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Electrochemical synthesis and properties of polyporphyrin films based on 5,10,15,20-tetra(4-pyridyl)porphyrin

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Materials and methods

Starting 5,10,15,20-tetra(4-pyridyl)porphyrin 1 was prepared by the known procedure with modifications [S1,S2].

To a boiling mixture of acetic acid (500 ml), nitrobenzene (150 ml) and acetic anhydride (27 ml, 0.286 mol) was added gradually a mixture of pyrrole (10 ml, 0.144 mol) and pyridine-4-carboxaldehyde (13.7 ml, 0.144 mol). The mixture was boiled for 1.5 h and cooled down. After the acetic acid was removed by rotary distillation, the residue was diluted with water (150 ml), after which the nitrobenzene was steam-distilled off. The residue was filtered, washed with water and dried in air. The residue was them boiled with methanol (100 ml) under stirring; the solid was filtered, washed with methanol and dried. The material was extracted by chloroform in a Soxhlet apparatus until discoloration of the solvent. The extract was evaporated to 200 ml, and the resulting precipitate was filtered out. The solution was chromatographed on a column (3 × 50 cm) with aluminum oxide, Brockmann grade II. The eluate was evaporated up to the minimum volume and product **1** was precipitated with methanol (50 ml). The porphyrin was filtered, washed with methanol (50 ml) and dried at 70 °C until a constant weight. Yield: 6.3 g (25%). R_f 0.80 (silufol, $CHCl_3$ / MeOH 5:1).

UV-vis (λ_{max}, nm (lgε), $CHCl_3$): 643 (3.43); 589 (3.82); 546 (3.79); 514 (4.30); 417 (5.62).

^{1}H NMR ($CDCl_3$): 8.87s (8H, β-H); 9.08d (8H, 2,6-H); 8.18d (8H, 3,5-H); -2.92s (2H, NH).

Mass spectrum, m/z: 619.27 $[M+H]^+$. The calculated value was 618.70.

Electrochemical measurements

The electrochemical measurements were made with an SP-150 potentiostat (Bio-Logic Science Instruments, France).

UV-Vis spectra.

The electronic absorption spectra in the range of 350-800 nm of the porphyrin solution in dichloromethane and polyporphyrin film on an ITO electrode were registered with a Cary 50 spectrophotometer (Varian, USA).

IR-spectra.

The IR-spectra of the studied compounds were obtained on a Bruker Vertex 80 spectrometer (Germany) in the region of 4500-350 cm^{-1} with the resolution of 0.1 cm^{-1}.

References

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S2 Q. Lin, C. Mao, A. Kong, X. Bu, X. Zhao and P. Fen, *J. Mater. Chem. A*, 2017, **5**, 21189.