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Environmental Feasibility of the Recycling of Carbon Fibers from CFRPs by Solvolysis Using Supercritical Water

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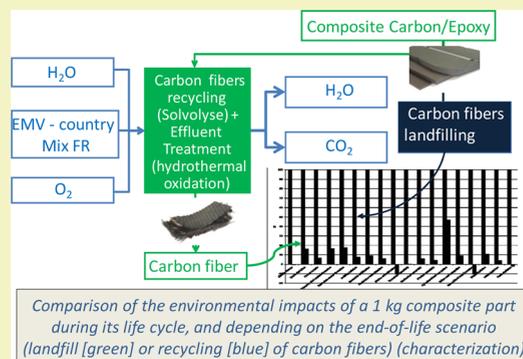
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ABSTRACT: Originally developed for high-tech applications in the aeronautic and aerospace industry, carbon/epoxy composites have been increasingly used in the automotive, leisure, and sports industries for several years. Nevertheless, the carbon reinforcement is an expensive constituent, and it has been recently shown that it is also the most environmentally impacting in a composite part manufacturing. Recycling these materials (even restricted to the reinforcement recovery) could lead to economic and environmental benefits, while satisfying legislative end-of-life requirements. The solvolysis of the matrix by water under supercritical conditions is an efficient solution to recover the carbon fiber reinforcement with mechanical properties closed to the ones of virgin fibers. This paper aims at demonstrating the environmental feasibility of the recycling of carbon fiber/thermoset matrix composites by solvolysis of the matrix in supercritical water. This demonstration is based on life cycle assessment that evaluates benefits and environmental challenges of this recycling loop.

KEYWORDS: Life cycle assessment (LCA), Supercritical water, Solvolysis, Recycling, Composites, CFRP



INTRODUCTION

Carbon fiber-reinforced plastics (CFRPs), or thermoset matrix composites, were originally developed for high-tech applications in the aeronautic and aerospace industry. For several years now, these materials have also been increasingly used in the automotive, leisure, and sports industries. In many applications in these sectors, one may seek aesthetic criteria or a simple feeling of high technology, more than highly technical properties. Thus, constituents' characteristics, and specifically reinforcements, are considered as a secondary matter and may be overemphasized regarding the function of the product. This is particularly true for nonstructural decorative parts (e.g., with a carbon look finish), for which the reinforcement is the most expensive constituent, and where glass fibers, much more less expensive, cannot be used.^{1,2}

Today, there is no, or a limited, deposit (or very few) of carbon fibers from airplanes at the end of life because airplanes integrating such materials are only currently being built and will become waste later. In the future, the expected amount will grow year after year. Therefore, the question is this: Could carbon fibers recycled from airplanes (or from production waste from aircraft and automotive production) substitute mechanically for the majority of carbon fibers currently used in the automotive, leisure, and sports industries, considering that the recycling can be done in a cost-effective way and that the aeronautic industry will not use recycled fibers? Subsequent questions are these: How can carbon fiber-reinforced plastics be

recycled? Is the recycling environmentally more sustainable than the production of virgin carbon fibers?

One of the first uses of the supercritical fluid technology in the field of recycling was applied to polymers. This technique has been developed extensively in Japan since 1995 and has been reviewed many times.³⁻⁵ Beyond plastics recycling, solvolysis in near- and supercritical fluids of thermosetting resins (phenol and epoxy resins) has attracted a great interest among the scientific community to recover materials like carbon fibers with a high added value in the past few years. To date, few studies have been carried out on the chemical recycling of these waste composites with near- and supercritical solvolysis technology.⁶⁻¹⁴ Compared to other recycling processes (mechanical recycling processes, pyrolysis, fluidized bed processes, low temperature solvolysis processes), near- and supercritical solvolysis has the huge advantage that clean carbon fibers are recovered with similar mechanical properties to pristine fibers.⁶ Moreover, these undamaged fibers are obtained at relatively low temperature, without using organic solvents or concentrated acids.

