

A technical feasibility study of an innovative spinning process for recycled carbon fibres

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Abstract: This paper presents a technical feasibility study of a properly modified long-staple spinning process developed at laboratory level for carbon fibres derived from manufacturing scraps or waste and blended with polyester. The study aims to validate the suitability of the new process to produce qualitatively acceptable textile substrates, as well as to detect the effect of the percentage of fibre mixing on the achieved quality of semi-finished products. Visual and laboratory tests indicate that the provided long-staple spinning process allows the production of textile substrates, but only when the carbon fibres are blended with polyester at a defined concentration. Finally, the study suggests that future tests at industrial level are needed to prove the technical feasibility of the proposed spinning process for large-scale production.

Keywords: Carbon fibres, recycling, spinning process, technical feasibility study.

1. Introduction

Carbon fibres (CFs) are one of the most commonly used fibres to reinforce thermoset and thermoplastic composite materials. Used for the first time in the ‘60s for military and aeronautical applications, they are currently adopted in many industries (Lefevre *et al.*, 2017). Due to their outstanding physical, chemical and mechanical properties (Giorgini *et al.*, 2015; Yao *et al.*, 2018) the demand of these fibres has continued to grow at impressive rates over the last years (Holmes, 2014). Indeed, since 2010 the request of CFs at worldwide level has more than doubled, reaching 70,500 tonnes in 2017. This trend is expected to continue in the coming years, with an estimated global demand of 120,500 tonnes in 2022 (Sauer *et al.*, 2018). The production of large quantities CFs has resulted in the generation of huge amounts of waste, both from manufacturing processes and products’ end-of-life. This condition has inevitably encouraged research centres and industrial organisations to develop new technologies and methods for recycling CFs in order to achieve economic advantages, as well as to avoid the environmental problems due to their incineration and disposal in landfill.

Scientific literature has revealed that recycled carbon fibres (rCFs) can be divided into three main categories: i) dry fibres extracted from manufacturing scraps and/or waste, ii) fibres coming from fabric prepreg residuals or waste, and iii) fibres retrieved from end-of-life carbon fibre reinforced plastics (CFRPs) (Hasan *et al.*, 2018; Hengstermann *et al.*, 2019). Regardless of their origin, rCFs do not possess the technical characteristics suitable for a traditional spinning process (Oliveux, Dandy and Leeke, 2015). Consequently, they cannot be used for

producing structural applications (Pimenta and Pinho, 2011), but can only be adopted for the generation of second-quality materials, including short fibres random mats, i.e. non-woven fabrics (Hengstermann *et al.*, 2017), and injection moulded composites (Hasan *et al.*, 2018). Researching and developing innovative spinning processes for rCFs emerges thus as essential to obtain high-quality yarns with repeatable physical, chemical and mechanical properties (Hengstermann *et al.*, 2019).

On these premises, this paper presents a technical feasibility study of an innovative process derived from a traditional long-staple spinning process for CFs extracted from manufacturing scraps and/or waste. The technical feasibility study of the new spinning process, that involves the modification of some operating parameters and the adjustment of some components of the carding machine, is carried out by following a two-step research protocol. The first step consists in a characterization of pre-selected rCFs to reveal their distinctive features. Subsequently, the rCFs are mixed with polyester in different percentage combinations and then spun adopting the new long-staple spinning process. The characteristics of the resulted semi-finished products are then assessed through visual and laboratory tests to identify the best fibre mixing and validate the technical feasibility of the proposed innovative spinning process.

In the following section, an in-depth literature review on CFs recycling and a description about traditional spinning process are presented. Section 3 describes the new long-staple spinning process, while Section 4 introduces the materials and control methods adopted to conduct the technical feasibility study. Section 5 presents and discusses the main results of the research. Finally,

the conclusions, limitations and future developments are reported in Section 6.

2. Theoretical background

In accordance with the purpose of this study, the analysis of the theoretical background was developed in two main parts. The first part introduces an analysis of the main factors affecting CF recycling, while the second presents a description of spinning processes.

2.1 CF recycling

The recycling process of CFs can be described through three main factors: i) the nature of CF to be recycled (input); ii) the type of the technology used for fibre recovery and iii) the main applications for rCF (Figure 1). A brief description of each factor is presented in the following.

