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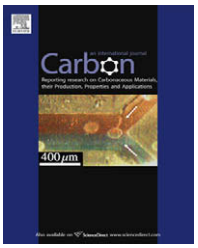

Hydrothermal preparation of carbon microspheres from mono-saccharides and phenolic compounds

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ABSTRACT

Isolated carbon microspheres in a diameter range of 1–4 μm were prepared by hydrothermal treatment of mono-saccharides (xylose for pentose and fructose for hexose) with phenolic compounds–phenol, resorcinol, and phloroglucinol. It was found that addition of these compounds, particularly phloroglucinol, into a sugar solution led to a substantial increase (roughly 20-fold) of the carbon yield. Mono-saccharides are dehydrated into furan compounds during hydrothermal carbonization, and these compounds subsequently react with phloroglucinol, as revealed by examination of high-performance liquid chromatograms. Through elemental analysis and spectroscopic studies, it was found that several oxygen functionalities including hydroxyl, carbonyl, carboxyl, ether, quinone, and ester groups were located throughout the carbon spheres derived from sugars and phloroglucinol. On the basis of the carbon formation mechanism, the C–O–C bond formed by linkage of OH groups of phenolic compounds preferentially exists at the core of carbon spheres whereas the outer carbon surface contains reactive oxygen functionalities such as hydroxyl, carbonyl, carboxylic, and ester groups, corresponding with results obtained with sugar-derived carbon materials.

1. Introduction

In recent years, there have been a great number of reports regarding size-controlled carbon microspheres synthesized via hydrothermal treatment of biomass from poly- [1–4] to mono-saccharides [5–8], hexose and pentose, where the size of carbon spheres, ranging from about 100 nm to micrometers, could depend directly on variation of the hydrothermal processing conditions, such as the reaction temperature, reaction period, and reactant concentration [5,6]. They have found use in numerous applications, including as a template for the preparation of hollow metal and metal oxide shell structures [9–11]. In addition, the hydrothermal treatment of sugars has been utilized to produce porous carbon [12,13], metal–carbon [6,14] or metal oxide–carbon composites [15,16], and hollow carbon [17].

In terms of hydrothermal carbonization of glucose and fructose, it was first considered that glucose first loses water through an intermolecular condensation reaction due to its stable pyranose structure [6], whereas fructose is initially converted to 5-hydroxymethyl-2-furaldehyde (HMF) through an intramolecular dehydration process [5]. However, recent studies have suggested that hydrothermal carbonization involves two steps: hexose (glucose and fructose) and pentose (mainly xylose) are converted into HMF and furfural, respectively [1,5]. The resulting furan compounds are subsequently condensed and polymerized to form carbon materials. In this respect, the local structure of carbon spheres derived from glucose was recently observed using the ^{13}C solid state cross-polarization magic-angle spinning (CP-MAS) nuclear magnetic resonance (NMR) technique, where α-carbon of the HMF units is

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