Near- and supercritical water and alcohols were mainly processed as solvolysis media. In fact, near- and supercritical water or alcohols play the role of solvent and reagent for the

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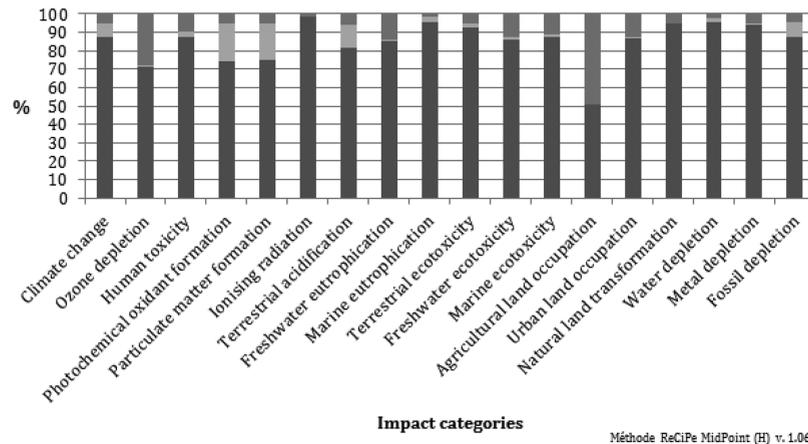


Figure 1. Environmental impacts due to the carbon reinforcement (dark gray), epoxy matrix (light gray), and injection molding process (intermediate gray), while processing a 1 kg carbon/epoxy composite part. The analysis is based on Duflou et al. data.¹⁸

74 depolymerization of condensation polymers by solvolysis into
 75 their monomers in a fast and selective way; it will be a
 76 hydrolysis reaction with water and an alcoholysis reaction with
 77 alcohols. Condensation polymers are constituted with ether,
 78 ester, or acid amide linkages, which can be broken by hydrolysis
 79 or alcoholysis. The example of polyethylene terephthalate
 80 (PET) bottle recycling is significant in term of quantity but also
 81 of development of supercritical fluid-based recycling technol-
 82 ogies. PET can be hydrolyzed in terephthalic acid (TPA), its
 83 monomers, and ethylene glycol in sub- and supercritical
 84 water.¹⁵ Composite plastics such as glass and carbon fiber-
 85 reinforced plastics can be decomposed into monomers and
 86 fiber materials. Some years ago, the successful hydrolysis of an
 87 isolated epoxy resin in sub- and supercritical water has been
 88 already carried out.¹⁶ The solvolysis of composite materials
 89 using near- and supercritical fluids, especially water and alcohol,
 90 was recently reviewed by our group.^{6,17} This way is very
 91 efficient in a technological point of view, but what is about
 92 sustainability?

93 In this paper, the results from an initial life cycle assessment
 94 of the supercritical fluid technology applied to CFRPs recycling
 95 is proposed for the first time in order to position recycled
 96 carbon fibers in its market between virgin carbon fibers and
 97 glass fibers with regard to sustainability and cost considerations.

98 ■ MATERIALS AND METHODS

99 **Environmental Assessment.** *Data for CFRPs Composites*
 100 *Manufacture.* Duflou et al.¹⁸ have some life cycle assessment
 101 (LCA)-based information on the environmental impacts due to
 102 petrochemical manufacturing of composite parts for vehicles as an
 103 alternative to steel, for lightening the vehicle, and for reducing life
 104 cycle air emissions beyond the benefits of plug-in vehicles.¹⁹ In a
 105 conventional car, the use phase has the greatest environmental impact
 106 due to high fuel consumption (directly related to the mass of the
 107 vehicle). In its lighter alternative version, it is the manufacturing phase
 108 that could become predominant.¹⁹ This is due to the carbon fiber
 109 manufacturing (see our analysis in Figure 1) based on data from
 110 Duflou et al.¹⁸ and recalculated relative to the mass of the chosen
 111 product, i.e., 1 kg of carbon fiber. Furthermore, the main source of
 112 impact for these carbon fibers is due to the use of fossil fuel that has an
 113 important carbon foot print.²¹ Hence, it might be of real interest from
 114 a sustainability point view to propose recycled fibers as a way forward
 115 to limit the environmental impacts of the composite parts of light cars.
 116 Due to the fact that the carbon reinforcement is the most impacting
 117 constituent in a carbon/epoxy composite's elaboration process (Figure
 118 1),¹⁸ recycling end-of-life composites (even restricted to the