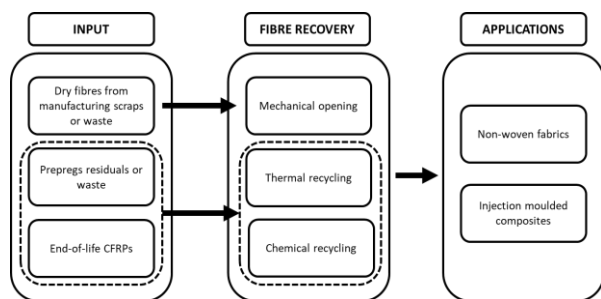


Figure 1: The three main factors characterising CF recycling

Input

Different sources of CF waste are available in industry. They can be categorised in three main groups (Hasan *et al.*, 2018; Khurshid *et al.*, 2020):

- 1) Manufacturing scraps and/or waste (dry fibres)
- 2) Prepreg residuals or waste (impregnated fibres)
- 3) End-of-life CFRPs (impregnated fibres).

Some distinctive characteristics and different treatment methods for fibre recovering, distinguish each group of CF waste. For instance, manufacturing scraps or waste, including offcuts, bobbin ends and selvage, are resin-free and still sized materials, that possess properties similar to virgin carbon fibres (Hasan *et al.*, 2018). Therefore, no specific actions are necessary to recycle them (Hengstermann *et al.*, 2019). At most, a mechanical opening operation may be required to open up their orthogonal weaves. Prepreg residuals or waste and end-of-life CFRPs, instead, are impregnated with resin. Therefore, further processing is needed to separate the CFs from the other elements.

Fibre recovery

Thermal or chemical recycling technologies are generally used to extract CFs from the resin (Pickering, 2006).

Thermal recycling technologies, including pyrolysis and fluidized bed, enable fibre recovering through the

decomposition of the polymer matrix. While the former consists of a polymer matrix degradation by heating the material in the absence of oxygen at 450°C-600°C, fluidized bed technology involves the decomposition of polymer matrix by a stream of high temperature air coming from a bed composed of fluidised silica sand. Both processes entail the production of char, and require oxidation post-treatments to obtain clean fibres and fillers (Oliveux, Dandy and Leeke, 2015). The CFs reclaimed by thermal recycling technologies are discontinuous, short and fluffy. However, they can also have modulus and tensile properties comparable to virgin fibres (Giorgini *et al.*, 2015; Naqvi *et al.*, 2018). Nevertheless, pyrolysis is the most used recycling technology in commercial applications, as it is considered the most reliable and efficient process in terms of energy and fibre recovery (Witik *et al.*, 2013).

Chemical recycling technologies involve the adoption of chemical reagents for degrading the polymer matrix. Specifically, the depolymerisation process, also called solvolysis, allows to recover not only clean fibres and fillers, but also the monomers of the resin (Yang *et al.*, 2012). The CFs recovered through chemical recycling technologies are characterized by high mechanical properties and fibre length (Pimenta and Pinho, 2011). However, they show a reduction in the bonding capacity with the new polymer matrix (Jiang *et al.*, 2009).

Applications

Currently, rCFs can be used for the production of non-woven fabrics or injection moulded composites (Hengstermann *et al.*, 2016). In general, obtained non-woven fabrics and injection moulded composites are both characterised by low mechanical properties due to randomly oriented fibres, low fibre content and high fibre damage during production (Pickering *et al.*, 2016; Wölling *et al.*, 2017). For instance, the strength of composites materials obtained from non-woven fabrics ranges from 200 to 300 MPa (Schinner, Brandt and Richter, 1996), reaching around 404 MPa in case of injection moulded composites (Gulich and Hofmann, 2013). For this reasons, they are adopted for producing non-structural applications such as aircraft and vehicle interiors (Pimenta and Pinho, 2011; Hasan *et al.*, 2018).

