BRIDGE

MURFITTS INDUSTRIES®

Industry Case Study

Murfitts Industries.
Thermal and Microscopic Analysis of Recovered Carbon Black Material

Background

Carbon black is a highly valuable material used in large quantities (>10 million tons) globally, especially in the automotive tyre industry. The high carbon content of this material means that there is an impetus to transition the production and use of carbon black into a circular process via novel recovery processes, primarily pyrolysis of tyres.

Tyre pyrolysis is a process that involves the controlled heating of waste tyres in the absence of oxygen. This process breaks down the rubber and other components of the tyres into a variety of products, including recovered carbon black (rCB). rCB is a valuable material that can be used as a reinforcement agent in the production of new tyres, plastics, and other products.

The carbon black extracted from tyre pyrolysis is often of comparable quality to virgin carbon black, making it an attractive alternative for manufacturers seeking to reduce their environmental impact.

However, the key functional properties of recovered carbon black (rCB) materials can vary dramatically depending on the rCB chemistry, morphology, and structure. Thus, there is a need to develop robust characterisation methods for this novel material to guide their production and quality control during manufacture.

Approach

The Bridge, at the University of Lincoln, UK, is equipped with a range of advanced analytical tools for materials characterisation. These include scanning/transmission electron microscopes (S/TEM), which have the capability to combine imaging with EDX elemental analysis to investigate chemical composition, and simultaneous thermal analysis (STA), which performs thermogravimetric analysis (TGA) which can be coupled with analysis of the evolved gases via a quadrupole mass spectrometer (MS).

Outcomes

Using the TEM the rCB material was shown to have a hierarchical structure. rCB particles were present as discrete small particles which themselves were organised into larger aggregate structures. Figure 1A shows an example TEM image illustrating several of the aggregates, as well as a histogram showing the range of aggregate sizes observed.

Figure 1B shows a high-resolution TEM image which shows the crystal structure of one of the rCB components. The structural information obtained from these types of images allowed Murfitts Industries to better understand the properties of the rCB material and allows a comparison to be made with virgin carbon black materials which can guide the optimisation of the recovery process.

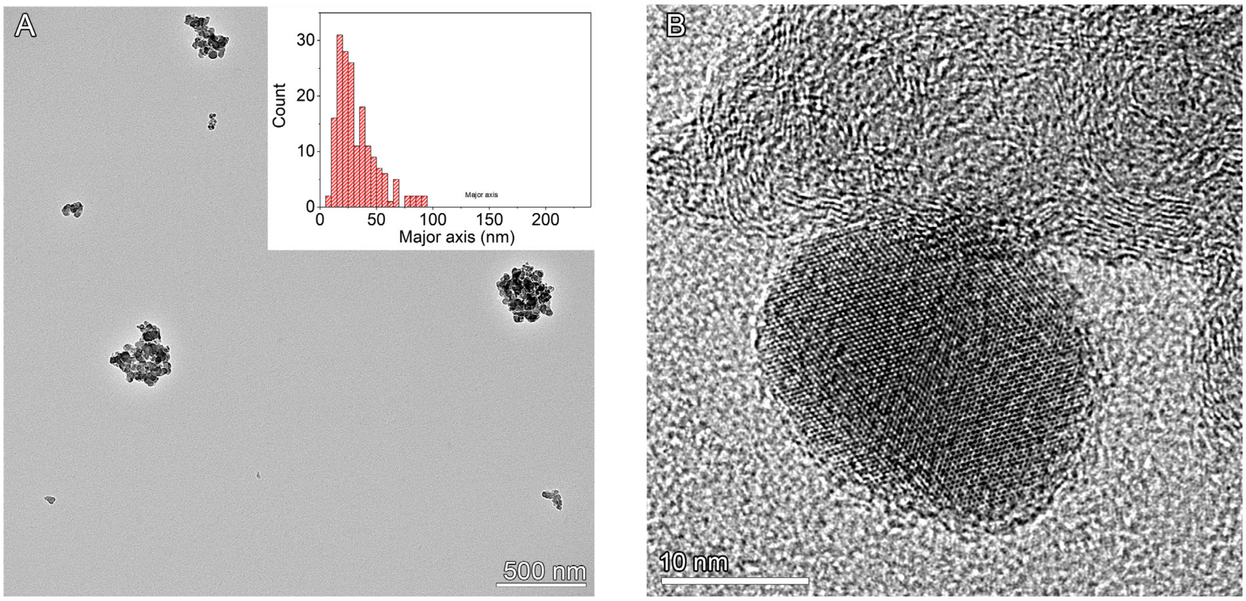

Figure 1. (A) A low magnification TEM image showing several rCB aggregates as used for TEM-based aggregate sizing. The inset shows a histogram of aggregate sizes for one sample. (B) A higher magnification image showing atomic contrast within one aggregate.

