



# Nano-carbons from waste tyre rubber: An insight into structure and morphology



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## ABSTRACT

This study reports on the novel and sustainable synthesis of high value carbon nanoparticles (CNPs) from waste tyre rubber (WTR), using an innovative high temperature approach. As waste tyres are composed, primarily, of carbon – accounting for some 81.2 wt% – they represent a promising source of carbon for many potential applications. However, cost-effective options for their processing are limited and, consequently, billions of waste tyres have accumulated in landfills and stockpiles, posing a serious global environmental threat. The rapid, high temperature transformation of low value WTR to produce valuable CNPs, reported here, addresses this challenge. In this study, the transformation of WTRs was carried out at 1550 °C over different reaction times (5 s to 20 min). The structure and morphology of the resulting CNPs were investigated using X-ray diffraction (XRD), Raman spectroscopy, X-ray photon spectroscopy (XPS), N<sub>2</sub> isothermal adsorption method and scanning electron microscopy (SEM). The formation of CNPs with diameters of 30 and 40 nm was confirmed by Field Emission Electron Microscopy (FE-SEM). Longer heating times also resulted in CNPs with regular and uniform spherical shapes and a specific surface area of up to 117.7 m<sup>2</sup>/g, after 20 min. A mechanism that describes the formation of CNPs through mesophase nuclei intermediate is suggested.

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## 1. Introduction

Worldwide, over 1.2 billion used tyres are thrown away annually and at least four billion or so have accumulated in landfills and stockpiles. Such vast volumes of waste tyres not only pose a risk to human health and the environment but represent a significant waste of a potentially valuable carbon-rich resource. The environmental, health and economic imperatives for better management of waste tyres – and the need to recover the value embedded within them -- is well recognised by governments, industries and researchers. In landfills, waste tyres are not readily biodegradable and risk leaching toxic chemicals into the surrounding environment. Waste tyre stockpiles are also significant fire risks, as they are long burning and emit hazardous fumes. And, although waste tyres are a complex and technically challenging waste stream, their composition and its high calorific value – a typical tyre has a carbon content of ~81.2 wt% (Zhang and Williams, 2016) and a hydrogen content of ~7.2% – means they are of considerable interest for resource and/or energy recovery. Yet, despite considerable research and numerous technological developments,

applicable processes for the cost-effective recycling of waste tyres does not exist. New approaches are urgently needed, as the ever the increasing mountains of waste tyres worldwide continue to outstrip gains in recycling. In the US, for example, some 40% of the approximately 300 million tyres discarded last year were processed,<sup>1</sup> a significant improvement in the total volume of waste tyres recovered over previous years, but still a shortfall of some 180 million waste tyres. In the European Union, where more than two decades of regulations, targets and extended producer responsibility schemes have likewise greatly increased recycling rates (Landi et al., 2016), almost a third of waste tyres in some EU nations (EU13) continue to go to landfills. In Australia some 20 million passenger tyre require disposal annually, of which only 23% are recycled, 64% go to landfills and the remainder are dumped illegally, despite bans on illegal disposal in landfills in some Australian states due to toxicity.

Although the composition of various tyre types differs according to their application (Sahajwalla et al., 2005a), all tyres are primarily made up of carbon black, steel, natural rubber (NR) and synthetic rubber (SR). Due to their high calorific value (CV) (~32 MJ kg<sup>-1</sup>), waste tyres have been traditionally used as fuel

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<sup>1</sup> <https://cld.bz/Sk9Sjhe#60> P.55.

(Sahajwalla et al., 2005b; Rahman et al., 2005; Li et al., 2009; Liu et al., 2010). Energy recovery options (Zhang et al., 2015) include using waste tyres as an energy source for power stations and cement kilns and co-incineration with other waste streams. In the EU, for example, approximately 38% of waste tyres diverted from landfill are absorbed in energy recovery (Kordoghli et al., 2016). Current incineration methods, however, require sophisticated filters to prevent the emission of harmful pollutants and other cleaner pathways and processes are being intensely investigated (Kordoghli et al., 2016). Materials re-processing options include rubber crumbing and shredding for use in road surfacing, flooring, sports fields and other outdoor surfaces, roofing, etc. (40% in the EU) and the use of whole or shredded waste tyres in civil engineering applications such as the construction of highway shoulders and barriers (8% in the EU). Some waste tyres can also be recovered for re-treading and reuse (10% in the EU). The use of WTR as a resource in electric arc furnace (EAF) steelmaking has been investigated in other works (Sahajwalla et al., 2005a,b; Rahman et al., 2005; Gupta et al., 2006; Zaharia et al., 2009a,b; Sahajwalla et al., 2010), leading to a new commercialized technology (i.e. Polymer Injection Technology, PIT).

