Thermal Demanufacturing Processes for Long Fibers Recovery

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Abstract The possibility of recycling glass (GF) and carbon fibers (CF) from fiber-reinforced composites by using pyrolysis was studied. Different fibers from composite waste were recovered with thermal treatment. The recycled fibers were evaluated as a reinforcement for new materials or applications. The main objective was to evaluate the fibers obtained from the different types of industrial composite waste considering the format obtained, the cleanliness and the amount of inorganic fillers and finally, the fibers quality. These characteristics defined the processes, sectors and applications in which recycled fibers can replace virgin fibers. These fibers were also evaluated and validated with tensile testing and compared to the tensile strength of virgin GF and CF.

Keywords Pyrolysis · Recycled fibers · Recycled carbon fiber · Recycled glass fiber

1 Introduction

GF and CF composites are interesting materials, they have many attractive properties such as high strength, good corrosion resistance, and light weight. They are widely used for applications such as automobiles, building insulation, leisure boats, construction materials, and leisure products [1]. The increase in the use of GF and CF composite materials due to their interesting properties and their light weight has generated an increment in waste of component at the end of life. Recycling of this

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waste is complex due to its inherent non-homogeneous nature. However, today there are different methods to recycle composite materials through mechanical, thermal, chemical processes and combinations of them [2].

Carbon fiber composites are currently being thermally recycled by companies such as ELG, with good results and with a potential market yet to be explored. Universities, Research Centers and companies have researched on composites recycling in the last 10 years [3]. The industrial thermal recycling of GF composites is currently challenging, mainly due to the low cost of the virgin raw material, that currently hinder obtaining an economically viable fiber material. This is the reason why the GF is recycled mainly by mechanical processes. Alternative processes are the subject of many research, particularly chemical processes which can recover value from the resin chemicals. Also, variants of pyrolysis, e.g. with a fluidised bed, or using microwave energy, have potential to provide cleaner fibers or use less energy. Environmental impact of different recycling processes shows that energy demand for chemical processes is typically higher than others. Thermal recycling is in the intermediate range, but only around 10% of the energy input required to produce virgin fiber. Mechanical grinding uses very little energy in comparison but produces a lower value product.

For CF there is scope to introduce variants of pyrolysis processing to optimize fiber surface properties and reduce energy consumption. The thermal process conditions for recover composites affects directly to the mechanical properties of the recycled fibers. In the case of recycled carbon fiber (rCF) the tensile strength could be reduced from 40 to 90% and in the case of recycled glass fiber (rGF) from 52 to 64%. Moreover, the thermal process removes the sizing from the fiber that must be replaced for a new sizing if the recycled fiber wants to be used in a technical application. The University of Strathclyde studied a suitable post-treatment for rGF that would permit their use in compounding application for automotive, but it would be necessary to scale-up this treatment with an economic feasibility able to compete with the virgin GF [4].

The present study has focused on developing and validating first, a preliminary laboratory-scale pyrolysis process for analysis the recovery of long GF and CF and after that, a pilot plant pyrolysis technology to validate the fiber in industrial application. In some of the cases it has been started with complete components and in another case the waste has been previously chopped. Thermally recovered GF and CF have been reused in high-strength, high-tech applications for the automotive and construction industries (Chap. 16). This study has focused on analysing the requirements and technical aspects necessary to obtain good quality rGF and rCF that can be used later in typical processes for plastics and composites sector.

1.1 Existing Pyrolysis Processes for Long Fiber Composites

Carbon fiber reinforced plastics (CFRP) pyrolysis is exploited by companies as ELG Carbon Fibrein UK, CFK Valley Stade Recycling and Hadeg Recycling Ltd both in

Germany, Carbon Conversions and Material Innovation Technologies RDF in USA or Karborek in Italy [5]. CFs are recovered in a controlled temperature and atmosphere furnace. They obtain fiber with 90% of the virgin properties [4].

Other companies, as Reciclalia or Formoso Technologies Group in Spain, Rymyc in Italy or Carbon Fiber Recycle Industry Co Ltd in Japan have pilot plants for fiber recovery [2]. Reciclalia for example, is working in the feasibility of recycling GF and CF Eolic blades to obtain long fibers to manufacture new hybrid thermoplastic fabric with high quality for automotive sector.

Figure 1 shows a CF recycling plant [6]. In this plant different stages are shown from the CF waste to the final product:

- Presorting: Crushing and sorting of materials according to type of fiber and state of processing: dry CF scraps, prepreg materials, end-of-life parts.
- Pyrolysis: Thermal treatment excluding oxygen in order to recover pure CF completely by means of thermal oxidation of pyrolysis gases.
- Refinement: Customized conditioning of fiber surface
- Cutting: Processing of CF into models "chopped" and "milled" accurately cut to desired fiber length
- Final product: At the end of the production process the client receives a customized product which absolutely fulfills the requirements

This means that not only the cost of the pyrolysis process must be considered in the final price of the rCF, but also of the rest of the pre-processes and post-processes.

