Microwave-assisted acid digestion method for purification of carbon nanotubes

Pełech I.*[1], Narkiewicz U.[1], Owodziń K.[1], Borowiak-Paleń E.[1]

[1]*Institute of Chemical and Environmental Engineering, West Pomeranian University of Technology, Pułaskiego 10, 70-310 Szczecin, Poland*
**e-mail: ipelech@zut.edu.pl*

Carbon nanotubes can be produced at a large scale using chemical vapor deposition method. The CVD method involves catalyst-assisted decomposition of hydrocarbons. The best results are obtained with iron, nickel and cobalt catalysts supported on various carriers. While the strong points of the CVD method are connected with its low production costs, the method's weak point is the quality of the obtained product – i.e. the presence of defects, amorphous carbon and metal particles. To purify CNTs from carbon impurities, chemical oxidation including gas phase oxidation using air or steam or by liquid phase oxidation using potassium permanganate, hydrogen peroxide or nitric acid is applied. The separation of carbon nanotubes from catalyst traces is most often carried out using acid reflux method in the presence hydrofluoric, hydrochloric, sulfuric or nitric acid.

The present work deals with the synthesis of multi walled carbon nanotubes by chemical vapor deposition using ethylene as a carbon source and nanocrystalline iron with an addition of a small amount (2-3 wt.%) of CaO and Al_2O_3 as the catalyst To the purification microwave-assisted acid digestion method was applied. Carbon materials directly after synthesis or additionally treated under air atmosphere was immersed in the Teflon vessel filled with nitric or hydrochloric acid. Then the whole was placed in the reactor heated with microwaves. Experiments were conducted under pressure in the range form 10 to 30 at. Reaction time from 5 to 60 minutes was changed.

To characterize CNTs different methods were employed. The phase composition of the samples was studied using X-ray diffraction method. Transmission electron microscopy was used to determine the type of carbon nanotubes obtained and to verify their structures. To determine metal quantity in the samples thermogravimetric analysis was applied.