reinforcement recovery) could lead to reduce some anthropogenic
 impacts by decreasing the use of first-generation raw materials (mainly
 petroleum) for their production. Besides, it would help design
 engineers to balance energy efficiency and cost, by opening new
 opportunities for developing second-generation composites first
 dedicated to the manufacture of medium or low loaded parts. Lastly,
 recycled carbon fabric could widen the range of reinforcements on the
 marketplace between first-generation carbon and glass fibers.

All this has to be done in line with European directives that already
 force industries to improve their products' recyclability (e.g., in
 automotive industry²²). However, making feasible this new recycling
 sector requires overcoming users' reluctances by ensuring the second-
 generation semi-product's validity from economic and environmental
 aspects. Therefore, we carried out a life cycle assessment (LCA) in
 which the resource efficiency and potential environmental challenges
 of the carbon/epoxy composites' recycling process are analyzed.

Life Cycle Assessment: Goal and Scope. Every stage of the life
 cycle of the composite part has to be modeled in the LCA, from its
 manufacture to its end-of-life treatment, following the usual steps
 defined by the ISO 14040 standards.²³ These ISO standards define
 LCA as the following: "Compilation and evaluation of the inputs,
 outputs and the potential environmental impacts of a product system
 throughout its life cycle". LCA is the only method that assesses the
 environmental impacts of a product or activity over its entire life cycle.
 It is a holistic approach that takes into account the extraction and
 treatment of raw materials, product manufacturing, transport and
 distribution, and product use and end-of-life. LCA is structured in the
 following phases: (a) goal and scope definition, (b) life cycle
 inventory, (c) life cycle impact assessment, and (d) interpretation.
 Life cycle impact assessment assigns life cycle inventory results to
 impact categories like climate change and ionizing radiation; the
 environmental profile consisting of the indicator results for the impact
 categories selected provides information on the environmental issues
 associated with the inputs and outputs of the product system under
 study.

As previously mentioned, we focus on carbon/epoxy composites.
 The resin is an epoxy one. The carbon fibers were furnished by
 industry partners; therefore, we do not have any information about
 their precise nature. The deposit of materials to be recycled consists
 possibly in end-of-life aeronautic parts but, most likely to date, in
 composite offcuts. The composite part chosen for the LCA is assumed
 to be processed in Europe with Japanese carbon reinforcement. Its
 mass is supposed to be 1 kg. Thus, we aim at studying the interest of
 recycling such materials more generally, such as the environmental
 feasibility of the recycling process.

Life Cycle Inventory. The following analysis is based on Duflou's
 data,¹⁸ which assessed the manufacturing of composite semi-structural
 panels in automotive industry. All of these data have been recalculated
 relative to the mass of the chosen product (i.e., 1 kg).

168 In our case study, the use phase is not taken into account. Indeed, to
169 the best of our knowledge, the only input data that can be taken into
170 account concern transport operations. Like so, as rather classically, the
171 present simulation shows that this factor did not contribute much to
172 the overall impacts (less than 5%).