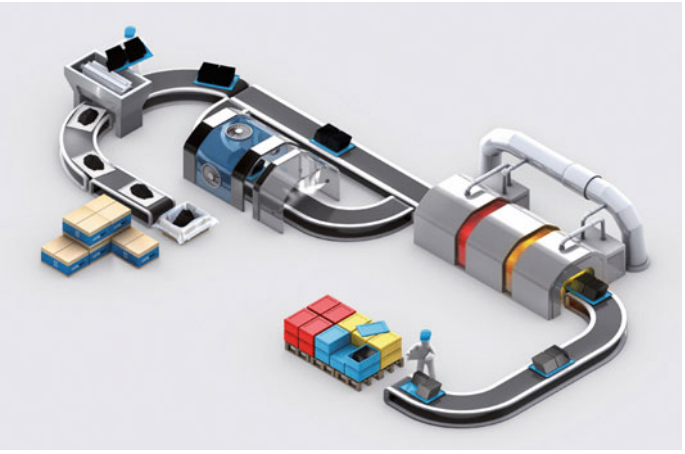


Fig. 1 CPK—valley pyrolysis plant [6]

1.2 Basic Principles of Thermal Demanufacturing Processes

Thermal processes include pyrolysis, fluidised-bed pyrolysis and pyrolysis assisted with microwaves [2]. Pyrolysis process consists of the chemical decomposition of the resin in an atmosphere without oxygen (e.g. nitrogen) and at high temperatures between 450 and 700 °C [7]. In these conditions, the resin (matrix) does not burn, it decomposes into lower-weight molecules and different sub-products are produced such us carbon dioxide, hydrogen and methane for example, and an oil fraction [2] that sometimes can be used by the petrochemical industry. Also, these molecules evaporated from the material can be used as an energy source because of their high heat capacity. Some studies suggest that the heat energy of the resin can be recovered, making the pyrolysis process self-sustaining [5]. Under these conditions, the fibers do not decompose, which makes this process interesting for recycling the fibers. However, several factors must be considered. GF suffer from the high temperatures and their mechanical properties can decrease. CF are less sensitive to temperature but alongside the CF, pyrolytic carbon coexists, which influences the mechanical properties of the regenerated fibers and depends to a large extent on the process parameters (furnace atmosphere, temperature, heating ramps and others). It is therefore possible to influence the properties of the fiber by acting on these parameters. This means that process optimization can and should be adapted to the mechanical requirements of the parts in which the recycled fibers are to be incorporated [8]. The parameters to consider are:

Temperature: Temperature is an important parameter in all stages and, therefore, in the final yield of the process. In particular, the proportions between solids, liquids and gas in the pyrolysis product are closely dependent on heating rate and the final temperature reached. At high heating rates and high final temperatures, most gas is produced, while at lower end temperatures and heating rates, most liquids or solids are produced.

Pressure: An increase in pressure makes gasification reactions more difficult, increasing the proportions of hydrocarbons and tars. Moving bed gasifiers usually operate at atmospheric pressure and fluidized bed gasifiers usually operate at pressure, reaching up to 30 bar in some cases.

Humidity: Moisture influences the thermal balance of the process as part of the heat produced must be used to evaporate this amount of water. It also influences the composition of the flue gases, even displacing some reactions.

Apparent density of material loads: Properly cut CFRP waste should be placed on trays, hooks, or charges. This implies a loss of volume to be treated in each batch with respect to the theoretical capacity of the kiln. The free spaces facilitate the gasification of organic matter and the movement of these gases towards the upper part of the installation for their elimination by burning.

Pyrolizer opening temperature: Cycle time on an industrial scale depends on the heating and cooling phases. The heating time depends mainly on the power of the oven. The cooling time depends exclusively on the temperature at which the system is opened. A quick opening will mean a higher emission of unburned gases into the

Table 1 Description of the studied material

Ref.	Quantity	Sector	Fiber/matrix and others	Manufacturing process	Part detail
A1 A2	200 g 9 kg	Construction	GF/polyester	Lamination	End of life composite roof
B	25 kg	Automotive	30% GF/polyester, inorganic fillers	SMC	Defective parts
C1 C2 C3	7.4 kg 130 kg 250 kg	Wind sector	GF/epoxy, foam, cores and gelcoat	Resin infusion	Dismantled blade part
D	20 kg	Aeronautical	CF/ epoxy, protective films	Uncured prepreg	Cutting operations scraps
E	150 kg	Aeronautical	CF/epoxy, protective film	Uncured prepreg	Expired prepreg
F1 F2	8 kg 150 kg	Aeronautical	CF/epoxy	Autoclave	Defective part

atmosphere. Depending on the nature of these gases (irritability, toxicity, etc.), the optimum opening temperature is determined.

