Polymerization Initiated by Graphite Intercalation Compounds Revisited. One-Pot Synthesis of Amphiphilic Triblock ABC Copolymers†

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† This paper is dedicated with best wishes to Professor Iliya Bl. Rashkov on his 80-th birthday

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Figure S1. Break-seal ampoule with Madagaskar graphite intercalation compound KC_{24}. Synthesized - December 1989; picture taken - April 11, 2022.

Calculation of the styrene (St), methyl methacrylate (MMA) and ethylene oxide (EO) molar fraction in the ABC copolymer using ^{1}H NMR spectroscopy.

A –integral intensity of St aromatic protons (5 H, 7.25-6.35 ppm); B - integral intensity of MMA –C(C$\underline{H}_3$)- protons (3H, 1.3-0.6 ppm); C - integral intensity of EO -C$\underline{H}_2$C$\underline{H}_2$O-protons (4H, 3.64 ppm).

$$St\ molar\ fraction = \frac{\frac{A}{5}}{\frac{A}{5}+\frac{B}{3}+\frac{C}{4}};\ MMA\ molar\ fraction = \frac{\frac{B}{3}}{\frac{A}{5}+\frac{B}{3}+\frac{C}{4}};\ EO\ molar\ fraction = \frac{\frac{C}{4}}{\frac{A}{5}+\frac{B}{3}+\frac{C}{4}}$$

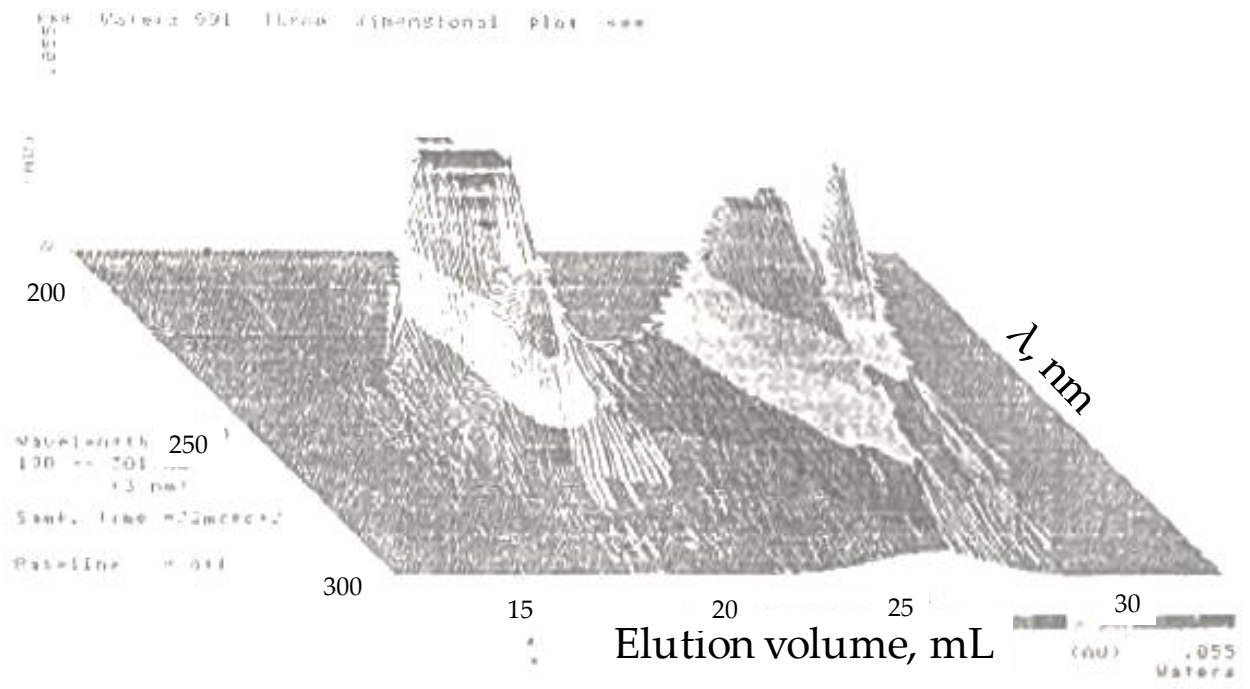
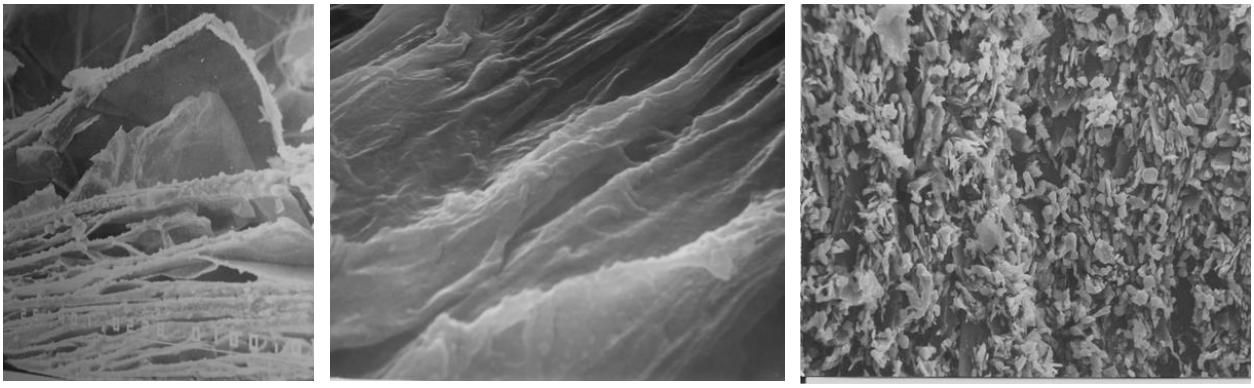


Figure S2. Size-exclusion chromatography of cyclohexane fraction extracted after consecutive addition of comonomers St – MMA – EO and bulk copolymerization (STEM 2, Table 1). 3D plot from the M991 UV-PDA Waters detector.



(a) **(b)** **(c)**

Figure S3. Delamination of HOPG KC_{24} during polymerization monitored by scanning electron microscopy. JEOL Super-probe 733 (JEOL Corporation, Japan). (a) Initial stage – graphene sheets start to separate with polymer protruding. Magnification 100×. (b) Final stage – sheets fully embedded in the polymer. Magnification 1100×. (c) Delaminated HOPG after polymer dissolution and extraction. Magnification 800×. HOPG - highly oriented pyrrolytic graphite (Union Carbide, USA).