable method to recycle both CFs and GFs without any problems regarding the chemical alteration or thermal instability of the recycled fibres. On the other hand, the mechanical recycling process is based on composite fragmentation, and the recovered fibres are usually characterised by short dimensions in a range that varies from powder to coarse fibres with a maximum length of almost 50 mm. Therefore, the recycled composite materials are reinforced with short fibres that limit the use of these materials in applications where high performances are required.

Currently, some aspects of mechanical recycling are still not clear, such as the mechanical properties of the recovered fibres that are not well defined as well the effects of the sieving grid geometry on the final aspect ratio of the fibres. Further investigations need to be performed to clarify how the process parameters influence the mechanical properties of the recovered fibres, matrix removal rate and the efficiency of the interface between the recycled material and the new resin.

However, based on a literature overview of the mechanical recycling methods, it is possible to conclude that all the mechanical recycling processes are characterised by numerous advantages in terms of environmental sustainability, low specific energy demand and the grade of industrial maturation.

The recycling methods discussed above and the main resulting outcomes in terms of the recovered fibre dimensions and mechanical properties of the recycled composite materials are summarised in Table 7.

Table 7. Main recycling methods of CFRP and GFRP composite materials.

Material	Recycling Method	Fibre Dimension	Mechanical Characteristics	Ref.
GF—Polyester	Subcritical solvolysis	Long fibres	Mechanical strength 50–60% reduction	Oliveux et al. [53]
GF—Epoxy	Microwave-assisted subcritical solvolysis	Long fibres	Tensile strength −7%	Zabihi et al. [62]
GF—Epoxy	Subcritical solvolysis	Long fibres	Tensile strength −10%	Kuang et al. [63]
CF—Epoxy	Supercritical solvolysis	Long fibres	Mechanical properties 2–10% reduction	Pinero-Hernanz et al. [57]
CF—Epoxy	Supercritical solvolysis	Long fibres	Comparable with virgin fibres	Liu et al. [64]
CF—Epoxy	Supercritical solvolysis	Long fibres	Tensile strength 18–36% reduction	Kim et al. [59]
CF—Epoxy	Electrochemical	Long fibres	Tensile strength −10% and +20% interfacial shear strength	Zhou et al. [69]
CF—Epoxy	Electrochemical	Long fibres	Fibre fragmentation	Oshima et al. [70]
CF—Epoxy	Electrochemical	Long fibres	Fibre fragmentation	Oshima et al. [71]
GF—Epoxy	Microwave-assisted acid digestion	Long fibres	Tensile strength −2.4% and Young's modulus −8.6%	Rani et al. [75]
CF—Epoxy	Acid digestion	Long fibres	Mechanical properties reduction 2–10%	Xu et al. [76]
CF—Epoxy	Acid digestion	Long fibres	Tensile strength −8%	Zabihi et al. [77]
GF—Epoxy	Pyrolysis	Long fibres	Flexural strength −19%	de Marco et al. [84]
CF—Epoxy	Pyrolysis and post-pyrolysis	Long fibres	Tensile strength −7% and Young's modulus −4%	Nahil and Williams [85]

