

MESOPOROUS SILICA FUNCTIONALIZED BY NICKEL-CYCLAM MOLECULES: PREPARATION AND RESONANCE RAMAN STUDY

MAGDALENA LASKOWSKA¹, ŁUKASZ LASKOWSKI², KAZIMIERZ DZILINSKI¹

1. Institute of Physics, Czestochowa University of Technology, PL-42-200 Czestochowa, Al. Armii Krajowej 19, Poland.

2. Department of Computer Engineering, Czestochowa University of Technology, PL-42-200 Czestochowa, Al. Armii Krajowej 36, Poland.

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Mesoporous silica SBA-15 functionalized by (1,4,8,11-tetraazacyclotetradecane) cyclam groups containing nickel ions (Ni-cyclam) was synthesized by two different approaches, and investigated by resonance Raman spectroscopy. Vibrational features of organometallic molecules are analyzed for (Ni-cyclam) groups grafted in the silica pores. An assignment of bands in resonance Raman spectra was done to monitor the structure and properties of the mesoporous silica material with regard to the methods of synthesis used in this study. It was shown, that Raman scattering can be useful for probing of functionalization's efficiency of mesoporous silica. On the base of the resonance investigation: Raman and EPR spectroscopy, distribution of the functional groups inside pores can be determined. In the present article the Raman spectroscopy is treated as a complementary research to EPR investigation.

It was shown that a clustering of the active groups alter significantly the resonance Raman spectra through broadening and shifts of the corresponding bands in comparison with separated molecules. Results obtained from the analysis of the resonance Raman spectra indicate significant differences between the samples prepared by the two procedures. The discussion of the Raman results was referred to EPR results, and on the base of this authors concluded about correct achievement of the functionalization.

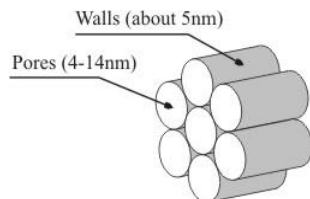
INTRODUCTION

Nanotechnology represents certainly one of the most promising developments of industry. One of the major challenges in this field is the control of physical or chemical properties of materials through modeling their structure in the nanometric scale. Elaboration of the synthesis route of MCM-41 mesoporous silica (Beck, Vartuli, Roth, Leonowicz, Kresge, Schmitt, Chu, Olson, Sheppard, McCullen, Higgins, Schlenkert, 1992) was a milestone in nanotechnology. This inorganic material has an open structure and a well-defined geometry. Mesoporous silica has a form of thin and long rods with walls built with amorphous silica (SiO_2). The rods

arrangement is usually regular, often hexagonal. Development of composite materials, such as mesoporous silica, containing pores which size can be easily and accurately controlled, has a great importance for science and technology. Nowadays, it is possible to obtain mesoporous silica with pores in the hexagonal, cubic or lamellar arrangement with diameter from 1,5 to 15 nm depending on synthesis conditions and a type of a surfactant.

Mesoporous silica SBA-15 (Santa Barbara Amorphous) (Zhao, Huo, Feng, Chmelka, Stuc, 1998) is the main object of investigation in this paper. The structure of this material is shown in Fig.1.

a)



b)

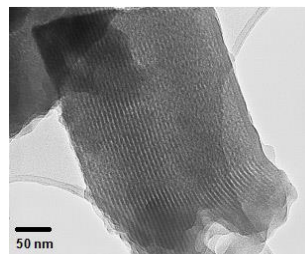


Fig.1. The structure of SBA-15 type mesoporous silica: scheme (a) and microphotography (b)