As highlighted by several scientific studies (Akonda, Lawrence and Weager, 2012; Hengstermann *et al.*, 2016; Hasan *et al.*, 2018), the spinning of rCFs could represent a way to widen the application of rCFs to more structural components, as yarns and their semi-finished products are both characterised by high fibre orientation and good compactness (Hasan *et al.*, 2018). Nevertheless, spinning of rCFs is not as well established as in the case of spinning of traditional textile fibres, e.g. cotton, hemp and wool. Indeed, CFs are characterized by brittleness, natural crimp absence and sensitivity to shear stresses (Hengstermann *et al.*, 2016), making traditional spinning process very difficult and ineffective. In such a context, the need to readjust the traditional spinning process emerges as necessary to create products suitable for structural applications.

2.2 Spinning process

As shown in Figure 2, the production of traditional yarn generally involves a multi-step approach, that can be summarized into four main phases: preparing, carding, drawing and spinning (Lawrence, 2003). The outputs of each phase are, respectively: proper fibre mixture, carded web and/or carded sliver, thinner sliver, and roving and/or yarn.

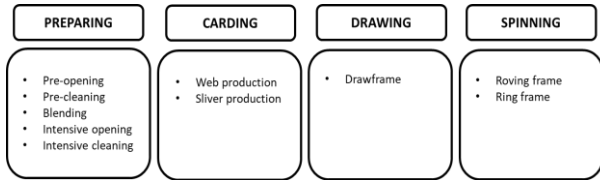


Figure 2: Main phases of a traditional spinning process

Different spinning processes exist. Among others, ring spinning is the most widely used within textile companies (Xu *et al.*, 2011). It can be divided into short- and long-staple spinning. Specifically, the former is used for cotton processing, while the latter for wool processing.

Carding phase plays a fundamental role within all spinning processes (Lawrence, 2003). Through the parallelization of the fibres, this step makes it possible to create a sliver with good mechanical characteristics. However, while in short-staple spinning, carding is carried out by means of cylinders covered by two or more stationary carding plates, in long-staple spinning process, it is performed through a set of rotating elements as shown in Figure 3.

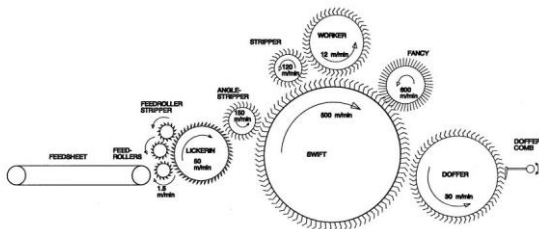


Figure 3: A traditional long-staple carding machine (Wood, 2009)

3. The innovative long-staple spinning process

This section introduces a description of the proposed innovative long-staple spinning process. As shown in Figure 4, the new process involves the same steps of a traditional long-staple spinning process with a double carding and drawing. It also entails some updating of the existing spinning machines, as well as new settings of their operating parameters, as detailed in the following.

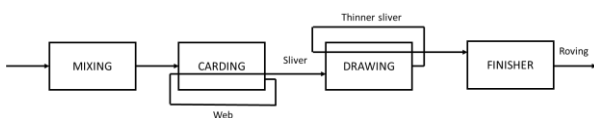


Figure 4: Spinning process adopted in the study

3.1 Updating of spinning machines

Before starting the innovative spinning process, a re-adaptation of long-staple spinning machines was carried out to deal with the distinctive characteristics of the CF, namely brittleness, natural crimp absence, and sensitivity to shear stress. Because of carding plays a fundamental role within the whole spinning process, special attention was paid to this process phase. In particular, a laboratory long-staple carding machine was used to ensure a very delicate carding process on rCFs. Nevertheless, the card clothing of cylinders was changed and set several times to achieve the optimum performance. In the end, in order to have a very gentle carding process and less damage on brittle rCF, a low dens card clothing was applied on the carding machine. Moreover, as the doffer’s speed regulation plays a significant role in the collection of the web, an inverter was installed on the carding machine to prevent breakages.

3.2 Main steps of the innovative spinning process

A fibre mass of 25 g (i.e. rCF and polyester blended at different ratios) was put on the carding feed conveyer. Initially, a prior opening was carried out manually for the polyester fibres and then the rCFs were spread on the surface of the polyester layer, as shown in Figure 5. To obtain a sliver with better quality, a double passage was made on the carding machine. At first passage a web was produced. Then, such web was used as input for the second passage where by means of a coiler a sliver was manufactured. The count of the produced sliver is approximately 4.3ktex.



