Analysis of hybrid yarns properties for good-quality recycled carbon fibre-reinforced plastic composites

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Abstract: This paper aims at assessing the performance of recycled carbon fibres and hybrid yarns in view of their application in carbon fibre-reinforced plastic composite materials for structural components. In detail, the hybrid yarns were manufactured by means of an innovative spinning process the authors have developed in a previous study. Recycled carbon fibres from manufacturing scraps or waste blended with a thermoplastic fibre (i.e. polyester or polyamide) to a weight of 50-50% were used as input. Quantitative laboratory tests, including scanning electron microscopy, tensile test, thermogravimetric analysis and differential scanning calorimetry, show that hybrid yarns are characterized by good mechanical properties. In particular, hybrid yarns composed of recycled carbon fibres blended with polyester are less ductile and, at the same time, more rigid than those where recycled carbon fibres are blended with polyamide. Furthermore, the percentage of recycled carbon fibres present in the final result is about 7-14% lower than the value originally entered. Finally, the paper paves the way for future research. First, it is fundamental to evaluate the mechanical properties of hybrid yarns composed of higher percentages of recycled carbon fibres. Second, the assessment of the mechanical properties of the carbon fibre-reinforced plastics that are being produced soon as well as the electrical conductivity of the hybrid yarns for both textile and composite applications, could be of interest.

Keywords: Carbon fibre, yarn, laboratory tests, mechanical properties, recycling.

I. INTRODUCTION AND BACKGROUND

Sustainability has been gaining momentum over the last years and, nowadays, is increasingly accepted and widespread among business strategies [1]. Indeed, there is significant pressure not only from customers [2], whether final users or other companies, but also from the political discussion [3]. In such a context, various legislation has been introduced to limit landfilling and incineration of waste in many industrial sectors, including automotive and electronics. Therefore, it is reasonable to foresee that in the coming years, analogous procedures will be launched for other industries. An example could be aeronautics where carbon fibre-reinforced plastics (CFRPs) are more and more significantly deployed [4], due to their outstanding properties, namely high strength and stiffness, low density, fatigue and corrosion resistance, etc. [5, 6].

The growing demand and usage of such kind of material [7] results in the generation of huge amounts of waste, both from manufacturing processes (i.e., bobbin ends, selvedge, and offcuts) and products' end-of-life, which must be managed to avoid significant environmental issues and substantial economic losses for companies [8]. Indeed, such a waste is currently mostly incinerated or disposed of in landfills [9, 10]. Contrariwise, when recycled, carbon fibres (CFs) may be used for the production of short fibres random mats (i.e., non-woven fabrics) and injection moulded composite materials [11, 12]. However, randomly oriented fibres, low fibre content and high fibre damage during production do not allow non-woven fabrics and injection moulded composites to have appreciable mechanical properties [13-15]. Therefore, they may not be adopted for the manufacturing of structural utilizations, such as load bearing structures, but only for second quality products suitable for specific niche applications, such as aircraft and vehicle interiors [12].

As highlighted by several scientific studies [12, 16, 17], spinning of recycled carbon fibres (rCFs) could represent a way to expand the application of rCFs to more structural components. Indeed, the properties of composite materials could be significantly improved by using a reinforcement consisting of yarns that exhibit high fibre orientation and good compactness [12, 18]. However, the spinning of rCFs is not as well established as in the case of spinning of traditional textile fibres. Indeed, CFs are brittle, without natural crimp and sensitive to shear stresses [17], making traditional spinning process ineffective and complex. Therefore, the research and the development of innovative spinning processes for rCFs is crucial to obtain good-quality yarns with repeatable chemical-physical and mechanical properties [18].

Despite of the potential advantages, this issue has not been widely addressed in the scientific literature, to date. Only few studies focused on such a topic. A modified spinning process to produce hybrid yarns composed of CF and polyamide 6 suitable for the production of unidirectional CFRPs was developed at laboratory level using virgin CFs cut in order to simulate the rCFs from manufacturing scraps [12, 17]. Furthermore, a recent study by the authors showed that it is possible to effectively spin rCFs from manufacturing waste and scraps blended with polyester fibres [19]. Nonetheless, further laboratory tests are necessary to assess their suitability for the production of recycled CFRP composites [17].