173 Regarding the product's end-of-life, two scenarios have been
174 modeled: The first one consists of burying the composite part,
175 which is what is currently done, and represents the reality for actual
176 composites at their end-of-life. The second one consists of the
177 recovery of the carbon reinforcement. We focus on the recycling
178 process by solvolysis described in Figure 2). We consider (i) an

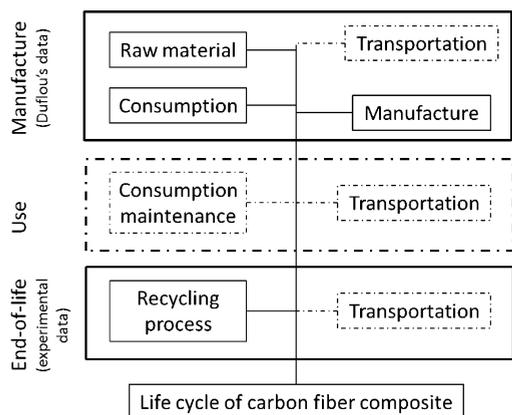


Figure 2. System boundary and life cycle stages. Dashed lines represent life cycle stages that were cut off.

179 aqueous solvolysis of the matrix by water under supercritical
180 conditions (temperature around 400 °C and pressure about 25
181 MPa) and (ii) a hydrothermal oxidation of the effluent to clear matrix
182 components from water at the end of the solvolysis process.

183 This technology allows the fiber to be recovered. Therefore, it is a
184 real (but partial) recycling and not a simple material valorization.⁶
185 Lastly, the process uses energy, water, and oxygen, and only emits
186 water and carbon dioxide.

187 Lastly, the research team from the Mechanics Institute of Bordeaux
188 has developed a prototype for packaging these second-generation
189 fibers in an attractive form for users (i.e., designers). Data matching
190 the remanufacturing stage have not been taken into account yet in this
191 very first LCA. However, this energy input is assumed to be very weak
192 compared to those involved in the first-generation reinforcement
193 process. As a consequence, the life cycle only loops after the
194 manufacturing of the first-generation carbon reinforcement, with no
195 specific additional remanufacturing.

196 *Life Cycle Assessment: Software, Database, and Method.* The
197 LCA is carried out with the SimaPro software (v.7),²⁴ Eco Invent
198 database (v.2),²⁵ and ReCiPe Midpoint (H) method.²⁶ As previously
199 mentioned, in the recycling stage, the avoided material is the
200 reinforcement. In other words, the production of a new raw material
201 with nonrenewable resources (i.e., first-generation carbon reinforce-
202 ment) is avoided.

203 ■ RESULTS

204 **Recycled Carbon Fibers Obtained by Hydrolysis in**
205 **Supercritical Water.** The hydrolysis of the epoxy resin matrix
206 in supercritical water ($p_c = 22.1$ MPa, $T_c = 374$ °C) has been
207 published many times as well as the alcoholysis in supercritical
208 alcohols (methanol, $p_c = 8.1$ MPa, $T_c = 239.3$ °C; ethanol, $p_c =$
209 6.1 MPa, $T_c = 240.8$ °C, or still isopropanol, $p_c = 4.8$ MPa, $T_c =$
210 235.1 °C). For instance, Okajima et al. have studied the
211 hydrolysis of epoxy resin of CFRPs in sub- and supercritical
212 water in the temperature range between 300 and 450 °C and 25
213 MPa. Water in the reactor was found to inhibit the coking and

enhance the decomposition of the resin compared with the case
of pyrolysis. As a result, clean carbon fiber was recovered, and
the resin was decomposed and removed from the carbon fiber.⁹
It can be pointed out that this solvolysis process is able to treat
all types of composites, no matter their surface quality,
geometry, size, density, etc. The only constraint is the reactor
geometry.

