

A Recycled Foam Coloring Approach Based on the Reversible Photo-isomerization of Azobenzene cationic surfactant

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Synthesis:

4-hydroxy-4'-butoxyl azobenzene (HBAzo):

6.35 g of DHAzo, 6.14 g of K₂CO₃ (1.5 eq) and 4.92 g of KI (1.0 eq) were placed in 30 mL EtOH and simultaneously stirred in the three-necked flask equipped with a mechanical stirrer and a reflux condenser at 80 °C for 0.5 h. Bromobutane (1 eq) dissolved in EtOH (40 mL) was then dropwise added to react with DHAzo at 80 °C for 8 h. Afterward, the crude was diluted with water and the product precipitated as a yellow solid. The solid was filtered, followed by further purified with column chromatography (Silica gel, petroleum ether/EtOAc 7/1). Finally, the product was dried under vacuum overnight. ¹H NMR spectrum is shown in Figure S1: (400 MHz, CDCl₃) δ 7.84 (dd, J = 18.7, 8.8 Hz, 4H), 6.95 (dd, J = 33.5, 8.8 Hz, 4H), 4.03 (t, J = 6.5 Hz, 4H), 1.85 – 1.76 (m, 2H), 1.57 – 1.46 (m, 2H), 0.99 (t, J = 7.4 Hz, 3H).

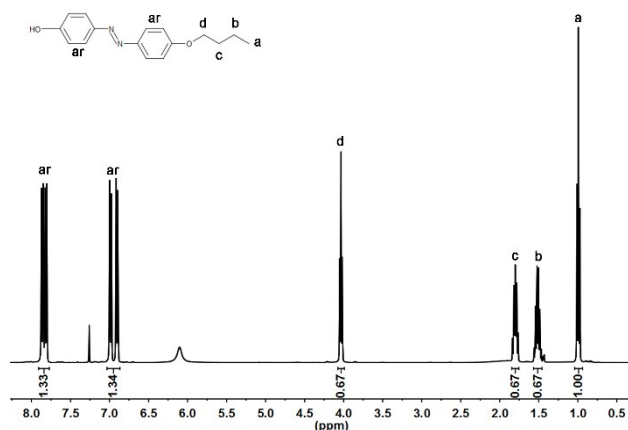


Figure S1. H-NMR spectrum of HBAzo.

4-bromoethyl-4'-butoxyl azobenzene (BEBAzo):

4.5 g of 1,2-dibromoethane (3 eq) and KOH (3 eq) were placed in 40 ml anisole and maintained at 75 °C for 0.5 h, accompanying with vigorous stirring in the three-necks flask. Thereafter, HBAzo (1 eq) dissolved in anisole was dropwise added to the flask and thoroughly stirred at 75 °C for 16 h in reflux condition. Then, the reaction system was cooled to ambient temperature, followed by removing anisole via a rotary evaporator under reduced pressure.

Furthermore, the crude was extracted by EtOAc. After removing the EtOAc, the product was further purified with column chromatography (Silica gel, petroleum ether/EtOAc 7/1) finally precipitated as a yellow solid after removing the eluent. The product was dried under vacuum overnight. ^1H NMR spectrum is shown in Figure S2: (400 MHz, CDCl_3) δ 7.87 (dd, $J = 8.8, 1.7$ Hz, 4H), 7.00 (dd, $J = 8.8, 7.2$ Hz, 4H), 4.36 (t, $J = 6.3$ Hz, 2H), 4.04 (t, $J = 6.5$ Hz, 2H), 3.67 (t, $J = 6.3$ Hz, 2H), 1.85 – 1.76 (m, 2H), 1.58 – 1.47 (m, 2H), 1.00 (t, $J = 7.4$ Hz, 3H).

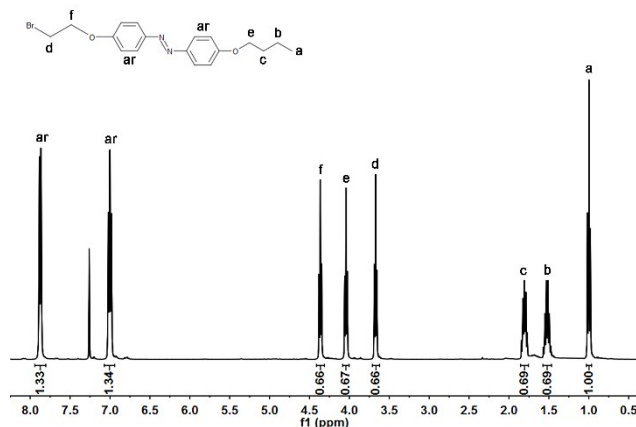


Figure S2. ^1H -NMR spectrum of BEBAzo.

4-butoxy-4'(trimethylaminoethoxy) azobenzene (BTAEAzO):

10 g of BEBAzo was dissolved in 400 mL solvent (ethanol/EtOAc (v/v)=1:2) and thoroughly stirred in the three-necked flask equipped with a reflux condenser at 65 °C for 0.5 h. A cold solution of 35% trimethylamine in ethanol (20 mL-4 eq) was then dropwise added in the reaction bath and kept in reflux for 48 h. After cooling to room temperature, the product (BTAEAzO) was precipitated. Finally, the precipitate of BTAEAzO was filtered, followed by recrystallized in 90:10 v/v solution of EtOAc/ethanol. ^1H NMR spectrum is shown in Figure S3: (400 MHz, MeOD) δ 7.86 (dd, $J = 14.6, 8.0$ Hz, 4H), 7.09 (dd, $J = 52.0, 9.0$ Hz, 4H), 4.62 – 4.56 (m, 2H), 4.05 (t, $J = 6.4$ Hz, 2H), 3.95 – 3.87 (m, 2H), 3.31 (s, 12H), 1.87 – 1.67 (m, 2H), 1.64 – 1.40 (m, 2H), 1.00 (t, $J = 7.4$ Hz, 3H).

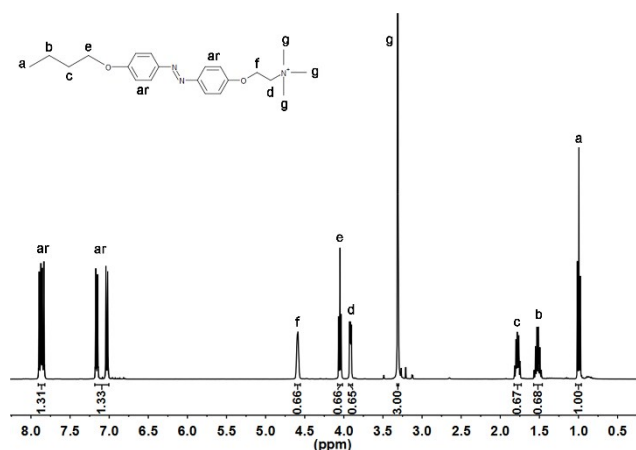


Figure S3. ^1H -NMR spectra of BTAEAzO.