On these premises, this paper aims to perform an initial evaluation of the performance of rCFs used and hybrid yarns manufactured through the process described by [19], using 50-50% weight blending ratio and diverse numbers of draw frame doubling in view of their applications in CFRPs suitable for structural components. To this end, a series of specific laboratory tests on the physical, chemical and mechanical properties of the yarns were carried out.

The reminder of this study is organized as follows: Section 2 introduces the materials and methods adopted to conduct the laboratory tests, while Section 3 presents and discusses the main results of the research. Finally, the conclusions, limitations and future developments are reported in Section 4.

II. MATERIALS AND METHODS

This section reports about the materials adopted in the study and the laboratory tests performed to assess the physical, chemical and mechanical properties of the rCFs, as well as the yarns manufactured through the innovative spinning process the authors have already assessed from a technical point of view.

A. Materials

The rCFs used for this study were supplied by an Italian company specialized in the production of CF fabrics and are recovered as scraps from manufacturing. The choice fell on such type of waste as it represents the main source of CFRP waste, nowadays [9,10]. Furthermore, it possesses similar properties to virgin CF [12], so it is likely easier to process. The average length of rCFs is about 60 mm. Since they have smooth surface and zero crimps, they are generally characterized by low fibre cohesion, which does not allow to produce a card web with proper mechanical characteristics. Therefore, rCFs require to be blended with a crimped staple fibre. In this work, staple polyester fibre and staple polyamide fibre, selected because of their good fibre-to-fibre cohesion and entanglement [11, 20], were adopted as carriers for rCFs. The staple length of polyester and polyamide was on average 60 mm. This value was selected to match the length of rCFs.

B. Methods

Diverse physical, chemical and mechanical properties of the rCFs, as well as of the hybrid yarns were investigated through laboratory tests. They are briefly outlined below.

B.1 Characterization of CFs

Length distribution, diameter and surface characterization

Commercially available rCFs are usually characterized by variable length. Therefore, the length distribution of rCFs was determined using a fibre mass diagram manually performed. The assessment of the distribution of rCF fibres' length was carried out by considering 1-gram fibre mass picked at random. This analysis was repeated five times in order to identify the average length of the rCFs exploited in this research. The statistical distribution of the diameter was assessed on the basis of a statistical sample of 30 measurements using a LEICA DVM6 optical microscope device. Lastly, the morphological characterization of the surface of the fibres was performed by a Scanning Electron Microscope Zeiss LEO 1530 SEM instrument.

Determination of sizing on rCF

The sizing amount present on CFs was computed in accordance with JIS R 7601 Testing Methods of Carbon Fibres. The sizing component was decomposed in a high temperature atmosphere. To calculate sizing percentage, the difference in mass before and after the decomposition was measured, by following Eq. (1).

$$\frac{W_0 - W_1 * f}{W_0} * 100 \tag{1}$$

where f is an experimental corrective factor equal to 1,0001. This value was previously determined using the same procedure applied on CFs with a known amount of sizing.

Specifically, a sized fibres specimen of approximately 2 grams was weighed to the fourth decimal (W_0). Subsequently, such a 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 rapid cooling, the specimen was again weighed to the fourth decimal place (W_1) in grams.

Tensile properties of individual rCF

Single-filament tensile tests (SFTT) were performed according to ASTM 3379, by using a Zwich Roell 1 K dynamometer, equipped with a loading cell of 5N. Samples were prepared bonding a single filament on a cardboard, using 10, 20, and 40 mm gauge length, which was then clamped between the tools of the dynamometer. Tensile tests consisted in 30 repetitions for each gauge length and were performed at a constant crosshead rate of 1 mm/min using a pre-load of 0,002N. Both tensile modulus and tensile strength are calculated deeming the nominal value of fibre's diameter previously found.

B.2 Production of rCF/Polyester and Polyamide spun yarn blends

The rCFs were blended with polyester fibres and polyamide fibres considering 50-50 weight %. To produce the yarns, the laboratory spinning process proposed and tested from a technical point of view in a previous study by the authors was adopted. Fig. 1 shows the different stages composing the process and the related outputs. For more information about the innovative spinning process used, please refer to [19].