Morin et al. have also performed the recycling of carbon
fibers from carbon fiber-reinforced composites in a semi-
continuous flow reactor. Experiments were carried out at a
temperature around the critical temperature of water for a
reaction time of about 30 min. The process has been optimized
in order to improve the solvolysis rate of the resin without the
degradation of the mechanical properties of the fibers. Water or
alcohols can be used as the solvolysis medium. They are
different in terms of energy consumption because the critical
coordinates of alcohols are generally lower than those of water.
Therefore, recycling of CFRPs using an alcoholysis process
could require less energy, but the hydrolysis process is safer and
greener. In this study, water was used as solvent for the
recycling of carbon fibers from CFRPs. The epoxy resin was
completely decomposed into lower molecular weight organic
compounds. Recovered carbon fibers were characterized using
thermogravimetric analysis (TGA) to determine the amount of
resin removed by the process, scanning electron microscopy to
observe the fibers, and single fiber tensile tests to evaluate the
mechanical properties of the recycled fibers. Recycled carbon
fibers from CFRPs are clean (Figure 3). All the resin was

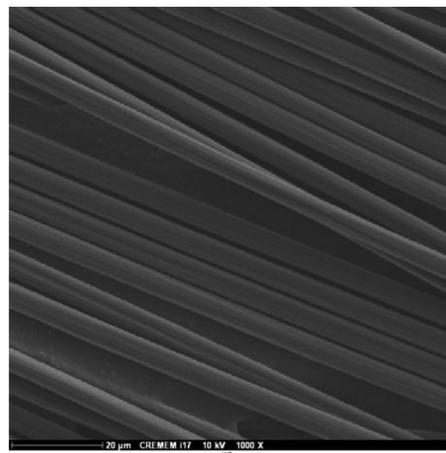


Figure 3. SEM image of recycled fibers after supercritical water treatment at 400 °C and 25 MPa at an ICMCB laboratory.

removed according to the TGA results. Furthermore, the
recycled carbon fibers present good mechanical properties; a
tensile loss close to the one of virgin fibers is obtained.⁶ The
final liquid phase is also analyzed by gas chromatography. The
monomers of the initial resins have been identified.

Environmental Evaluation. The LCA of a 1 kg composite
part that takes into account the recycling of the reinforcement
clearly shows the interest of this end-of-life option. Actually, it
almost offsets the whole environmental impacts of the
composite manufacturing (Figure 4).

By recycling a product mainly sourced with carbon fossil fuel
(Figure 1), impacts on climate change or fossil depletion can be
almost completely avoided (Figure 4). For marine eutrophica-
tion, recycling allowed for a larger avoidance than the impacts
of manufacturing. This is due to the use of European electricity

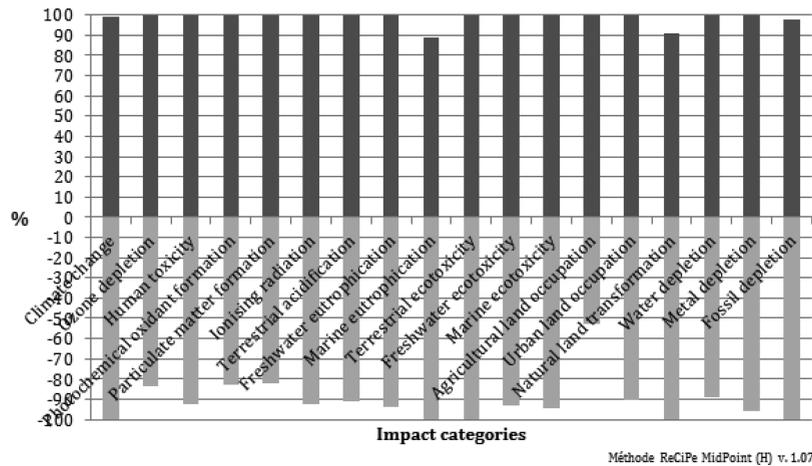


Figure 4. Life-cycle impact assessment of the landfill of a 1 kg carbon/epoxy composite part (dark gray) compared with the reinforcement's recycling (light gray). The analysis is based on the ReCiPe Midpoint (H) method.

