

Synchrotron based techniques for characterization of the chemical properties of recycled carbon fibers

THE INDUSTRIAL CHALLENGE

Carbon fibers from composites are already today a considerable waste stream and is foreseen to increase further. The interest from the automotive, energy and infrastructure segments on using recycled carbon fibers is rapidly increasing and the need for appropriate recycling technologies are substantial. This would also reduce the production of carbon fibers from fossil sources. To address this challenge, Enviro Systems AB has been a pioneer in the recycling of tires via pyrolysis into high value carbon products such as carbon black. A better understanding of the chemical and structural properties of recycled carbon products in general is important since these can be directly linked to the material properties observed at macroscale. For example, the chemical state of the surface of carbon fibres are critical for a good binding at the fiber /matrix interfaces in fiber-reinforced composite. Earlier studies on recycled fibers indicate that the surface properties are significantly affected by the pyrolysis process. In order to commercialize recycled carbon fibers chemical properties should be traceable and need to be properly analysed through the recycling process. The analysis is important for seeing the batch-to-batch variation, as well as for optimising and selecting the appropriate settings of the pyrolysis process.

WHY USING A LARGE SCALE FACILITY?

There are several lab-based analytical tools for analysing carbon fiber materials. However, synchrotron-based techniques may provide more detailed information regarding morphological/structural and chemical properties. A better knowledge of these properties can help developing a more profound understanding of the recycled product and thus enable optimisation of the thermal treatment process.

THE RESULTS AND EXPECTED IMPACT

For the chemical properties, two synchrotron based techniques were identified as

suitable, i.e. X-ray Photoelectron Spectroscopy (XPS) for studying the surface properties, and near edge X-Ray absorption fine structure (NEXAFS) for studying the bulk properties of the fibers. The latter possibly also when in the matrix. Both techniques provide information about the oxidation state and hybridization level of the carbon in the material, properties that are critical for the properties of the new material. XPS exists both as lab-based and synchrotron-based, where the latter provides much better energy resolution and more complex test environments. This enables the detection of adsorption status of organics and moisture in fine detail (e.g. by AP-XPS) or to perform depth profiling by varying the energy of the incoming photons (e.g. HAXPES). As a complement, NEXAFS can be used, revealing similar but also additional information as XPS such as oxidation state, chemical speciation of the carbon, valence, metal impurities, coordination geometry or near-neighbour distances. NEXAFS however goes deeper into the materials compared to XPS, capturing only the most outer surface layer. NEXAFS requires continuously tuneable X-ray energies, which is only available at synchrotron radiation facilities.

Another technique identified of used for analysing structural properties of the fibres is small angle X-ray scattering (SAXS), which can be used to determine orientation, shape, size and volume fraction of structural features resulting from different processing temperatures.

By using different setting in the thermal treatments a combination of X-ray spectroscopy (XPS and NEXAFS), lab-based Raman spectroscopy, and possibly SAXS, could result in a relatively complete understanding of the influence of the process on the critical properties of the fibres, and thus enabling optimisation of the settings. This will lead to a more efficient recycling and a better product – meeting the need of both society and industry.

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Vinnova's project No: 2019-02553 **Duration:** August 2019 -- May 2020

Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.