2 FiberEUse Pyrolysis Processes

2.1 Materials

In the FiberEUse project, composite material waste from different sectors were considered as study materials. The objective was to recover clean fibers after removing the polymer matrix. Table 1 shows a summary of the waste materials studied together with their main characteristics.

The above classification helps to estimate the amount of fiber that is expected to be obtained after the thermal recycling process. It was also possible to predict the final format of the fiber that would be obtained or even the degree of cleaning. In this way, from wastes that contain a high amount of inorganic fillers, it is expected to obtain dirty fibers or from wastes with fabrics in their original textile format is expected to be recovered.

2.2 Pyrolysis Equipment and Media

Different types of ovens were used for the heat treatments of the different described materials. First, small laboratory equipment was used and later bigger ovens, all

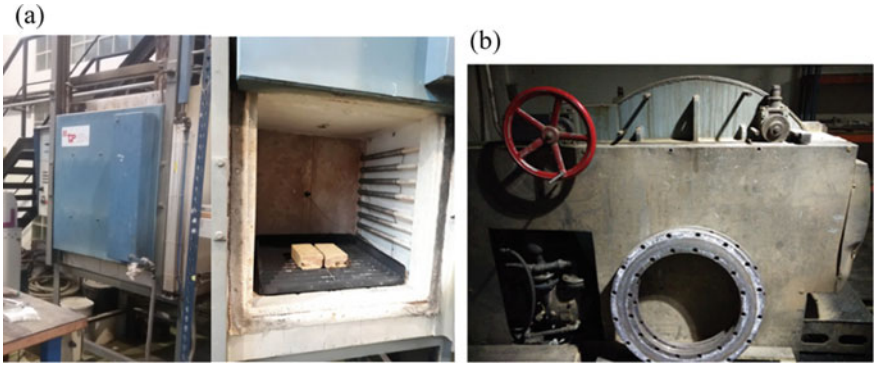


Fig. 2 **a** HC500 Oven for thermal treatment. **b** Semi-industrial oven Solvo Line 166

of them in TECNALIA. Finally, pilot plant was installed in RIVIERASCA. The GF materials were treated initially in ovens with the presence of air, while the CF composite materials were pyrolyzed in ovens with inert atmosphere.

Ovens with air atmosphere (Fig. 2):

- A laboratory oven HC500 with unforced air circulation and with dimension of 600*600*600 mm and maximum working temperature of 500 °C was used for small GF composite samples.
- Semi-industrial oven consisting of a VOCs (Volatile Organic Compounds) treatment system and SOLVO Line 166-Type 120 oven 600*600*1000 mm, loading capacity of 500 kg/m and equipped with an automatic unit control, able to work to 500 °C and 0.65 bar was used for large GF composite samples.

Ovens with inert atmosphere (Fig. 3):

- Small laboratory oven for the pyrolysis of small CF composite coupons. It is composed of a glass tube resistant to high temperature in which a controlled flow of argon circulates. It is jacketed with an resistors oven capable of reaching 500 °C.
- Nabetherm lab oven with a continuous nitrogen flow and a heating rate of 11.5 °C/min. The internal dimensions are 200*200*200 mm.
- Nabertherm semi-industrial oven. Argon Oven with gas extraction and filters to concentrate liquid/solid sub-products. The oven internal chamber has a dimension of 1000*500*250 mm.
- Industrial plant: oven with inert atmosphere and a capacity of 500 kg
- Pilot plant KOREC: thermochemical depolymerization in the presence of a controlled carbon dioxide environment that allows to recover both the inorganic part (carbon fibers or glass fibers) and the organic part (resin) in the form of an organic liquid fraction. This organic liquid contains C=C unsaturations and can be co-formulated with virgin polyester resins and take part in the following polymerization reactions that produce new thermosetting composites. This process has been developed and patented by Korec (EP 3114191, 2018). The innovation in

