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RAFT Based Synthesis of In-house Polymers

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Caroline Brown[1,2,3], Snehasish Ghosh[1,2,3], Kallol Gupta[1,2,3]

[1]Nanobiology Institute, Yale University, West Haven, CT, USA;
[2]Department of Cell Biology, Yale University School of Medicine, New Haven, CT, USA;
[3]Aligning Science Across Parkinson's (ASAP) Collaborative Research Network, Chevy Chase, MD, 20815

C Caroline Brown
Yale University

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We use this protocol and it's working

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Abstract

The protocol gives details for synthesizing the ChloroSMA series of membrane active polymers.

Chemicals and reagents

1 All reagents were purchased from Sigma-Aldrich and used without further purification unless specified
otherwise.

Synthesis of ChloroSMA20

2 To a 50 mL round bottom flask add [M] 1.52 millimolar (mM) chloro-styrene , [M] 1.52 millimolar (mM) maleic anhydride , and [M] 0.076 millimolar (mM) 2-(dodecylthiocarbonothioylthio) propionic acid .

3 Dissolve in dimethylformamide.

4 Add [M] 0.004 millimolar (mM) 1,1-Azobis(cyclohexanecarbonitrile) .

5 Cap the flask with a rubber stopper and bubble with nitrogen for 00:15:00 minutes. 15m

6 Heat to 90 °C for 16:00:00 hours. 16h

7 Precipitate in isopropanol or water, filter, and dry in vacuo.

Synthesis of ChloroSMA 40 16h 15m

8 To a 50 mL round bottom flask add [M] 3.1 millimolar (mM) chloro-styrene , [M] 3.1 millimolar (mM) maleic anhydride , and [M] 0.076 millimolar (mM) 2-(dodecylthiocarbonothioylthio) propionic acid .

9 Dissolve in dimethylformamide.

10 Add [M] 0.004 millimolar (mM) 1,1-Azobis(cyclohexanecarbonitrile) .

11 Cap the flask with a rubber stopper and bubble with nitrogen for 00:15:00 minutes. 15m

12 Heat to 90 °C for 16:00:00 hours. 16h

13 Precipitate in isopropanol or water, filter, and dry in vacuo.

Synthesis of ChloroSMA 60 16h 15m

14 To a 50 mL round bottom flask add [M] 4.56 millimolar (mM) chloro-styrene , [M] 4.56 millimolar (mM) maleic anhydride , and [M] 0.076 millimolar (mM) 2-(dodecylthiocarbonothioylthio) propionic acid .

15 Dissolve in dimethylformamide.

16 Add [M] 0.004 millimolar (mM) 1,1-Azobis(cyclohexanecarbonitrile) .

17 Cap the flask with a rubber stopper and bubble with nitrogen for 00:15:00 minutes. 15m

18 Heat to 90 °C for 16:00:00 hours. 16h

19 Precipitate in isopropanol or water, filter, and dry in vacuo.

Synthesis of ChloroSMA 80 16h 15m

20 To a 50 mL round bottom flask add [M] 6.1 millimolar (mM) chloro-styrene , [M] 6.1 millimolar (mM) maleic anhydride , and

[M] 0.076 millimolar (mM) 2-(dodecylthiocarbonothioylthio) propionic acid .

21 Dissolve in dimethylformamide.

22 Add [M] 0.004 millimolar (mM) 1,1-Azobis(cyclohexanecarbonitrile) .

23 Cap the flask with a rubber stopper and bubble with nitrogen for 00:15:00 minutes. 15m

24 Heat to 90 °C for 16:00:00 hours. 16h

25 Precipitate in isopropanol or water, filter, and dry in vacuo.

End-group removal of poly(4 chlorostyrene-alt-maleic anhydride 6h 15m

26 Dissolve precipitated polymer in dimethylformamide and add [M] 9.9 millimolar (mM) benzoyl peroxide to a round bottom flask.

27 Seal with a rubber stopper and bubble with nitrogen for 00:15:00 minutes. 15m

28 Leave the escape needle in the flask and heat to 90 °C for 06:00:00 hours. 6h

29 Precipitate polymer with isopropanol or water and dry in vacuo.

Hydrolysis to produce poly(4 chlorostyrene-alt-maleic acid) 1d 16h

30 In a round bottom flask dissolve 800 mg anhydride polymer in 2 g KOH and 20 mL water .

31 Reflux for 04:00:00 hours. The mixture should become clear. 4h

32 Dialyze the hydrolyzed polymer using a 3.5KDa mwco membrane for 36:00:00 hours. 1d 12h

33 Add water to precipitate the polymer.

34 Wash polymer using 0.1N HCl, 3 times, and dry the polymer under nitrogen.