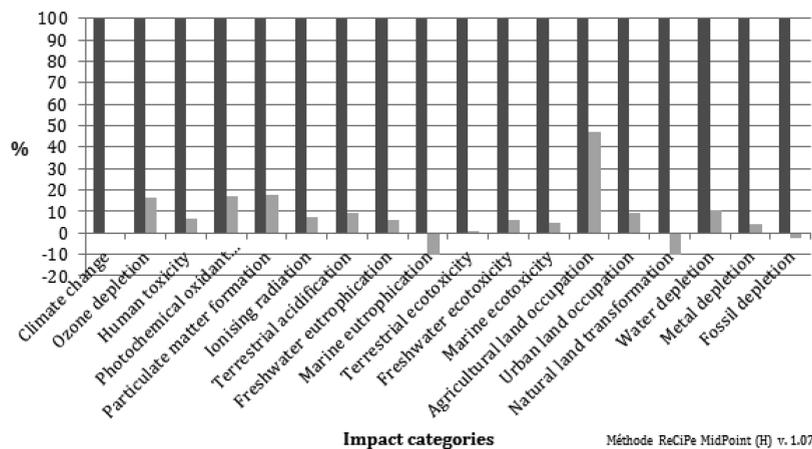


Figure 5. Comparison of the environmental impacts of a 1 kg composite part during its life cycle, depending on the end-of-life scenario of carbon fibers (landfill in dark gray; recycling in light gray).

257 for the injection molding of the matrix, while we use a French
 258 mix for recycling process (it impacts systematically onto this
 259 indicator).

260 When comparing the environmental impacts of a 1 kg
 261 composite part during its life cycle, depending on the end-of-
 262 life scenario (landfill or recycling of carbon fibers), and despite
 263 electricity consumption in the recycling process, emission of
 264 greenhouse gases may be divided by 10 (Figure 5). The
 265 environmental gain is on average about 80%, according to the
 266 ReCiPe Midpoint (H) method. For the climate change
 267 indicator, it is about 100%. This is because of the use of a
 268 French electricity country mix, which is mainly sourced by
 269 nuclear energy, which is energy that has no impacts on climate
 270 change (it does impact principally on the ionizing radiation
 271 category).

272 Negative impacts (for eutrophication and natural land
 273 transformation indicators) do not mean that they are “good”
 274 for the environment. This only means that it is an avoided
 275 impact; to recycle, allows for avoiding some impacts due to the
 276 manufacture stage.

277 **Economic Validation.** We recently made a market study
 278 showing that there will always be relevant uses for recycled
 279 reinforcements or for semi-products based on second-
 280 generation fiber, whatever their mechanical characteristics are
 281 and as long as the price remains reasonable.²⁶ The integration

of recycled carbon fiber is only interesting if the mechanical 282
 performance/price ratio is higher than that of glass fiber. 283
 Therefore, in light of excellent second-generation reinforce- 284
 ment mechanical properties,²⁷ this ratio should be much higher 285
 than for new carbon fibers. Thus, the feasibility of recycling will 286
 be provided if the second-generation semi-products price does 287
 not exceed 70–80% of the new ones. 288

■ DISCUSSION 289

In the present context, the use of carbon/epoxy composite is 290
 ever increasing. As indicated, these composites can be recycled 291
 by solvolysis,⁶ keeping good mechanical properties.²⁷ Anticipat- 292
 ing that they may soon be subjected to regulation, it is essential 293
 to show it is feasible that a composite recycling network can be 294
 set up that is both economically and environmentally favorable. 295

The recovery of the carbon reinforcement (which is the most 296
 environmentally impacting constituent in the composite 297
 manufacturing) by an aqueous solvolysis of the composite’s 298
 matrix leads to an average gain of about 80% for all eco- 299
 indicators compared to the landfill end-of-life option. 300

Lastly, the remanufacturing process developed allows for 301
 obtaining a semi-product easily usable. Consequently, from an 302
 economic point of view, the mechanical performance/price 303
 ratio of the second-generation carbon fiber should be higher 304
 than that for the virgin carbon fibers or the glass reinforcement. 305

306 The next step in the maturation of this technology is the
307 development of a pilot scale facility for the recycling of carbon
308 fibers from CFRPs using the supercritical fluid technology.

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313 Notes

314 The authors declare no competing financial interest.

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