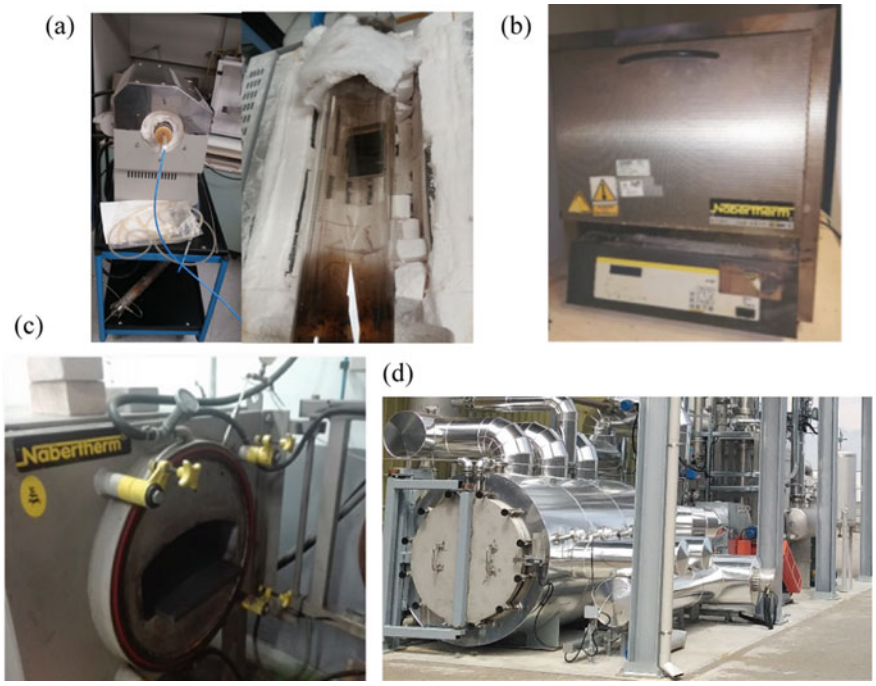


Fig. 3 **a** Pyrolysis lab oven with argon circulation. **b** Nabetherm nitrogen oven. **c** Nabertherm Argon Oven. **d** Korec industrial pilot plant

the Korec process lies in the recovery of resin fraction that, as a reactive blending component, can be reintroduced into the production chain of composites and this is what makes the process more economically sustainable. CO_2-containing environment with a predetermined CO_2 volume concentration. The capacity of the pilot plant is 950*950*2500 mm.

2.3 Methodology and Process Parameters

In order to obtain different fiber formats as a product of the thermal process or pyrolysis, the samples were introduced in the ovens with different sizes. The target format is related to the future use of the fiber. Thus, for laminating, resin infusion or RTM (Resin Transfer Moulding) processes, the preferred formats are mat or fabric, while for compounding processes (extrusion-injection processes) the preferred format is shredded fiber between 6 and 15 mm. The samples from construction (A), Automotive (B), Wind (C1) and Aeronautic (D and E) have been pyrolyzed in their original format, that is, as the waste was received. However, the samples from Wind (C2 and C3) and Aeronautic (F1 and F2) have undergone a preliminary shredding process

Table 2 Processes parameter

Equipment / Parameters↓		HC50 oven	Solvo Line pilot plant	Glass tube lab oven	Nabertherm pilot plant	Nabertherm oven	Ind. plant	Korec pilot plant
Ramp 1	Heating rate (°C/min)	2	30	3.5	11.5	11.5	11.5	10–12
	Temperature (°C)	600	320	450	450	450	450	350–450
	Time (h)	4	0.83	2	0.62	0.62	0.62	0.55–0.60
Ramp 2	Heating rate (°C/min)	–	5.3	–	–	11.5	–	–
	Temperature (°C)	–	400	–	–	550	–	–
	Time (h)	–	5	–	–	0.15	–	–
Ramp 3	Heating rate (°C/min)	–	20	–	–	–	–	–
	Temperature (°C)	–	500	–	–	–	–	–
	Time (h)		0.1		–	–	–	–
Stabilisation Time (h)		2	6.5	1	2.5	30 (Ramp1) +15 (Ramp2)	6	0.2
Atmosphere		Air circulation	Vacuum + air circulation	Argon	Argon	Ramp1: Nitrogen Ramp 2: Air	Inert	CO_2
Cooling		Yes	Yes	Yes	Yes	Yes	Yes	No

(see in this chapter) to obtain shredded composite material with the defined size (6–15 mm).

The cycles used in each of the oven have been adjusted to the characteristics of the oven/technology itself, such as the rate of heating, the capacity or volume or the type of atmosphere. The parameters used are summarized in Table 2:

3 Description of the Process for Each Material

3.1 Material from Construction Waste (Ref. A1)

This waste was introduced into the oven without any previous process. An oven with air atmosphere (HC500) was used for the heat treatment. After the defined cycle, GF were obtained in mat format with no apparent residues (Fig. 4).