Figure 2 shows a comparison between a TEM image and the associated EDX map from which it is possible to identify the chemical components present within an individual aggregate. rCB properties are critically dependent on the nature and concentration of chemical species thus, this information allows the final product properties to be explained and predicted in the future thereby allowing the recovery process to be optimised.

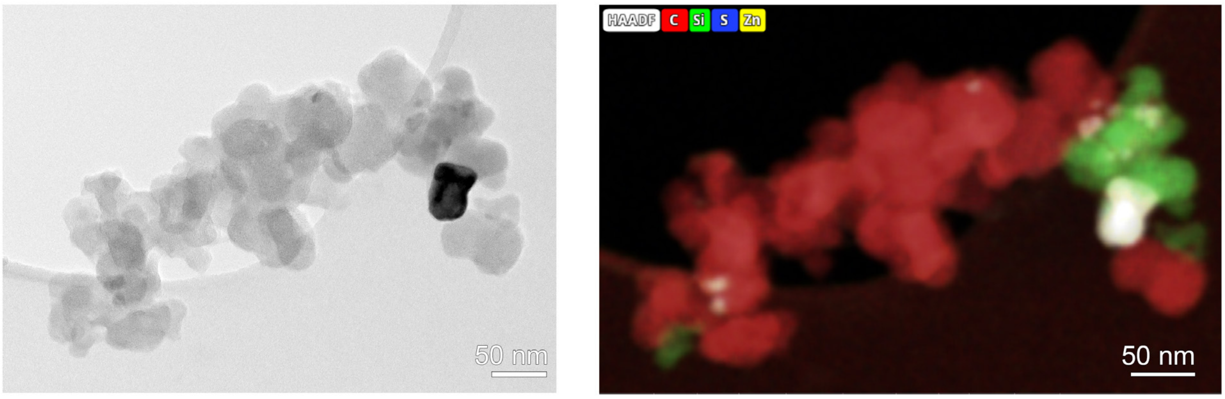

Figure 2. (A) TEM image of an rCB aggregate containing multiple materials. (B) TEM-EDX map identifying individual elements within the aggregate.

Using the STA-MS it is possible to observe not only weight loss as the rCB material is heated, but to chemically identify the gases evolved during heat treatment. This method is extremely sensitive and can be applied to a wide range of temperature and gas conditions.

Figure 3 illustrates the gravimetric response over time as a sample of rCB material was heated from room temperature to 900°C. In addition, Figure 4 shows the identity of evolved gases during the temperature ramp of the STA measurement.

Data such as this allowed Murfitts Industries to better understand the composition of their material, predict functional properties, optimise recovery, and confirm the absence of potentially harmful or environmentally damaging components before the products go to market.