Recently, research interest has re-focused on pyrolysis. Although the pyrolysis of waste tyres has long been technically established, its commercial implementation has been hampered by challenges in finding viable markets for its products. New interest, however, has been sparked by the potential for the production of value added products, such as carbon nanoparticles, the subject of this study. In established waste tyre pyrolysis processes, WTRs are heated, typically, to between 400 and 600 °C in an oxygen free (or oxygen poor) environment and undergo thermal cracking, or thermal degradation, to produce three potentially useful products, an oil, gas and char (Kordoghli et al., 2016). The oil can be used directly or added to petroleum refinery stock, upgraded using catalysts to a premium fuel or used as a chemical feedstock. The gas has sufficient energy to fuel the pyrolysis process itself and the char typically consists of carbon black filler and the tyre rubber char. The char can be used as a solid fuel, carbon black or upgraded to produce activated carbon (Williams, 2013).

The diversity, physical and chemical characteristics and abundance of carbon materials has attracted significant research interest and effort over recent years. These materials have unique structures which result in superior properties such as excellent electrical conductivity and good mechanical resilience and they exhibit remarkable performances as insulating or semiconducting cathode materials. As such, they are widely used as electrode materials for renewable and green energy technologies (Lou et al., 2014; Wang et al., 2013). This group of materials, including activated carbon, nano-porous carbon, carbon nanotubes, graphene sheets and many other forms, have also been widely used in supercapacitors

(Frackowiak, 2007; Nishihara and Kyotani, 2012; Ghosh and Lee, 2012), lithium ion batteries (LIB) (anode materials) (Kaskhedikar and Maier, 2009; Cheng et al., 2011), and lithium-air/oxygen batteries (gas diffusion cathodes) (Christensen et al., 2012). Specifically, CNPs have exhibited superior electrochemical performance in LIBs and supercapacitors. High-surface-area and porosity are key factors that determine the suitability of carbons for use as electrode materials. The pores in electrode materials facilitate ion (i.e., charge) mobility, while the high-surface-area provides greater space (i.e., interface) for charge separation between the electrode surface and ions in the liquid electrolyte (Luo et al., 2016). The synthesis of carbon nanoparticles has been achieved through different techniques including the pyrolysis method (Ding and Olesik, 2004), microwave plasma chemical vapor deposition using iron and nickel as catalysts (Yu et al., 2002), electrolysis in molten alkali halide salts using carbon electrode (Hsu et al., 1996); microemulsion polymerization at low temperature (Jang et al., 2002).

In this study, we investigate rapid, high temperature (1550 °C) transformation of WTR to synthesise high value carbon. Although considerable research has been carried out on pyrolysis of WTR at temperatures below 900 °C over lengthy processing times (Jang et al., 2002, Cuesta et al., 1998, Sing et al., 1985, Brooks and Taylor, 1965, Cormia et al., 1962, Mileva et al., 2012, Nieto-Marquez et al., 2011, 2009a, 2009b, 2010, Sobkowicz et al., 2008), there is no data, to the best of our knowledge, on the rapid thermal transformation of WTR at high temperatures over short times periods (<5 min), nor has the characterization of the carbon residue obtained from such conditions previously been reported. This study introduces a clean, sustainable process for the thermal transformation of WTRs to form nano-carbon particles (CNPs) of 30–40 nm; a product with considerable technical and commercial potential for many applications. Although the process involves high temperatures, the overall energy consumption for the process is reasonable as the decomposition of WTR and the formation of CNPs occurs rapidly, and completely. At the same time, this rapid process minimizes the generation of toxic gases. Within five minutes, all the impurities, such as sulphur, zinc, and oxygen, have been eliminated. The CNPs synthesized using this new approach, and the possible mechanism of their formation, is discussed.

## 2. Materials and experimental procedure

The WTR samples of 1–2 mm rubber shreds from waste passenger tyre were supplied by OneSteel, Australia, as shown in Fig. 1a. Their composition is detailed in Table 1. To get rubber powder from waste tyres, several steps are involved (This process is performed by industry). The first step is shredding and separating (steel and iron made) wires and mesh from tyre and breaking them into small pieces using a machine. The Granulators are used in the

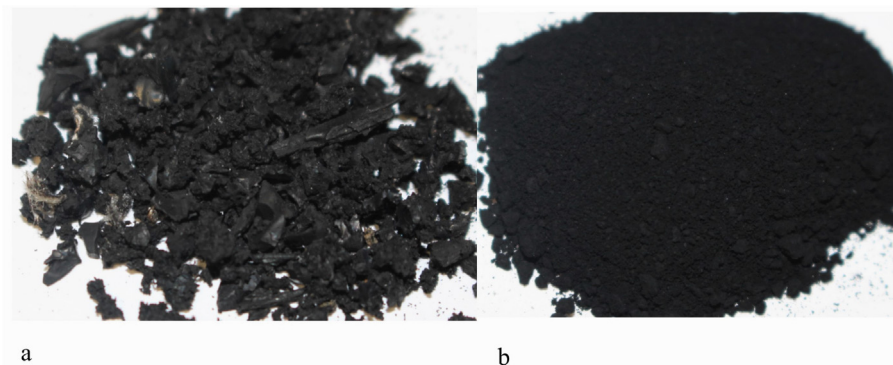


Fig. 1. The general feature of the a) WTR and b) the carbon residue after heat treatment.

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