Figure 5: Mixing phase before carding

Subsequently, in order to produce a thinner sliver with well-distributed and oriented fibres, a double drafting was applied to the carded sliver using a laboratory drafting joint with a finisher (long-staple flyer). Specifically, doubling was carried out with 6 strands of carded sliver count 4.3 ktex passing through the drafting zone that is made up of break draft and main draft, whose values are respectively calculated as the ratio between the rotation speed of feeding and intermediate (secondary) rollers and the ratio between the rotation speed of intermediate (secondary) and delivery rollers. The delivered drafted sliver was passed again through the

drafting zone and then through the false twister to produce a roving. The linear density of the roving manufactured is approximately 1.34 ktex. The values of break draft and main draft applied in draw frame and finisher are reported in Table 1.

Table 1: Break draft and main draft setting applied in the draw frame and finisher

Drafting zone	Break draft	Main draft	Winding draft	Total draft
Draw frame	1.2	3.3	0.0	4.0
Finisher	1.2	3.3	1.2	4.8

4. Materials and control methods

This section reports a detail about the materials used in the study and the control methods adopted to assess the technical feasibility of the new spinning process.

4.1 Materials

For this investigation, rCFs derived from dry fibres from manufacturing scraps and/or waste were processed. This choice was made as such source of waste represents around 40% of all CFRP generated waste (Pickering, 2006). Furthermore, it possesses similar properties to virgin CF (Hasan *et al.*, 2018), so it is expected to be easier to process. Specifically, the samples were taken from an initial set of 17 alternatives supplied by an Italian company specialized in the production of fabrics in CF. Only those samples with the lowest variation in length were pre-selected. In detail, the sample with the minimum length variation was selected for the analysis (Figure 6).



Figure 6: Fibrogram of the length distribution of rCF. y-axis: Fibre length (mm).

The smooth surface and zero crimps of CF generally result in low fibre cohesion that does not allow to produce a card web with proper mechanical characteristics. This requires to blend rCFs with a crimped staple fibre. In this study, staple polyester fibre, selected because of its good fibre-to-fibre cohesion and entanglement (Akonda *et al.*, 2014), was adopted as carrier for rCF in different mixing ratio from 10 to 60 weight % respectively.

The staple length of polyester was on average 40mm. This value was chosen to match the length of rCF. Moreover, the use of white polyester staple fibre together with black CF made it possible to visually assess some features of the semi-finished products (e.g. presence of rCFs, orientation, and length of the CF).

4.2 Control methods

Three different types of control were carried along with the new long-staple spinning process to validate it and assess the best fibre mixing to be processed. As shown in Figure 7, these included a characterisation of CFs, two visual checks and a final quantitative control.

The adopted control process was ‘pass-fail’. In other words, if a blend did not pass a check, it was directly discarded without subsequent controls.

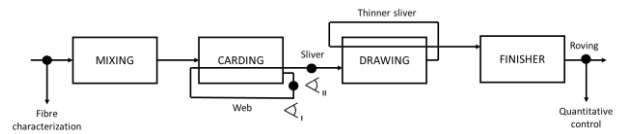


Figure 7: Spinning process control points

4.2.1 Characterization of CFs

Different physical and mechanical properties of the rCFs were investigated during CF characterization. They are briefly described in the following.

Length distribution

Since commercially available rCF usually contains fibres with variable length, the length distribution of rCF was determined by means of a fibre mass diagram manually performed. The evaluation of the distribution of rCF fibres’ length was performed considering 1-gram fibre mass taken randomly. This analysis was repeated 5 times in order to identify the average length of rCFs employed in this study.

Diameter distribution

A Scanning Electron Microscope Zeiss LEO 1530 SEM instrument was used for a morphological evaluation of the surface of the rCFs. The statistical distribution of the diameter was evaluated based on a statistical sample of 30 measurements.

Tensile properties of individual rCF

In order to quantify the mechanical behaviour of the fibres, some single-filament tensile tests (SFTT) were performed according to ASTM 3379, by using a Zwisch Roell 1K dynamometer, equipped with a loading cell of 5N. Samples were realized bonding a single filament on a cardboard using 20mm gauge length, which was then clamped between tools of the dynamometer. Tensile tests

consisted in 16 repetitions and were performed at a constant crosshead rate of 1 mm/min on sample lengths of 20mm and using a pre-load of 0,002N. Both tensile modulus and tensile strength are computed considering a nominal value of fibre’s diameter previously found.

