

## CHARACTERISATION OF NANOPOROUS POLYMERIC SPME COATINGS WITH USE OF SAXS MEASUREMENTS

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Keywords: solid phase microextraction, electropolymerisation, small angle x-ray scattering

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The recent trend in sample preparation methods are focus on the miniaturization and development of new selective materials. The number of studies set the conducting polymers as one from the most essential group of materials which can be applied with success as an adsorbent in solid phase microextraction (SPME). These kinds of polymers are the area of our interests because of a several important aspect. Mainly due to possibility to synthesis of these polymers on electrochemical polymerization way. This method allows for a broad of variety changing while preparing fibers *i.e.* thickness, existence of counter ions and porosity. Thickness of the fibers may be rapidly measured by use of Scanning Electron Microscope (SEM) (Fig. 1) but porosity investigations of such materials contains very small pore size procure plenty of embarrassment.

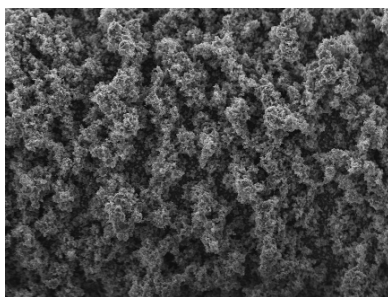


Figure 1. SEM micrographs with structure of polypyrrole SPME fiber obtained on the electrochemical polymerisation way.

First investigation was directed on the measurement with use of low temperature adsorption of nitrogen but with use of this technique we could not obtain a satisfactory results caused by low resolution of apparatus and small pores existed in porous polymeric materials. Simultaneously a rehearsal investigation with applying a Small Angle X-ray Scattering measurement was applied. This examination was performed with use of diffractometer NanoSTAR (Bruker-AXS) and HiSTAR detector with high resolution.

Measurements with use of SAXS were performed for polypyrrole and polythiophene SPME coatings obtained on the electrochemical polymerisation way with thickness in range 90-95 and 145-155  $\mu\text{m}$ , respectively.

Calculated pore size range for these materials were in range from 400 to 1300  $\text{\AA}$  both for polypyrrole and polythiophene coatings. Example plot is presented in Fig. 2. However some differences between these materials especially in small and high pore size range were observed.

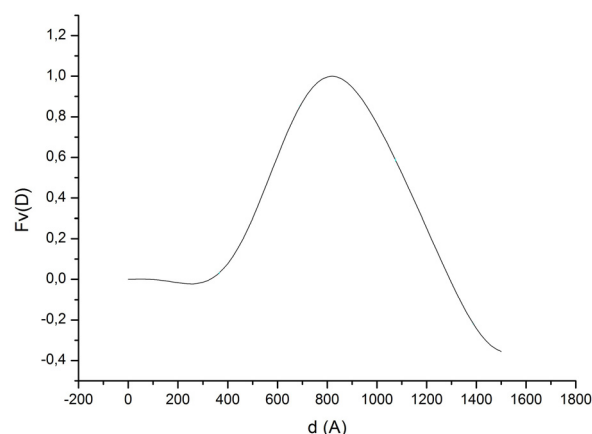


Figure 2. SAXS measurements of polythiophene SPME coating.

Synthesized and characterized fibers were used in SPME for sampling a medically important antibiotics from standard solutions, human plasma and whole blood. These investigations create a possibility to apply the described method in experienced biomedical analysis [1].

**Acknowledgments:** This work was supported by Ministry of Science and Higher Education (Warsaw, Poland) Grant. No. 2680/B/H03/2010/38 and by European Social Found, Polish National Budget, Kujawsko-pomorskie Voivodship Budget (within Sectoral Operational Programme Human Resources) "Krok w przyszłość" and "Stypendia dla doktorantów 2008/2009-ZPORR".

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