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HIGH PRODUCT YIELD IN A NARROW COLUMN CONFIGURATION OF CARBON NANOTUBES: A PATHWAY FOR NANOSYNTHETIC MACHINE

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Abstract

Multiwalled functionalized carbon nanotubes have been aligned in a narrow column configuration of 3.1 cm X 0.2 cm through which the organic reactants are allowed to travel through gravity. The products coming out of the column have been examined by GC/MS and UV-VIS absorption spectroscopy. With organic reactants such as aniline and hydrogen peroxide in 1:1 molar ratio, a high yield of azo compound has been obtained through the coupling of nitrosobenzene and aniline. This suggests that the column configuration is ideal for organic synthesis and for the development of nanosynthetic machines.

Key words: Multiwalled carbon nanotubes, functionalized carbon nanotubes, narrow column configuration, azobenzene, nitrosobenzene, aniline, nanosynthetic machine

1. INTRODUCTION:

Carbon nanotubes are interesting materials for carrying out chemical reactions in confined spaces. The carbon nanotubes have diameters ranging from a few angstroms to several nm (1-3). Several inorganic reactions have been carried out (4-6) inside the nanotubes; these reactions require special conditions of filling the nanotubes such as condensing vaporous materials or flowing molten liquid of salts at high temperatures. Many metals have been deposited inside the carbon nanotube by these methods. The capillary action of the carbon nanotubes have also been reported (5,7). Functionalization of the carbon nanotubes generally helps the filling of the nanotubes especially when charged groups are present. It also helps the solubilization of the carbon nanotubes (8-12).

We have carried out organic chemical reactions with the functionalized multiwalled carbon nanotubes (carboxylic acid group) in a column configuration. The column provides the catalytic pathway for the reaction to occur. We obtained high yield of the products of oxidation of aniline, p-toluidine and N-methylamine.

2. EXPERIMENTAL:

Aniline (Aldrich) was purified by distillation and a sample distilling at 180° C was collected and stored in a brown bottle. p-Toluidine (Aldrich) was used as received. N-Methylamine was 40 wt. % solution in water (Aldrich) and used as such in the experiments. 30% (Vol.:Vol) Hydrogen peroxide (Baker, analytical grade) was used for the oxidation.

Multiwalled carbon nanotubes were either bought from Deal International (Rochester, NY) or laboratory synthesized at RPI. The synthesized samples showed higher responses compared to the commercial samples. The tubes were functionalized as discussed in the literature (13). The TEM of the functionalized carbon nanotubes are shown in Figure 1.



Figure 1. TEM of the functionalized carbon nanotubes

The TEM, IR spectrum and sample analysis by Atomic absorption spectroscopy suggests the absence of any metals in the functionalized carbon nanotubes.

3. RESULTS AND DISCUSSION

The oxidation of aniline (aniline:hydrogen peroxide:1:1) in narrow column configuration produced azobenzene as the single oxidation product when the solution was analyzed by GC/MS. Table 1 gives the mass numbers identified in the GC/mass fragments. In these experiments, the reactants run through the column in about eight minutes and hence the product contained a large excess of unoxidized aniline. The mass spectrum showed a primary peak with a mass number of 182 which was due to the azobenzene. The fragments arising are identifiable with the authentic sample of the azobenzene sample. The product obtained in the oxidation of p-toluidine was identified as 4,4'-dimethylazobenzene with a mass number of 210. These results are further

supported by UV-VIS absorption spectroscopy. Two well defined peaks with maxima at 347 and 425 nm appeared for the aniline oxidation products which are characteristic of azobenzene. The spectral features of the oxidation products of p-toluidine (Figure 2) showed a maximum at 464 nm which is the characteristic of 4,4'-dimethylazobenzene. The experiments carried out with N-methylamine are complicated by the purity of the starting material. Since it is a gas at room temperature, the commercial samples are 40% in water. Hence, the oxidations were carried out using mixed solvents such as acetone-water or acetonitrile-water. The oxidative reaction is exothermic and produces azomethane and other products. We hope to investigate this reaction in detail using pure N-methylamine.

Table 1: GC/MS Fragmentation Data

Species oxidized	Mass Nos.
Aniline ^a	28,77,93,105,152,182
p-Toluidine ^a	28,92,120,210
N-Methylamine ^b	15,28,43,58

a: Solvent: acetonitrile b: Acetone

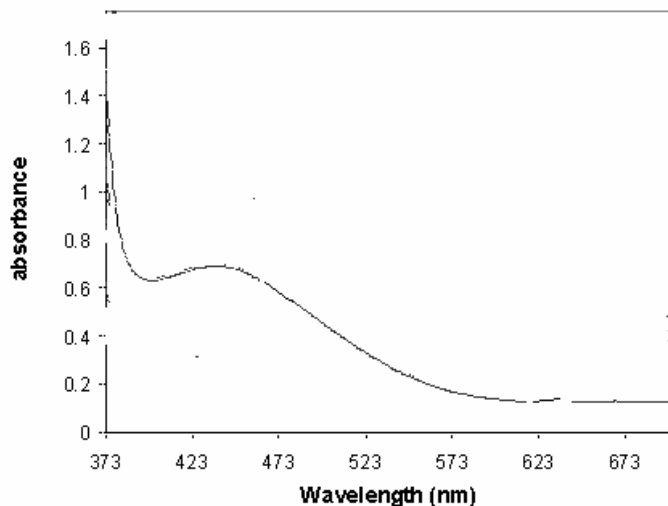


Figure 2. Oxidation product of p-toluidine absorption spectrum at carbon nanotubes

The results of these studies show that the column configuration produces products of high yield of azo compounds.

4. CONCLUSIONS:

It has been demonstrated that the functionalized carbon nanotubes in a column configuration provides a method to reduce the oxidative reactions in solution and increase the confinement reaction. The coupling of nitrosobenzene with aniline proceeds efficiently in the column configuration.

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