

# CHARACTERIZATION OF CARBON FIBRES RECLAIMED FROM STEAM-THERMAL RECYCLING PROCESS

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## Introduction

Presently, most of the carbon fibre reinforced composite wastes from outdated pre-pregs, manufacturing scrap and End-Of-Life (EOL) components are landfilled. The increasing amount of composite wastes generated with the increasing demand of carbon fibres as well as the non recyclability of cross-linked thermoset composite materials cause not only huge wastes of non renewable resources but also environmental issues. Hence, recycling valuable carbon fibres from Carbon Fibre Reinforced Polymer (CFRP) wastes offers both environmental and economic incentives.

Several types of carbon fibre recycling processes have already been proposed and developed to offer a viable solution, such as pyrolysis [1], chemical solvent extraction [2,4] as well as steam-thermal treatment [3,4]. The steam-thermal recycling process, which extracts the organic matrix of the composite materials using superheated steam, provides a new alternative approach to recycle thermoset as well as thermoplastic CFRP wastes.

Our paper aims to give both qualitative and quantitative characterizations on the carbon fibre samples reclaimed via steam-thermal treatment in our laboratory. The microstructure of both recovered carbon fibres and virgin ones was subject to scrutiny of Environmental Scanning Electron Microscopy (ESEM). Elemental analysis has also been conducted to determine the exact composition of carbon fibre samples. In addition, X-ray Photoelectron

Spectroscopy (XPS) was employed to examine the surface chemistry of the carbon fibre samples in terms of the functional groups.

## Experimental

### Materials

Epoxy-sized virgin carbon fibres provided by Toho Tenax Europe GmbH were investigated as reference.

Scrap composite samples were cut off from commercial composites made via Resin Transfer Molding (RTM) and Vacuum Infusion methods, which contain same virgin carbon fibres.

The reclaimed carbon fibres were obtained from the scrap composite samples via steam-thermal treatment.

### Apparatus and Procedures

The steam-thermal treatment was realized in a thermogravimetric analyser (TGA) and also in a bench-scale reactor. Both are equipped with a humidity generator and inerted with nitrogen.

The ESEM was carried out on platinum-coated samples to acquire high resolution images.

The elemental analysis was performed with a CHNS analyser.

The XPS spectroscopy was performed with a Thermo Scientific K-Alpha monochromated (Al K $\alpha$  1486.6 eV) XPS spectrometer. All the samples were washed in an ultrasonic wash system using acetone and hexane solution and then dried in an oven for a week at 80°C before investigation under XPS analysis.

**Results and Discussion**

The ESEM results indicated that the surface of carbon fibres recovered via superheated steam-thermal process were smooth and virtually undamaged. Almost all the resin matrix has been extracted by the treatment (see Fig. 2). The streaks caused by the mechanical spinning process during manufacturing can be clearly identified on both virgin and steam-thermal recovered carbon fibres.

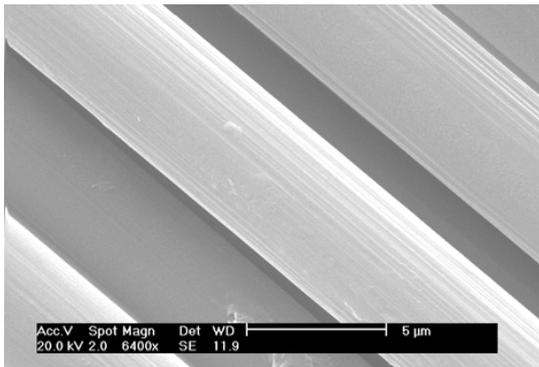


Fig. 1 ESEM image of sized virgin carbon fibre surface

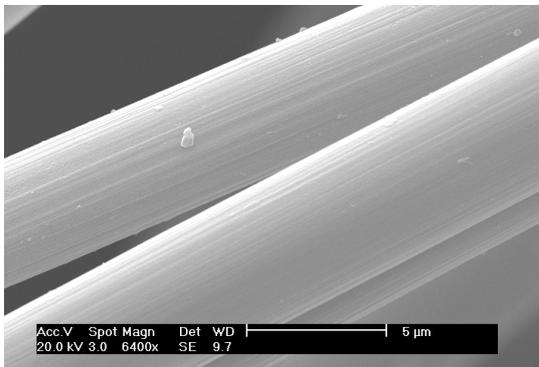


Fig. 2 ESEM image of steam-thermal reclaimed carbon fibre surface

The elemental analysis showed that the reclaimed carbon fibres are close to the sized virgin ones in composition. (see Table 1)

Table 1 Elemental analysis results on the sized virgin and the reclaimed carbon fibres

Sample	N %	C %	H %	S %
sized virgin carbon fibres	2,57	95,69	0	0

steam-thermal treated ones	3,23	93,19	0,09	0,01
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Table 2 shows the XPS survey of virgin carbon fibres and reclaimed ones. Not much difference of atomic ratios between the two was found. However, the reclaimed ones showed a slight decrease of C-O functional groups while an increase of C=O functional groups probably due to steam oxidation.

Table 2 XPS functional group proportion and atomic ratios of the sized virgin carbon fibres and the reclaimed ones

Sample	C-C/H	C-O	C=O	OC=O	O/C	N/C
Sized virgin CF	56,65	19,86	2,08	1,39	0,18	0,027
Reclaimed CF	62,05	12,5	4,51	3,93	0,18	0,028

**Conclusion**

The visual appearance, the composition and the surface chemistry of steam-thermal reclaimed carbon fibres were investigated. The carbon fibres recovered from the steam-thermal process are free of resin and have a composition close to the virgin ones. In fact, the viability and the cost-effectiveness of CFR recycling process mostly depend on the quality of the reclaimed carbon fibres. Further investigations including tensile strength tests of the recovered carbon fibres are in progress.

**References**

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