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## Research article

# Recycling and characterization of carbon fibers from carbon fiber reinforced epoxy matrix composites by a novel super-heated-steam method

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## ABSTRACT

In order to manufacture high quality recycled carbon fibers (R-CFs), carbon fiber-reinforced composite wastes were pyrolysed with super-heated steam at 550 °C in a fixed bed reactor for varying reaction times. The mechanical and surface properties of the R-CFs were characterized with a single fiber tensile test, interface shear strength (IFSS), scanning electron microscopy (SEM), and X-ray photoelectron spectroscopy (XPS). The surface analysis showed that there was no matrix char residue on the fiber surfaces. The tensile strength and IFSS values of the R-CFs were 90% and 115% compared to those of virgin carbon fibers (V-CFs), respectively. The recycling efficiency of the R-CFs from the composites were strongly dependent on the pyrolysis temperature, reaction time, and super-heated steam feeding rate.

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## 1. Introduction

The world-wide demand for carbon material composites is growing rapidly due to the applications for renewable energy systems (Al Hamouz et al., 2017; Orge et al., 2016), aerospace (Kim et al., 2010), and automotive (Raghubanshi et al., 2016). However, the increased use of carbon fiber reinforced plastics (CFRPs) also has generated an increasing amount of CFRP wastes. For instance, the leadoff aircraft with CFRP structural components will soon be decommissioned (Pimenta and Pinho, 2011). Also, the same thing will happen to current commercial aircraft (the service life of 8500 commercial planes will end by 2025), with each aircraft bring forth more than 20 tons of CFRPs wastes (Roberts, 2007). Automotive waste is also a serious apprehension due to the wide application of CFRPs for structural strengthening (Lambert et al., 2015). In addition, the since the first production of carbon fiber in the late 1960s demand for carbon fibers is growing rapidly.

According to statistics, the global market value for CFRPs was about 25 billion USD in 2013 and this value is expected to double in 2022, signifying a growth rate of over 7.6% per year. The increasing requirement of CFRPs presented a high environmental and economic recognition among researchers for the need to recycle CFRP waste materials in a suitable way (Liu et al., 2004).

A variety of methods have been investigated for reusing high value carbon fiber from carbon fiber reinforced composites through recycling. The main recycling points is to decompose the polymeric matrix with each different methods leaving clean carbon fibers (Jiang et al., 2009). So far, several methods to recycle CFRP wastes have been investigated. The typical recycling methods could be classification into three systems: mechanical recycling (Kouparitsas et al., 2002), chemical process (Bai et al., 2010; Dang et al., 2005; Hyde et al., 2006) and thermal processing (Nahil and Williams, 2011; Yip et al., 2002). Other recycling methods like microwave treatments have also been investigated (Lester et al., 2004). Mechanical recycling contains disintegration CFRPs by crushing, milling, shredding, or other mechanical processes (Braun et al., 2001). This method is not suitable for CFRP wastes because the high valuable carbon fibers are destroyed. In the chemical process

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used under the condition of low temperature, solvents are used to decompose epoxy resin into a soluble fraction, and then carbon fibers are recovered. Several solvents, such as tetralin, nitric acid, supercritical/subcritical alcohols, and supercritical/subcritical water (Fromenteil et al., 2000; Liu et al., 2009) have been used as media for the chemical processing of epoxy resin. The tensile strengths of recycled carbon fibers (R-CFs) gleaned from chemical process can be kept in a good condition (approximately over 80% of virgin carbon fibers (V-CFs)). Extreme example, Liu et al. (2009) used 8 M nitric acid to break down epoxy resin/carbon fiber composite at 90 °C for 20–100 h. The loss of tensile strength was about 1.1%. As such, the mechanical properties of the carbon fibers are, for the most part, not damaged; however, increased price of reagent and processes of massive harmful gases are the major drawback for the recycling of carbon fibers from chemical process. Also, it is difficult to scale-up most chemical methods. The last method is a thermal process of the most widely used recycling processes for CFRPs (Blazco, 1997; Cunliffe et al., 2003; Sun et al., 2015). The present commercial-scale carbon fiber recycling operations in the world are all based on the pyrolysis process. In this process, thermal is used to pyrolyze organic molecules. The polymeric matrix is pyrolyze into smaller molecules, and comparatively clean carbon fibers can be recovered (Feraboli et al., 2011; Marsh, 2008). The tensile strength of the R-CFs obtained from this process is about 70–80% of V-CFs (Chen et al., 2013; Schinner et al., 1996).

Unlike conventional pyrolysis recycling studies ( $N_2$ ,  $O_2$ ), this study modified the pyrolysis method by introducing superheated steam into the process to maintain the mechanical properties of R-CF considerably. Super-heated steam is a readily available material (water) and has the characteristic that uniform heating is possible. In addition, using the air in second oxidation step reduces the oxidation of CFs and has the characteristic of being able to decompose the resin intensively. Also, the CFs regenerated by the superheated steam method can naturally reform the surface during the process without any additional surface treatment process, thereby improving the interfacial property of CFRPs. Unlike the conventional recycling process, this study can be recovered to tar without vaporizing the resin. Therefore, the emission of carbon dioxide is considerably less than that of conventional processes.