Determination of sizing contamination of rCF

The sizing amount present on CFs was calculated according to JIS R 7601 Testing Methods of Carbon Fibres. The sizing component was decomposed in a high temperature atmosphere. The difference in mass before and after the decomposition was then measured in order to calculate sizing percentage.

A sized fibres specimen of approximately 2g was weighed to the fourth decimal (W_0). Then, specimen was put for 15 minutes in an electric furnace previously heated to 450°C and purged out with nitrogen gas for a minute. After a fast cooling, the specimen was again weighed to the fourth decimal place (W_1) in grams.

Sizing Amount was calculated by:

$$\frac{W_0 - W_1 * f}{W_0} * 100 (\%)$$

Where, f is an experimental corrective factor equal to 1,0001, previously determined by same procedure applied on CFs with a known sizing amount.

4.2.2 Visual and quantitative controls

Two visual controls were made. The first control was conducted on the web after the first passage on the carding machine, while the second was carried out on the carded sliver as it exited the coiler. In each control the surface of the semi-finished product was checked to ascertain the absence of any imperfections or holes. Subsequently, due to the difference in colour of the two fibres used, both the orientation of the CFs and the homogeneity of the individual outputs were assessed.

Finally, a quantitative control of both the carded sliver and the roving obtained after passing through the finisher was carried out. It consisted of calculating the amount of CFs remaining in the semi-finished products through a chemical investigation. Specifically, in accordance with ASTM D 629, a 90% phenol solution at 80 °C was applied to a 1g specimen of sliver and roving weighted after oven drying in order to dissolve polyester fibres. Remained fibres were removed and after drying in the oven for 1.5 hour at 105°C were placed in the desiccators to cool at room temperature. The rCF mass remaining were weighed and the rCF percentage composition in the final sliver and roving was derived.

5.Results and discussion

This section reports and discusses the main results achieved from the analyses made on rCF and semi-finished products.

5.1 Characterisation of rCF

Microscopic measurements allowed to evaluate the average rCF diameter, the tensile properties of single rCF, and their sizing contamination.

The average diameter is $5.26 \pm 0.26 \mu\text{m}$ as shown in Figure 8 that reports the statistical distribution of the diameter evaluated with a statistical sample of 30 measurements. The load-extension curves derived from the single fibre tensile test are shown in Figure 9. Finally, it has been possible to highlight the relevance of sizing on the surface microstructure of CFs which provides a very smooth surface, as evidenced by the micrographs reported in Figure 10. Specifically, the sizing amount presents around the rCFs used is 1.54%.

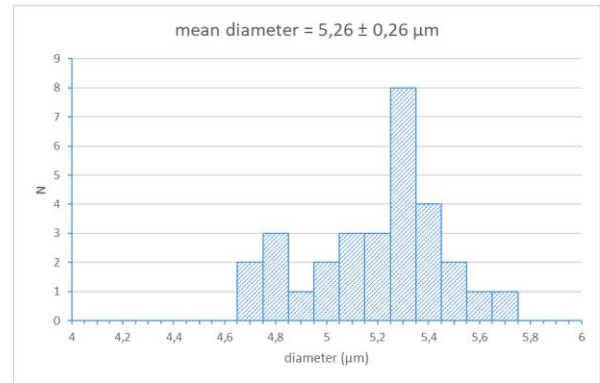


Figure 8: Statistical distribution of the diameter of rCFs evaluated with a sample of 30 fibres