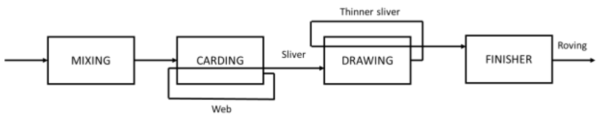

Fig. 1. Spinning process adopted in the study

In detail, a 25 grams fibre mass (i.e. rCF and polyester or polyamide blended) was used. After the production of the card web, different slivers were manufactured thanks to the coiler. Subsequently, doubling was implemented with 3 or 5 strands of carded sliver (i.e., draw frame doubling) passing through the drafting zone and false twister to produce the roving. Fig. 2 shows an example of the sliver and roving obtained. Finally, the hybrid yarns were produced with the support of an Italian industry, leader in the production of textile devices at both laboratory and production level. The general features of the resulting hybrid yarns are reported in Table I.

Fig. 2. Sliver (above) and roving (below) obtained through the innovative spinning process

B.3 Characterization of hybrid yarns

Count

The count of each hybrid yarn was measured by means of the well-known formula for direct *dtex* counting in Eq. (2):

$$10000 * \frac{P\ (g)}{L\ (m)} \tag{2}$$

where *P* is the weight in grams of the skein of yarn of length 0.5 m.

Specifically, yarn skeins, which were subsequently tested for the determination of the related count, were prepared manually. 30 measures were considered for the calculation of the mean values using 0.5 m sample length.

TABLE I
GENERAL FEATURES OF THE HYBRID YARNS ASSESSED

	1	**2**	**3**	**4**
rCF weight [%]	50	50	50	50
Polymer type	PL*	PA*	PL*	PA*
N. of draw frame	3	3	5	5
Twist per metre	300	300	300	300

*PL=Polyester; PA=Polyamide

Tensile properties

Tensile tests of the manufactured yarns were performed in accordance with ISO 3341 by means of an MTS ALLIANCE RT50 2 kN dynamometer. For each hybrid yarn, 10 repetitions were performed at a constant crosshead rate of 200 mm/min using flat clamps. The length of the yarn samples was 500 mm. Both tensile modulus and tensile strength are calculated from the previously computed diameter values for each hybrid yarn using Testworks ®4 software.

Thermogravimetric analysis and Differential scanning calorimetry

A simultaneous thermal analyser was adopted. In detail, the TA instruments SDT Q600 was used to concurrently perform thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). TGA was carried out to capture the actual amount of rCFs present in the hybrid yarns produced. Instead, the melting behaviour of polyester and polyamide was analysed through DSC using a heat cycle with a heating rate of 10°C/min and a temperature range of 25-800°C. The weight of the yarn samples entered into the equipment is around 10 mg. All samples were tested under nitrogen.

III. ANALYSIS OF RESULTS

This section shows and discusses the main outcomes obtained from the analyses of rCFs and hybrid yarns.

A. Characterization of CFs

A SEM micrograph of the CF used in this analysis is illustrated in Fig. 3, highlighting the relevance of sizing which provides a smooth surface, free of carbonaceous deposits. In particular, the amount of sizing present around rCFs used is 1.54%. Furthermore, Fig. 4 outlines the statistical distribution of the diameter assessed with a statistical sample of 30 measurements. In detail, the average diameter is 7.27 ± 0.72 µm. Typical load-extension curves derived from the single fibre tensile test of 30 fibres are shown in Fig. 5.

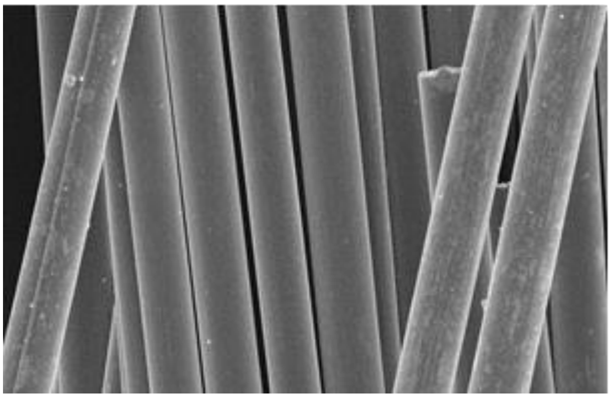

Fig. 3. Surface microstructure of rCFs

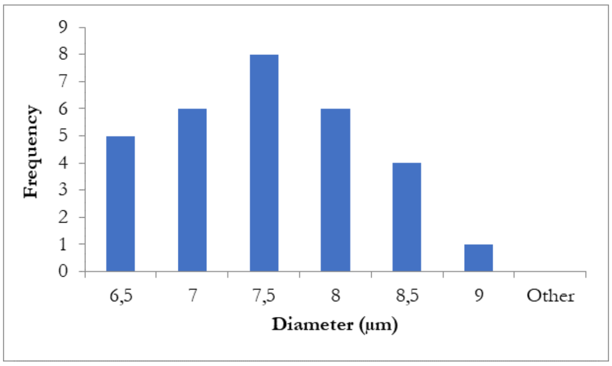

Fig. 4. Statistical distribution of the diameter of rCFs assessed with a sample of 30 measurements