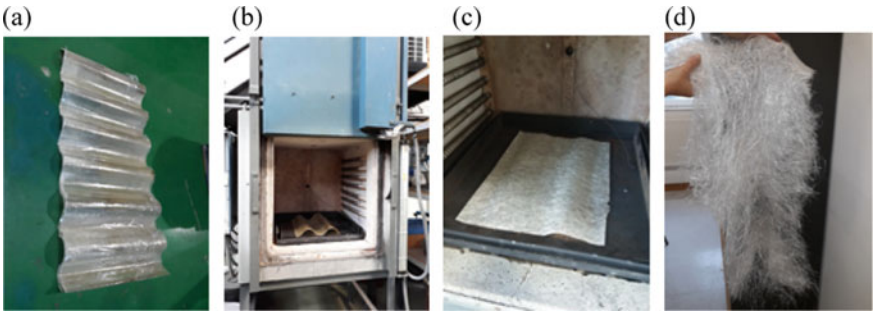


Fig. 4 **a** Waste Ref. A1, **b** Ref. A1 in HC500 oven, **c** Obtained product, **d** Mat of rGF

Fig. 5 **a** Waste Ref. A2 in Solvo Line oven tray, **b** Obtained product, **c** Clean rGF with mat format

3.2 Material from Construction Waste (Ref. A2)

In the same way, this waste was introduced into the oven without any previous process. An oven with air atmosphere (Solvo Line) was used for the heat treatment. After the defined cycle, the results were the same in both ovens, although the waste amounts treated were much higher (Fig. 5).

3.3 Material from Automotive Waste (Ref. B)

This waste was also introduced into the oven without any previous process. Again, an oven with air atmosphere (Solvo Line) was used for the heat treatment. After the defined cycle, GF were obtained in their original format, this long cut fibers. In this case, the high amount of inorganic fillers contained in the resin did the recovered fiber especially dirty and contaminated (Fig. 6).

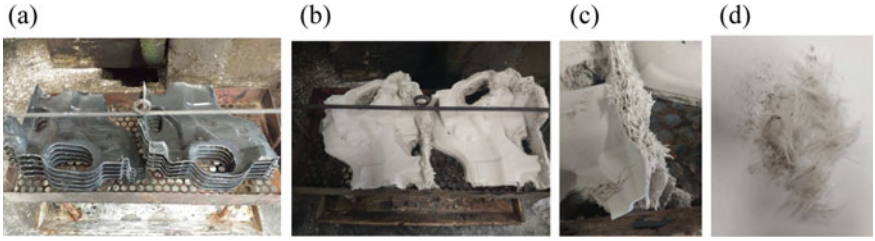


Fig. 6 **a** Waste Ref. B in Solvo Line oven tray, **b** Obtained product, **c** and **d** Long rGF with high filler content

3.4 Material from Wind Sector Waste (Ref. C1)

This material from large wind blades (length between 10 and 40 m) was previously cut by the owner company to optimal sizes for this study (maximum 0.5 m), but it was not treated with a shredding process. Again, due to the material contained GF, an oven with air atmosphere (Solvo Line) was used for the heat treatment. After the defined cycle, GF were obtained as a mix of different formats. This could be explained because the production process uses fibers in very different formats as roving, mat or fabrics. Furthermore, the presence of other materials such as foam cores and especially, gelcoats make the fiber obtained with a variable percentage of inorganic fillers. In this case, because the content of fillers is not very high, it can be removed by a mechanical process (Fig. 7).

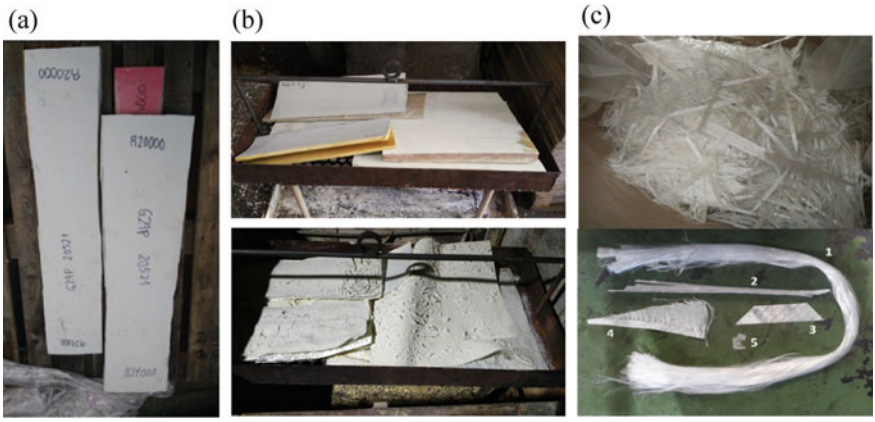


Fig. 7 **a** Waste Ref. C1, **b** Ref. C1 after and before the cycle, **c** Obtained product (mix formats)