Table 7. *Cont.*

Material	Recycling Method	Fibre Dimension	Mechanical Characteristics	Ref.
CF—Epoxy	Pyrolysis	Long fibres	Tensile strength +6.4% and Young's modulus +5%	Mazzocchetti et al. [87]
CF—Epoxy	Pyrolysis	Long fibres	Tensile strength −20%	Yang et al. [88]
CF—Epoxy	Water stream-assisted pyrolysis	Long fibres	Tensile strength −10%	Kim et al. [89]
GF—Epoxy	Microwave-assisted pyrolysis	Long fibres	Flexural strength −68% and flexural modulus −39%	Akesson et al. [90]
CF—Epoxy	Microwave-assisted pyrolysis	Long fibres	Flexural strength −14% and flexural modulus −25%	Jiang et al. [91]
GF—Polyester	Fluidised bed	Short fibres	Flexural strength −50%	Pickering et al. [93]
CF—Polyester	Fluidised bed	Short fibres	Flexural strength 50% reduction–no appreciable variation in flexural modulus	Kennerley et al. [94]
CF—Epoxy	Fluidised bed	Short fibres	Tensile strength −18%	Pickering et al. [95]
GF—PA66	Mechanical–hammer milling	Short fibres	Tensile strength −29% and Young's modulus −23% Flexural strength −28% and flexural modulus −24% after the first recycle	Pietroluongo et al. [106]
GF—Polyester	Mechanical–shredding	Coarse fibres Fine fibres Powder	Flexural strength 41–69% reduction as function of the fibre percentage	Beauson et al. [103]
CF—Epoxy	Mechanical–hammer milling	Coarse fibres Fine fibres Powder	Flexural strength −9% and flexural modulus −3% and impact strength +7%	Palmer et al. [107]
CF—Epoxy	Mechanical–grinding	Powder	Flexural strength +30% and compression strength +20% in comparison with pure resin	Thomas et al. [108]
CF—Epoxy	Peripheral down-milling	Coarse fibres Fine fibres Powder	Flexural strength −15% (fine fibres), +45% (coarse fibres) flexural modulus +80% and +160% (fine and coarse fibres, respectively)	Durante et al. [109]

3. Conclusions and Future Perspectives

The recycling of end-of-life and offcut waste composite materials is a relevant aspect for the sustainable use of these materials in industrial applications, such as in the transport, automotive, aeronautic and marine sectors. Considering different industrial fields, it came to light that for marine applications, CFRPs and GFRPs are the most used types of composite materials for which recycling represents a mandatory aspect to consider for the future. Based on these considerations, the present state of the most mature recycling processes, including chemical, thermal and mechanical methods used for CFRPs and GFRPs, was analysed in this review. Each of these technologies was discussed in detail, analysing the required time, energy consumption, environmental impacts, suitable materials and final properties of the recycled components.

At the end of the present overview, it was pointed out that the mechanical properties of the recovered CFs are not strongly influenced by chemical and thermal recycling methods as they preserve a large part of their mechanical properties in comparison with virgin fibres. However, the recovered CFs are not usually used as reinforcement in structural composite materials.

The state of the art on the recycling of composite materials highlights that:

- Chemical and thermal recycling processes require high energy consumption and appropriate laboratory apparatus and are affected by severe environmental impacts;
- The mechanical recycling method, even if it leads to composite materials reinforced with short fibres, appears to be an appealing method to recycle CFs as it allows the above-mentioned aspects to be overcome;
- GFs are strongly affected by chemical and thermal methods as a severe reduction in the overall mechanical properties was observed as a consequence of the recycling processes. This aspect makes these recycling processes unattractive for the recovery of GFs;
- The mechanical methods for GFRPs, even if they produce recycled composite materials reinforced with short fibres and are characterised by reduced properties, they do not lead to a drastic collapse of the mechanical properties because the recovered fibres are not affected by thermal alteration;
- Among the recycling methods treated in this review, the mechanical ones, such as shredding, hammer milling, milling and grinding, are the more appreciable recycling processes to recover GFs;
- Chemical and thermal processes are more prone to industrial scalability thanks to the high volume of recycling material processed; however, there are critical aspects to be considered about energy consumption and environmental impacts;
- Further advances in these recycling processes are not expected in the imminent future as the optimised parameters in terms of chemical solutions (in the case of chemical recycling) and oxygen amount (in the case of thermal recycling) are largely affirmed.

The optimisation of recycling processes and the improvement of the interfacial efficiency between fibres and the matrix are required as future perspectives. Innovative and economically competitive recycling methods are effectively required to recover competitive rCFs and rGFs and produce composite components that contain recycled fibres, leading to a close-loop process in accordance with the philosophy of the circular economy.

Indeed, new mechanical recycling methods are still under investigation, and future innovations will be usable in this area in the coming years.

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