## 2. Experiment details

### 2.1. Sample preparation

The carbon fibers (CFs) studied in this work were polyacrylonitrile based unidirectional CFs (T700, Toray Co., Japan). The epoxy resin that was used as a matrix was diglycidyl ether of bisphenol-A (DGEBA, YD-128, Kukdo Chem., Korea). The epoxide equivalent weight was 185–190 g/eq, and the viscosity was 11500–13500 cps at 25 °C. Diaminodiphenylmethane (DDM, Tokyo Chem., Japan) was selected as a hardener, and methylethylketone (MEK, Daejung Chem., Korea) was used to reduce the high viscosity of the DGEBA. The carbon fiber reinforced plastics (CFRPs) were prepared with the hot press method at 175 °C. And the CFRPs had a resin content of 36% volume.

A fixed SiC furnace was used to pyrolyze the CFRP composites. Alumina tube, with a length of 1000 mm and an inner diameter of 80 mm, was horizontal mounted in an electrical resistance furnace (15 kW) and heated to final temperatures of 550 °C. A removable porcelain crucible containing the sample was positioned in the centre of the quartz tube. The CFRPs were heated to 550 °C at 10 °C/min in a SiC furnace under  $H_2O$  (liquid flow 2 ml/min) and kept at the target temperature for 30 min to obtain carbonized CFRPs. Then, the gas flow was switched to air ( $O_2$ -21% under  $N_2$ ) at a rate of 200 ml/min while the temperature was raised to 550 °C and held for 30–75 min, respectively. Recycled carbon fibers (R-CFs) series were obtained after cooling down to room temperature.

### 2.2. Characterization

The thermal decomposition temperature of the CFs and epoxy resin was measured in nitrogen at 10 °C/min over a range of temperatures up to 900 °C with a thermogravimetric analyzer (TGA-50, SHIMADZU, Japan).

The functional group on the surface of the R-CFs was confirmed and analyzed using Fourier transform infrared spectrometry (FT-IR, Nicolett id tm10, Thermo Scientific, USA) at 4000–500  $cm^{-1}$  wavelengths. The R-CFs sample was ground with KBr and pressed into a thin disc.

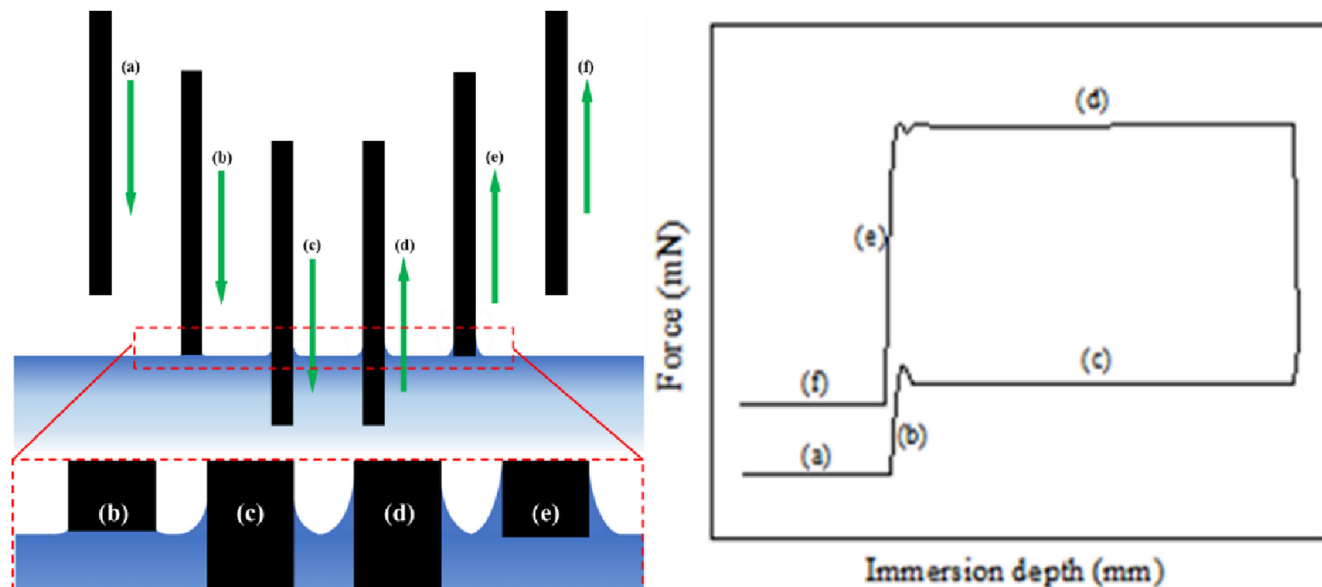


Fig. 1. Schematic diagram of the single fiber contact angle test; (a), (b), and (c) as the fiber enters the wetting liquid, the force is measured for the advancing angle; (d), (e), as the fiber is removed from wetting liquid, the force is measured for the receding angle (Kim et al., 2014).

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