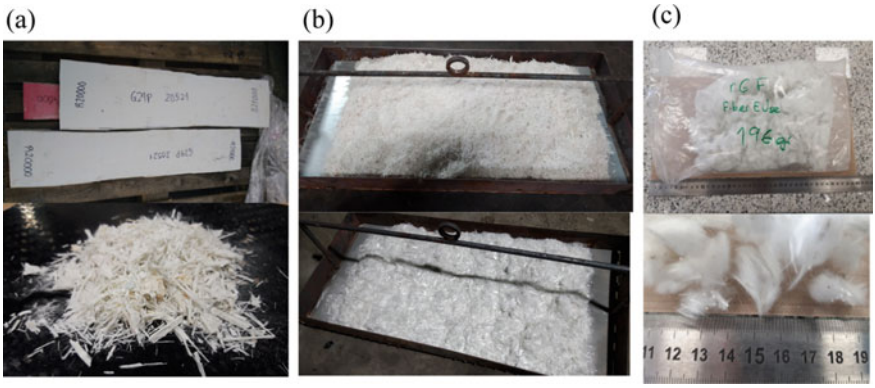


Fig. 8 **a** Waste Ref. C2, **b** Ref. C2 after and before the cycle, **c** Obtained short rGF

3.5 Material from Wind Sector Waste (Ref. C2)

This material was the same that the previous one, but in this case, it was treated with a shredding process. The same oven (Solvo Line) with air atmosphere was used for the heat treatment. In this case a tray with small holes for aeration was needed. After the defined cycle, short GF were obtained with length between 6 and 15 mm. The fiber was obtained quite clean, although some agglomerates of inorganic fillers were observed (Fig. 8).

3.6 Material from Aeronautic Waste (Ref. D)

This material was introduced into the oven without any shredding process. First, some small coupons of this material were used in the glass tube laboratory oven with inert atmosphere. The results obtained after the defined cycle were small pieces of clean CF fabric. The rest of material was introduced in Nabertherm argon oven. In this case the obtained fibers were covered of char. This effect was reduced when the waste was pyrolyzed after a curing process (Fig. 9).

3.7 Material from Aeronautic Waste (Ref. E)

This material was sent to an industrial recycling plant in its original roll format. It was unrolled, pyrolyzed and once a clean recycled CF fabric was obtained, it was rerolled. The fabric obtained was clean with any residue of char (Fig. 10).

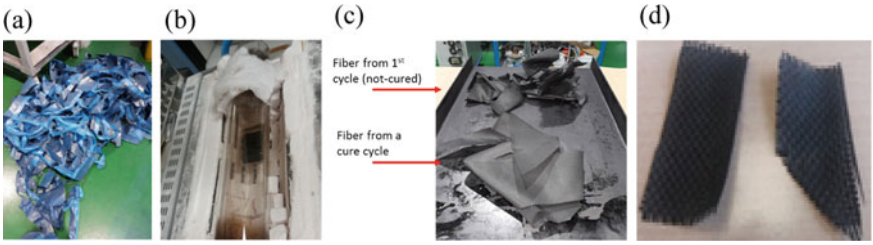


Fig. 9 **a** Waste Ref. D, **b** Ref. D in Glass tube oven, **c** Ref. D after the pyrolysis in Nabertherm oven, **d** Obtained product

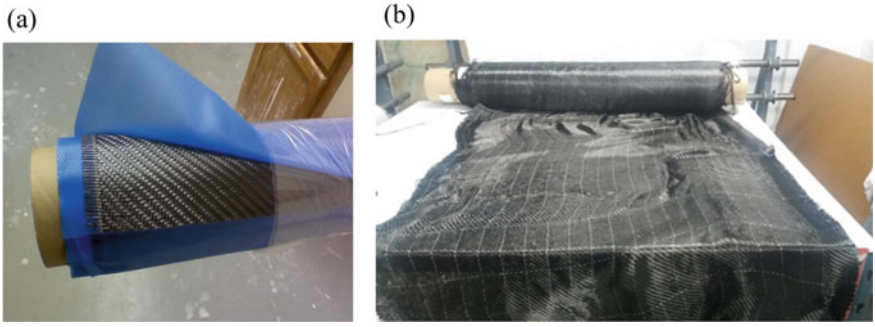


Fig. 10 **a** Waste Ref. E, **b** Obtained product

3.8 Material from Aeronautic Waste (Ref. F1)

This material was introduced into the oven after a shredding process. An oven with nitrogen atmosphere (Nabertherm) was used for the pyrolysis treatment. The results obtained after the defined cycle were short CF with length from 1 to 15 mm. The recovered CF was char covered. To recover the rCF with clean surface, the residual char was removed by mechanical processes (decompressing and sieving). The final products were those obtained in the different fractions of the sieves (Fig. 11) (Table 3).