The strength data was interpreted by applying the Weibull statistic, as it is generally used to describe the strength distribution of single fibres with the assumption that defects exist [21, 22]. Both scale (σ_0) and shape (β) parameters were identified by linearizing the cumulative distribution function as already done by [23]:

$$\ln\left(\ln\left(\frac{1}{1-F}\right)\right) = \beta\ln(\sigma) - \beta\ln(\sigma_0) + \ln(AL_f) \quad (3)$$

where σ is the tensile strength of a single fibre, A the fibre cross section, L_f the gauge length of a single fibre during the test and F the probability of failure.

In detail, according to [24], a median-order approach of experimental data about tensile strength was followed to obtain F, as shown in Eq. (4):

$$F = \frac{i - 0.3}{N + 0.4} \quad (4)$$

where i is the i-th stress datum and N the total number of samples.

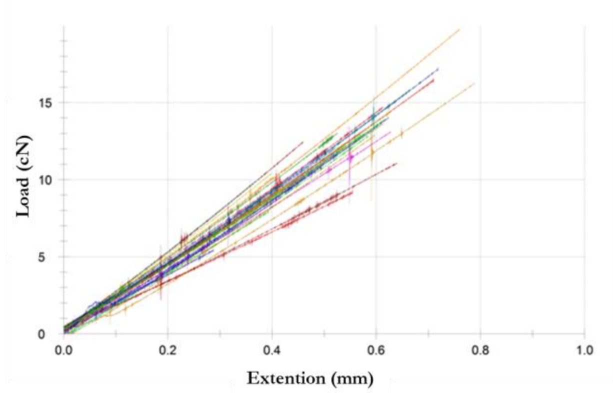

Fig. 5. Load-extension curve for 40 mm gauge length

A typical plot, with its linear-fitting procedure, is shown in Fig. 6. The values of σ_0 and β for rCFs according to the gauge length used are reported in Table II.

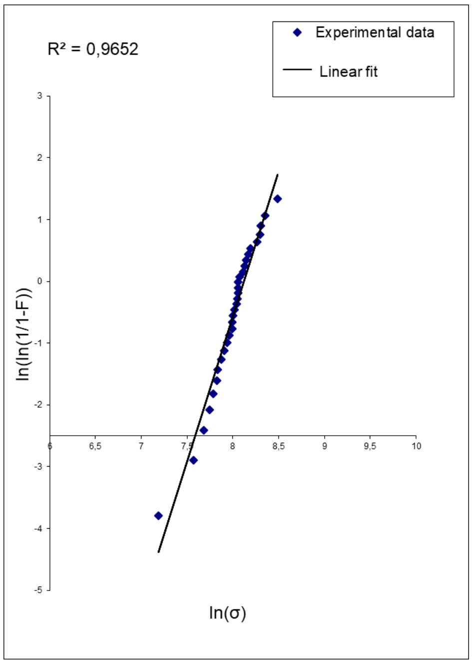

Fig. 6. Weibull linear-fitting plot for 40 mm rCFs

Lastly, as it is recognized that the tensile strength rides on the fibre length, it is possible to determine the tensile strength as the length of the fibre (L) changes while considering a specific breaking probability by inverting Eq. (3).

Therefore, Eq. (3) becomes:

$$\sigma = \sigma_0\left(\frac{L_f}{L}\right)^{1/\beta} \quad (5)$$

Fig. 7 shows the graph drawn from Eq. (5) using parameters reported in Table II and experimental data obtained following Eq. (4) considering a reliability value of 50%. Overall, it is possible to claim that the experimental data is well aligned with the prediction of the Weibull model.

