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RAFT Based Synthesis of In-house Polymers

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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  1.52 millimolar (mM) chloro-styrene ,  1.52 millimolar (mM) maleic anhydride , and  0.076 millimolar (mM) 2-(dodecylthiocarbonothioylthio) propionic acid .
- 3 Dissolve in dimethylformamide.
- 4 Add  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  3.1 millimolar (mM) chloro-styrene ,  3.1 millimolar (mM) maleic anhydride , and  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.

36:00:00

1d 12h

33 Add water to precipitate the polymer.

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