Finally, the up scaling was carried out in the KOREC pilot plant. The C3 and F2 wastes were used for the final project demonstrators manufacturing, so large amounts of waste had to be treated. In this case, the composite waste was first shredded since the recovered fiber will be used in extrusion-compounding processes and in lamination processes from mat that will also be manufactured with short fiber.

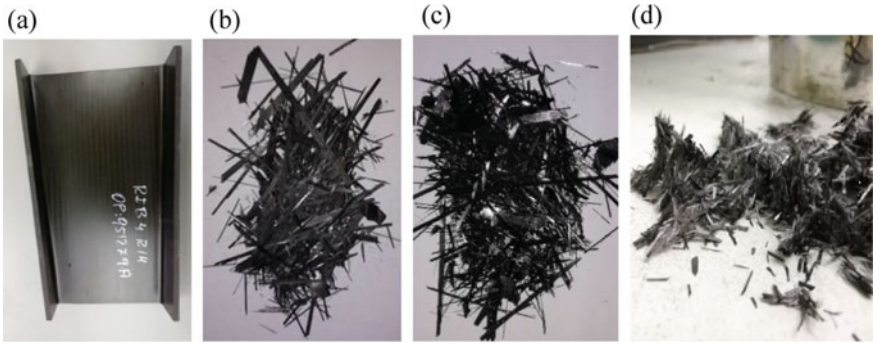


Fig. 11 **a** Waste Ref. F1, **b** Ref. F1 after shredding, **c** Obtained product, **d** rCF after sieving

Table 3 Mass of each fraction obtained

Fraction	Mass (kg)
L > 1 mm	3.048
0.5 < L < 1 mm	1.005
D < 0.5 mm	0.669
Total	4.053

3.9 Material from Wind Sector Waste (Ref. C3)

This material was similar to Ref. C2. The treatment involved the following steps: (i) feeding the composite materials waste into the reaction chamber; (ii) removing oxygen from the chamber until air is substantially eliminated from the reactor; (iii) creating a CO_2-containing environment in the chamber with a predetermined CO_2 volume concentration; (iv) heating the waste in the chamber and reaching a temperature set between 350 and 550 °C; (v) maintaining the CO_2-containing environment and controlling the temperature of said reactor so as to maintain said reaction temperature in the chamber, for a predetermined residence time, obtaining a gas mixture containing products of depolymerization of the resin, and a solid residue comprising the glass fibers or carbon fibers; (vi) extracting the gas mixture from the reaction chamber and cooling it down to a predetermined temperature so as to condensate the products of reaction from the gas mixture, in order to obtain a condensate liquid phase comprising a main amount of the product of depolymerization that it is separated from the uncondensed gas; (vii) extracting the solid residue from the chamber (Fig. 12).

The rGF from C3 was treated to be matrix compatible, this is, re-sized (see Chap. 7) and after this, it was transformed in plastic pellet for injection process and in mat for laminating process. Finally, the rGF was the reinforcement of two demonstrators (Chap. 16): Cowl top support and Light transmitting single skin profiled GFRP sheet.

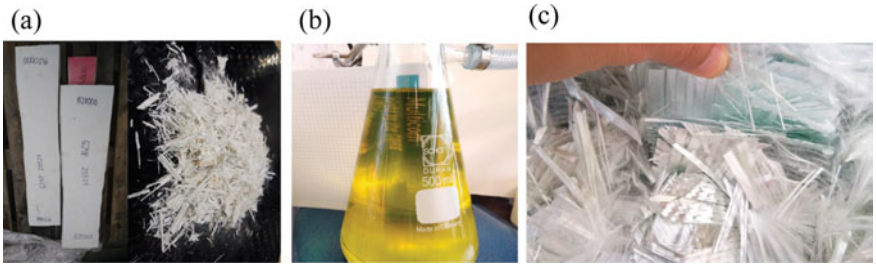


Fig. 12 **a** Waste Ref. C3, **b** Organic liquid recovered from the process, **c** Obtained short rGF

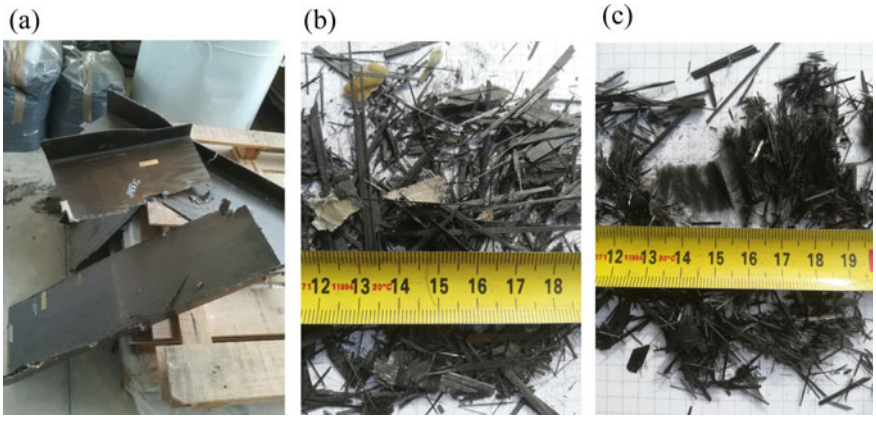


Fig. 13 **a** Waste Ref. F2, **b** Grinded waste, **c** Obtained short rCF

3.10 Material from Aeronautic Waste (Ref. F2)

This material was similar to F1 and the process was also similar to the carried out for C3 (Fig. 13).

