

Dispersion stability of the aminosilane-grafted mesoporous carbons in different solvents

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ARTICLE INFO

Keywords:

Nanomaterials
Particle size distribution
Modification of mesoporous carbons
3-aminopropyltriethoxysilane
Multiple light scattering
Zeta potential

ABSTRACT

In this study, 3-aminopropyltriethoxysilane (APTES) was grafted on the surface of oxidized mesoporous carbon of cubic structure. The process of modification was carried out at 40 °C for 8 h with increasing amount of aminosilane (1.2, 3, 6 mL). The new hybrid materials were characterized by FT-IR spectroscopy, low temperature nitrogen sorption, scanning electron microscopy, elemental analysis, thermogravimetric methods, Boehm titration, laser diffraction, dynamic and multiple light scattering. Functionalization of carbon with aminosilane significantly affected its structural, textural and surface properties. The chemical interaction of aminosilane with the oxidized carbon material was confirmed by FT-IR spectra. Thermogravimetric analysis of the functionalized mesoporous carbons exhibited three peaks at temperatures in the range 60–650 °C that correspond to the degradation of chemically grafted aminosilane on the materials surface. Multiple light scattering was applied to assess the stability of the materials in water, toluene and Tween 80 non-ionic surfactant solutions. In polar solvent all aminosilane-grafted mesoporous carbons were more stable than the unmodified material. In toluene and surfactant solution the sample modified with the lowest amount of APTES showed the highest stability. Additionally, it was proved that the more acidic groups were on the surface of mesoporous carbons the more stable was the dispersion in surfactant solution.

1. Introduction

Synthesis of ordered mesoporous carbons with a large number of organic functional groups uniformly dispersed on pore walls has been intensely studied for many years [1–5]. Such materials are of interest for application in adsorption, catalysis, chromatography, nano- and biotechnology [2–4]. The content of functional groups on the carbon surface is the main factor determining acid-base properties of these materials, their sorption capacities towards dyes [1,3,4], toxic metal ions (e.g. mercury, cadmium, arsenic, lead, copper) [6–8], or biologically active substances [9,10], ion-exchange capacities, surface hydrophobicity and hydrothermal stability [11,12]. The type and character of organic groups applied for functionalization as well as their dispersion affect the surface activity of carbon materials and accessibility of their pores to certain molecules. The most often applied and most effective method of mesoporous carbon modification, leading to generation of carboxyl, hydroxyl, lactone, carbonyl or ether groups which can serve as adsorption centers or grafting points for other species (usually achieved via covalent bonding) is oxidation [1,13,14]. Many functional groups can be covalently attached in this

way to the surface of ordered mesoporous carbons, including basic and acid functionalities [1–6].

A very interesting and relatively new direction of studies is modification of carbon materials with trialkoxysilanes [6,15]. The head group of trialkoxysilanes is $-\text{Si}(\text{OR})_3$, where R is an arbitrary alkyl group, usually a methyl group $-\text{CH}_3$ or ethyl group $-\text{C}_2\text{H}_5$. With the help of this group the trialkoxysilane molecules can make covalent bonds with the hydroxyl groups present on surfaces of supporting materials [16–18]. At the first stage of functionalization trialkoxysilane undergoes hydrolysis prompted by the water adsorbed on the surface or in solution. This process generates trisilanols that are weakly bound to the surface through hydrogen bonds. Trisilanols undergo condensation due to the reaction of silanol groups (Si-OH) with the surface hydroxyl groups [16–18]. Barczak et al. have reported functionalization of oxidized mesoporous carbon of hexagonal structure (CMK-3) using N-(3-trimethoxysilylpropyl)diethylenetriamine [6] and 3-aminopropyltriethoxysilane [15]. It has been proved that both oxidation and functionalization treatment lead to deterioration of mesoporous carbon structure, reduction of its surface area and pore volume [6,15]. The carbon modified with N-(3-

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trimethoxysilylpropyl)diethylenetriamine has been tested as adsorbent for removal of heavy metals (Pb, Zn, Cu, Cd) and had much greater sorption capacities than the pristine material [6]. A very effective modification of the ordered mesoporous carbon CMK-3 has been proposed by Hamad et al. [19]. They have functionalized the pristine material with 3-aminopropyltrimethoxysilane (APTMS), and used the obtained samples for adsorption of lead and copper ions. The samples obtained have shown high sorption capacities towards Pb^{2+} ($q_e = 3.5 \text{ mmol/g}$) and Cu^{2+} ($q_e = 8.6 \text{ mmol/g}$).

The correct choice of organic functional groups for modification of mesoporous carbon materials permits extension of their sorption capacities towards different type pollutants and changes their neutral and hydrophobic surface into more hydrophilic one [3,4,15,19]. From the point of view of adsorption processes in liquid phase, very important is the stability of dispersion of functionalized carbons in different solvents [11,20]. Industrial wastes contain many different chemical compounds not only in the form of water solutions but also in hardly water soluble liquids, emulsions, suspensions and tars. Industrial wastes often contain toluene, benzene, aniline, phenol, amines, nitro-compounds, alcohols, esters, salts, inorganic acids (mainly HNO_3 , H_2SO_4), dyes, heavy metal ions and surfactants in large amounts [3,6,21–27]. Therefore, the stability of dispersion of a potential adsorbent in different solvents is of key importance, similarly as the rate of sedimentation, particle size distribution or zeta potential.