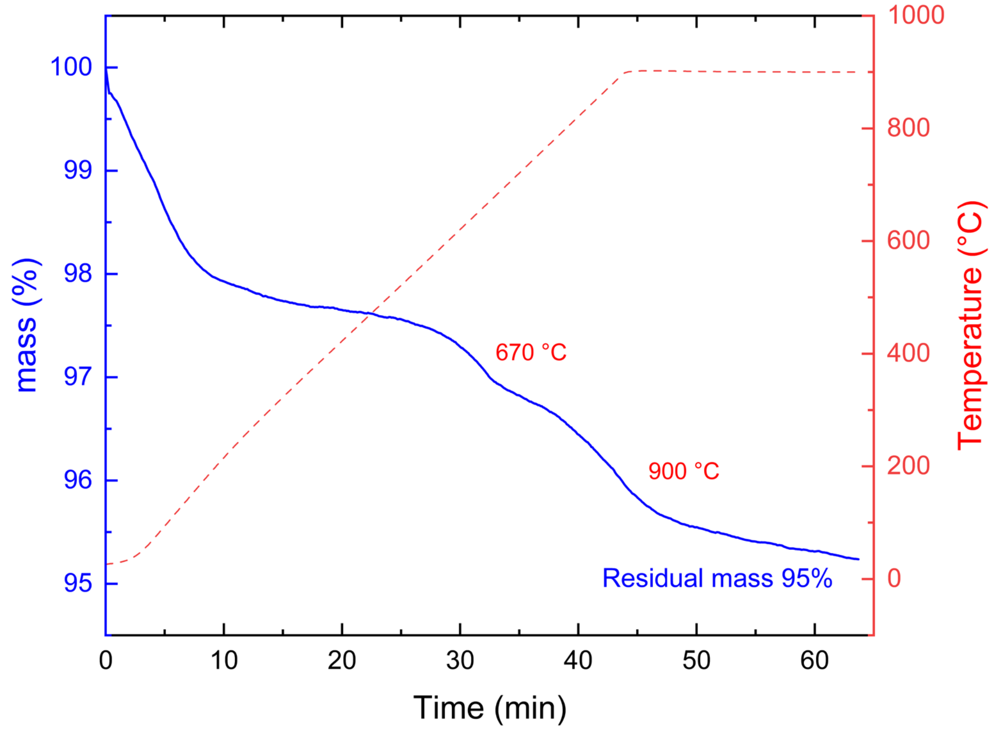

Figure 3. TGA thermogram showing mass loss versus temperature as a sample of rCB is heated to 900°C. The indicated temperatures show inflection points related to high temperature mass-loss events.

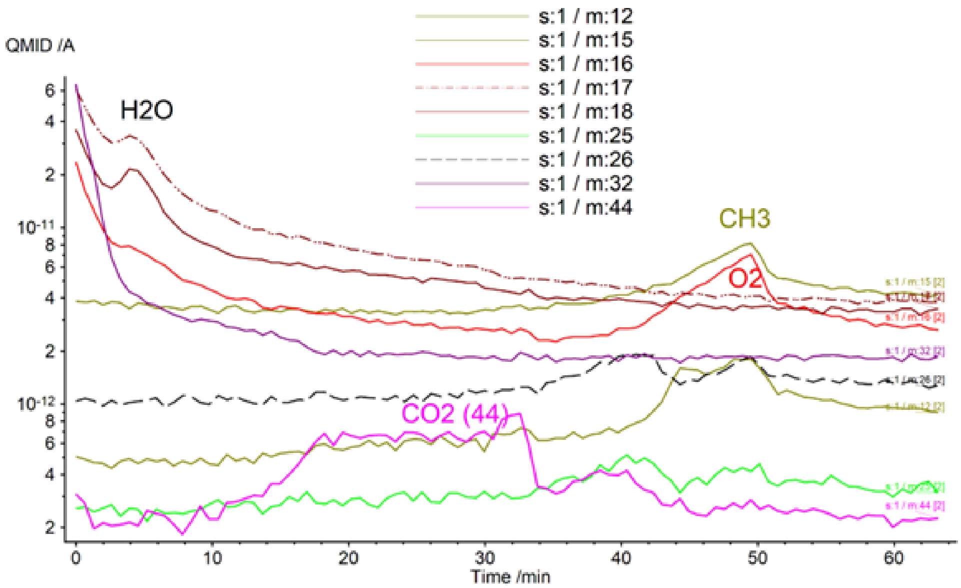

Figure 4. Quadrupole MS trace showing the evolution of gas species as a sample of rCB material is heated to 900°C.

Summary

TEM coupled with EDX analysis, and TGA coupled with mass spectrometry were applied to study recovered carbon black samples from Murfitts Industries.

TEM studies provide key functional parameters, such as particle and aggregate size and shape, and combination with EDX provides an understanding of the chemical components in the carbonaceous nanomaterial after recovery.

TGA yields information on the thermal stability and behaviour of the material, as well as the total mass content of volatile components present. Combining this with mass-spectrometry gives even greater levels of insight, confirming exactly which chemical species are released from the samples at specific temperatures.

By combining suite of advanced analytical techniques at the Bridge, it has been possible to obtain detailed insight into the structure of recovered carbon black materials. This in-depth understanding can shorten product development, accelerating the transition to circular production of products containing carbon black.

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