The rGF from F2 was also re-sized (Chap. 7) and after this, it was transformed in plastic pellet for injection process for manufacturing the Pedal bracket and the Front-end carrier demonstrators (Chap. 16).

4 FiberEUse Pyrolysis Products Characterization

The tensile strength of single fiber filaments was defined with the Fibrobotics device [9]. At least 50 fibers were tested with a gauge-length of 23.5 mm and crosshead velocity of 0.008 mm/s.

Table 4 Tensile test results

Fiber	Tensile strength (MPa)	Modulus (GPa)
Virgin CF (aeronautic sector)	1328 ± 494*	102 ± 16
rCF (Ref. F1)	534 ± 287	105 ± 25
rCF (Ref. F2)	1869 ± 747	137 ± 36
Virgin GF (construction sector)	1111 ± 485	52 ± 6
rGF (Ref. C2)	437 ± 166	51 ± 16
rGF (Ref. C3)	422 ± 204	53 ± 12

*The reference and quality of virgin fiber could be not the same of the recycled, this data is un-know for the fiber from waste

Tensile tests were carried out to characterize the different materials obtained in the pyrolysis processes. Tests were also carried out on GF and virgin CF to carry out the comparison. The results obtained are presented in Table 4.

Afterwards, the recycled fibers obtained through thermal processes have been validated through the indirect mechanical characterization of the products in which they have been reused (Chap. 7).

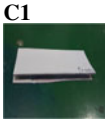
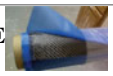
5 Conclusions

As a final technical conclusion of the thermal treatment study, Table 5 summarize the results obtained for each type of composite waste evaluated in FiberEUse project. This table gives a recommendation of the processes in which the recycled fiber from the different sector or applications could be integrated. This recommendation is based on the format, cleanness or properties results obtained for each recycled fiber, combined with the typical requirements of plastics and composites processes.

The up-scaled process in FiberEUse project has some sustainability advantages. The Korec technology is cost effective thanks to the recovery of the organic part of the waste, that can be re-introduced into the composite's production chain. Thanks to this technology the environmental impact is strongly mitigated:

(a) there is an elimination of the volume of FRP waste in landfills and incinerators;
(b) no hazardous chemicals are used during the process;
(c) incondensable gases and residues are used for internal energy recovery;
(d) atmospheric emissions are easily reduced by EU Best Available Techniques (the available techniques which are the best for European Commission for preventing or minimising emissions and impacts on the environment, see reference documents BREFs) and fully respect the limits imposed by European law;

Table 5 Resume of the thermal treatment, the result and de recommended re-use of the FiberEUse waste parts

Ref	Thermal treatment	Result	Recommended Re-use
A1 & A2 	T^a 500 °C Time 12,5 h Air circulation	• Fiber clean • Mat format • 50% properties reduction	• Liquid molding composite processes as infusion or lamination
B 	T^a 500 °C Time 12.5 h Air circulation	• Fiber + high filler content • Cut long fiber format	• Filler in a casting or liquid process
C1 	T^a 500 °C Time 12.5 h Air circulation	• Fiber clean • Mixed formats • Mainly UD long fiber • 50% properties reduction	• Filler in a casting or liquid process
C2 &C3 	Korec technology with CO_2 atmosp. after shredding process	• Short GF (15 mm)	• Compounding with thermoplastic
D 	T^a 450 °C Time 3 h Argon	• Fiber Char covered • Small pieces of fabric	• Liquid molding composite processes as infusion or lamination
E 	T^a 450 °C Time 6 h Inert gas circulation	• Fiber clean • Fabric format	• Liquid molding composite processes as RTM or T-RTM
F1 	T^a 450 °C Time 2 h Nitrogen after shredding process	• Fiber clean after sieving post-treatment • Short CF (15 mm) • 50% properties reduction	• Compounding with thermoplastic • rCF mat manufacturing
F2 	Korec technology with CO_2 atmosp. after shredding process	• Short CF (15 mm)	• Compounding with thermoplastic

(e) there is reduction of the process carbon footprint of the fiberglass and composites supply chain (virgin resins are products of fossil origin).

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