The aim of our study was to check the effect of mesoporous carbon functionalization with different amounts of 3-aminopropyltriethoxysilane (APTES) on its structural, textural, surface properties and stability of dispersion in water, toluene and aqueous solution of non-ionic surfactant Tween 80. The dispersion stability of the aminosilane-grafted mesoporous carbons was analyzed by multiple light scattering. Additionally, the dynamic light scattering with non-invasive backscatter optics was applied to determine the zeta potential of nanoparticles.

2. Materials and methods

2.1. Sample preparation

2.1.1. Synthesis of mesoporous carbon

Mesoporous carbon material ($\text{C}_{\text{KIT-6}}$) of cubic structure was obtained by the hard-template method with the use of ordered mesoporous silica KIT-6 as a solid matrix and sucrose as the carbon source, according to the reported procedure [21,22,28].

The hydrothermal synthesis of mesoporous silica KIT-6 was carried out as follows. A portion of 4 g of triblock copolymer Pluronic P123 ($\text{EO}_{20}\text{PO}_{70}\text{EO}_{20}$, Aldrich) was dissolved in a mixture of 144 g of distilled water and 7.9 g of hydrochloric acid (POCh) at 35 °C. Then 4 g of butan-1-ol (POCh) was added upon stirring. The mixture was stirred for 1 h at 35 °C, then 8.6 g of tetraethyl orthosilicate (98% wt, Aldrich) as a source of silicon, was added dropwise. The solution was stirred for 24 h at 35 °C and subjected to hydrothermal treatment in a polypropylene bottle (24 h at 100 °C). The product was filtered off and dried at 100 °C for 24 h. The template was removed by calcination at 550 °C for 8 h. Then 1 g of KIT-6 silica was subjected to impregnation with 1.25 g of sucrose (Aldrich) dissolved in 0.14 mL of sulfuric acid (POCh) and 5 mL of distilled water. The obtained material was heated in an oven for 6 h at 100 °C, then for the next 6 h at 160 °C. The impregnation was repeated with the use of 0.8 g of sucrose, 0.09 mL of sulfuric acid and 5 mL of distilled water. Once again, the silica-carbon composite with partly polymerized sucrose was heated for 6 h at 100 °C and then for 6 h at 160 °C. The obtained material was subjected to carbonization at 900 °C (8 h) in argon atmosphere and silica was washed out with a 5 wt % solution of hydrofluoric acid.

2.1.2. Oxidation of mesoporous carbon

Firstly, pristine mesoporous carbon $\text{C}_{\text{KIT-6}}$ was oxidized with the use of 1 mol/L ammonium persulfate solution (Aldrich) – APS (prepared in 2 mol/L sulfuric acid). For a typical treatment, 0.5 g of mesoporous

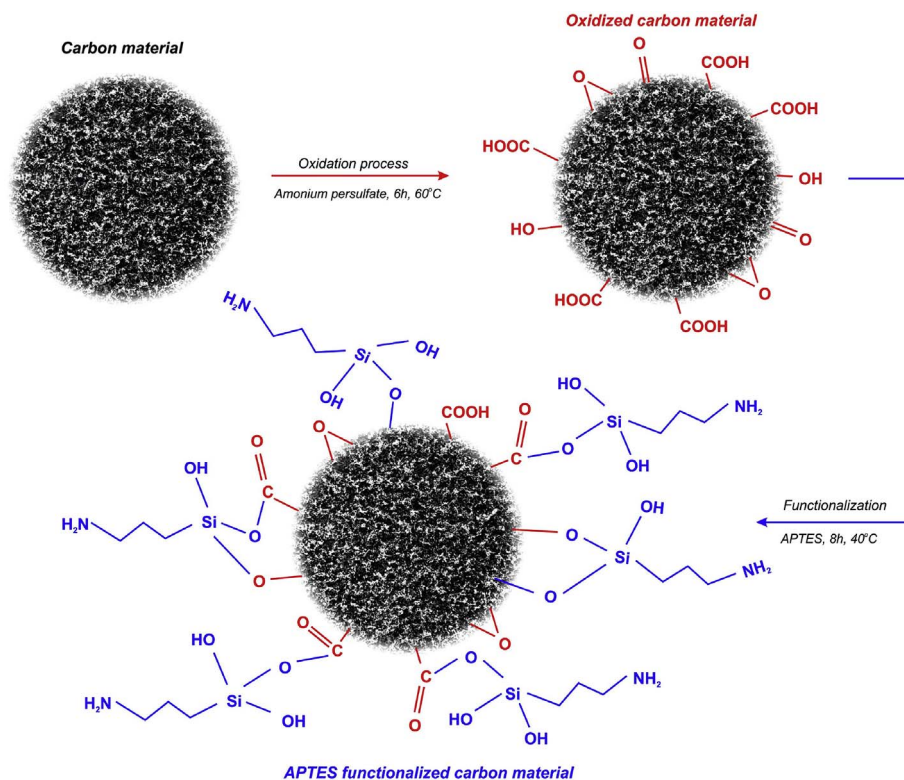


Fig. 1. The way of functionalization of ordered mesoporous carbon.

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