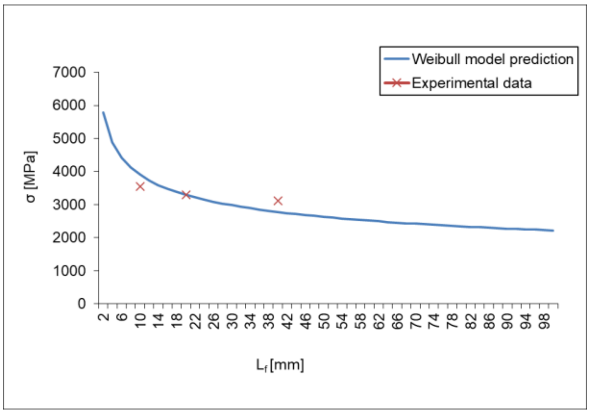

Fig. 7. Tensile strength of the sample used as a function of gauge length (reliability = 50%)

TABLE II
WEIBULL PARAMETERS OF RCFs FOR EACH GAUGE LENGTH

	β	*σ_0 (MPa)*
rCFs (10 mm)	5.41	3802
rCFs (20 mm)	4.08	3601
rCFs (40 mm)	4.70	3368

B. Characterization of hybrid yarns

Table III reports the count of the different hybrid yarns. Overall, it appears that the hybrid yarn blended with polyester and 3 draw frame doubling has a higher count than the hybrid yarn with the same thermoplastic fibre and draw frame doubling equal to 5. In the case of polyamide, however, the opposite seems true. The hybrid yarn with 5 draw frame doubling has a higher count that the hybrid yarn produced using draw frame doubling equal to 3. In general, the high variability (σ) in the results obtained for the physical characterization may be explained by two motivations. On the one hand, the technology readiness level (TRL) is quite low (equal to 3 or 4), since the spinning process is innovative. Therefore, it potentially needs improvements. On the other hand, the final spinning machine was set to maintain a yarn twist of 300 rpm.

TABLE III
VALUE OF HYBRID YARNS COUNT

	1	**2**	**3**	**4**
Mean value [dtex]	577.3	461.7	515.8	734.0
σ	178.7	123.4	153.5	135.2

Furthermore, the results concerning the tensile tests are summarized in Table IV. Looking at the values within the table, it is possible to draw some considerations.

TABLE IV
TENSILE PROPERTIES OF THE MANUFACTURED HYBRID YARNS

#	**Tenacity [cN/tex]**		**Strain at break [%]**		**Young's Modulus [MPa]**	
	Mean	σ	Mean	σ	Mean	σ
1	10.3	4.4	6.5	0.9	917.2	197.4
2	11.7	4.3	13.4	2.0	253.5	53.3
3	13.1	5.4	6.6	0.7	771.1	149.5
4	8.5	3.1	13.1	2.2	278.9	72.7

Overall, the hybrid yarns composed of rCFs blended with polyester have higher tenacity, but lower strain at break than those made of rCFs and polyamide, concurrently. Therefore, it may be stated that hybrid yarns #1 and #3 are less ductile than hybrid yarns #2 and #4. Moreover, hybrid yarns made of rCFs and polyamide are less rigid than those blended with polyester; therefore, they deform more easily. This result emerges from the fact that hybrid yarns composed of polyester are characterised by higher values of Young's modulus. Finally, the number of draw frame doubling appears to have a controversial role. Indeed, for polyester, a number of draw frame doubling equal to 5 shows an increase in tenacity, an equal value of strain at break, and a decrease in the Young's Modulus at the same time, thus highlighting that hybrid yarns # 1 and #3 are more or less characterised by the same ductility, but hybrid yarn #1 is more rigid. In the case of polyamide, instead, a number of draw frame doubling equal to 5 shows a decrease in tenacity, an equal value of strain at break, and a slight increase in the Young's Modulus at the same time. This result highlights that hybrid yarns #2 and #4 are more or less characterised by the same ductility, but hybrid yarn #4 is slightly more rigid. An example of load-extension chart is reported in Fig. 8.

The amount of rCFs present in the hybrid yarns after their manufacture is reported in Table V.