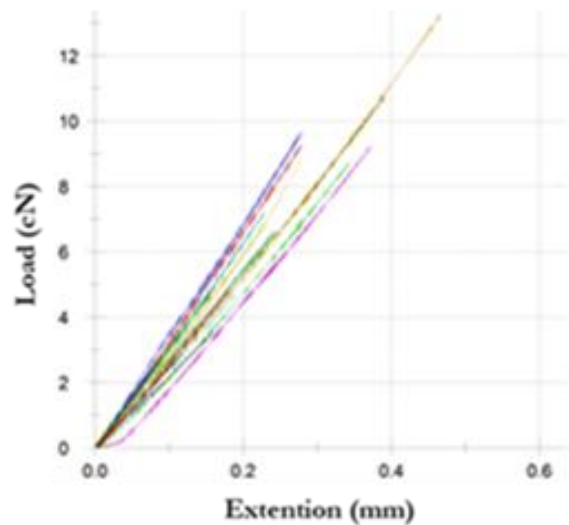


Figure 9: Load-extension curves of rCFs

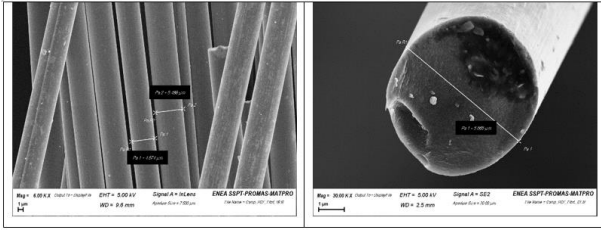


Figure 10: Surface microstructure of rCFs

According to Table 2, it can be stated that the type of rCF investigated in this study possessed the requirements to be spun and used for the production of textile substrates.

Table 2: Physical properties and tensile properties of single rCFs (average values based on 16 repetitions)

Properties	rCFs
Fibre diameter (μm)	5.26 ± 0.26
Sizing amount	1.54%
Tensile strength (MPa)	3327
Tensile modulus (GPa)	144
Elongation at break (mm)	0.25

5.2 Visual and quantitative controls

By means of the visual and final quantitative controls, it was possible to identify the most suitable mix of CFs and polyester for the generation of qualitatively acceptable semi-finished products. According to Table 3, it seems that with high percentages of rCF it is difficult to produce semi-finished products of acceptable quality. Indeed, the 90%-10%, 80%-20% and 70%-30% blends did not even pass the first visual inspection. In these cases, it was very hard to produce a satisfactory web as the most CFs were stuck to the card clothing of the carding machine. The 60%-40% and 50%-50% blends, instead, passed the first visual control, but following the second carding, still showed high inhomogeneity, making the sliver unsuitable for drawing.

Anyway, it could be argued that the more the quantity of polyester, the more the quality of the semi-finished products. However, a high presence of CF ensures higher mechanical properties. Therefore, it is necessary to find a compromise between the percentage of fibre mix and the quality of the semi-finished products. The test suggested that the percentage of fibre mixing which provides the semi-finished products with the best properties is 40% of rCF and 60% of polyester. Its outputs, indeed, overcame all the control points, while quantitative control shows 35% of rCF presence in the hybrid carded sliver and 32% for the hybrid roving,

which are good values considering the 40% of rCF in input.

Table 3: Mixing ratios and related outputs control points

Mixing ratio	Visual control I	Visual control II	Quantitative control (on 40%)
90%-10%	KO	n.a.	n.a
80%-20%	KO	n.a.	n.a
70%-30%	KO	n.a.	n.a
60%-40%	OK	KO	n.a
50%-50%	OK	KO	n.a
40%-60%	OK	OK	Sliver: 35% Roving: 32%

6. Conclusion, limitations and future research

This paper reports a technical feasibility study of an innovative long-staple spinning process for rCFs based on dry fibres from manufacturing scraps or waste and blended with polyester. The achieved results indicate that the investigated rCFs are suitable for spinning, but require to be blended with another fibre in such a concentration as to allow the CFs to form a sliver and a roving with good properties. Furthermore, the study suggests that in order to obtain suitable semi-finished products, carding operations must be changed by repeating this phase twice and using specific working parameters and appropriately revised equipment.

This study was carried out in the laboratory, working with special machines and equipment. Therefore, in the near future it would be interesting to test the new long-staple spinning process at industrial level in order to prove its feasibility for large scale production. Moreover, an economic and environmental analysis could be implemented to provide a complete feasibility study of the proposed spinning process. Finally, as the research was conducted by using a specific type of rCFs, it would be interesting to replicate the study adopting rCFs with longer staple length or derived from prepreg residuals or waste and end-of-life CFRPs to understand whether the process is able to work also CFs of different origin.

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