TABLE V
RCFs WEIGHT PERCENTAGE MEASURED BY TGA

	1	**2**	**3**	**4**
Weight [%]	40.5	36.3	43.1	39.5
σ	3.1	2.9	5.4	1.6
Δ%	9.5	13.4	6.9	10.5

Considering that the percentage of rCFs entered for the production of the four hybrid yarns is 50%, it is possible to state that the remaining quantity decreases in all types of yarn by about 7-14%. Such a decrease could be attributed to the loss of CFs during one or more steps of the yarn manufacturing process. Moreover, regardless of the type of thermoplastic fibre used, it appears that hybrid yarns produced using a number of draw frame doubling equal to three have higher percentage reductions. For illustrative purposes, Fig. 9 shows the thermal behaviour of hybrid yarn #1 and hybrid yarn #2. Generally, the upward curves highlight exothermal reactions, while downward curves endothermal ones. Overall, in this study, the DSC graphs are only characterized by downward curves probably due to the CFs presence. It can be stated that for polyester the melting phase occurs at around 255°C. For polyamide, the same reaction takes place at around 260°C. These behaviours are outlined by the first detectable peaks in the related graphs. Instead, peaks between 400 and 500°C represent the thermal decomposition of the thermoplastic fibres. In addition, the decomposition of polyester and polyamide ensures that the TGA curves remain constant for temperature values above 500°C.

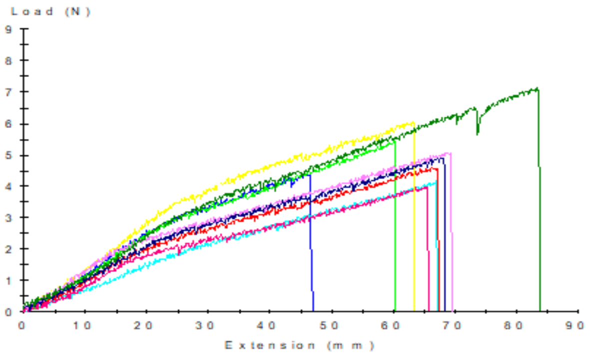

Fig. 8. Load-extension curves for the hybrid yarn #3

IV. CONCLUSION

In this study, a comprehensive analysis of the properties of rCFs from manufacturing scraps or waste has been carried out. Furthermore, the properties of hybrid yarns made up of such fibres and polyester or polyamide fibres in 50-50 weight % and with diverse number of draw frame doubling have been investigated and compared to assess their suitability for good-quality CFRPs production. Results show that the innovative spinning process can be performed regardless of the type of thermoplastic fibre with which the rCFs are blended. Obviously, such a fibre affects the mechanical characteristics of the resulting yarn. Overall, it seems that hybrid yarns composed of polyester are less ductile and more rigid than those blended with polyamide. Moreover, the innovative spinning process is responsible for the decrease in the amount of rCFs present in the hybrid yarn produced.

Future improvements should be related to the production of hybrid yarns using other blending ratios in order to capture potential differences in the goodness of mechanical properties. In addition, upcoming research should be devoted to the manufacturing of unidirectional and woven CFRPs and their characterization in order to assess whether they are suitable for structural applications. Furthermore, considering that CFs are characterized by good electrical conductivity [25], an in-depth analysis of the obtained hybrid yarns on this aspect is strongly recommended.

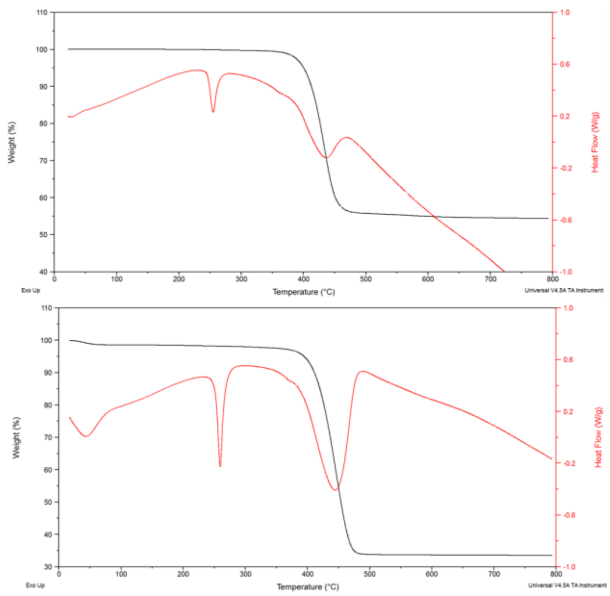

Fig. 9. TGA (black) and DSC (red) curves for hybrid yarn #1 (above) and hybrid yarn #2 (below)

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