Synthesis of alkyl polyglycosides using SO₃H-functionalized ionic liquids as catalysts

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1. ¹H NMR spectra were recorded on a Bruker spectrometer (600 Hz) in deuterium oxide (D_2O). The results of four SFILs are as follows:

[PSmim]HSO₄:

¹H NMR(600 MHz, D₂O): δ ppm= 2.29(m, 2H), 2.89(t, 2H), 3.86(s, 3H), 4.32(t, 2H),

7.41(s, 1H), 7.48(s, 1H), 8.70(s, 1H)

[PSmim][pTSA]:

¹H NMR(600 MHz, D₂O): δ ppm= 2.30(m, 2H), 2.37(s, 3H), 2.89(t, 2H), 3.85(s, 3H),

4.31(t, 2H), 7.34(d, 2H), 7.40(s, 1H), 7.47(s, 1H), 7.67(d, 2H), 8.68(s, 1H)

[PSPy]HSO₄:

¹H NMR(600 MHz, D₂O): δ ppm= 2.36(m, 2H), 2.86(t, 2H), 4.65(t, 2H), 8.00(s, 2H),

8.47(s, 1H), 8.76(d, 2H)

[PSPy][pTSA]:

¹H NMR(600 MHz, D₂O): δ ppm= 2.41(s, 3H), 2.49(m, 2H), 2.99(t, 2H), 7.37(d, 2H), 7.70(d, 2H), 8.10(t, 2H), 8.57(t, 1H), 8.90(d, 2H)

2. The Fourier transform infrared-spectra (FTIR) were obtained in the range of 400-4000 cm⁻¹ on a Thermo Nicolet 380 spectrometer. The results of four SFILs are as follows:



Figure S1 The FT-IR spectrum of four SFILs

3. Electrospray ionization mass spectrometry (ESI-MS) were obtained in both positive and negative modes with a Bruker micrOTOF-Q II. [PSmim]HSO₄

The molecular weight (MW) of [PSmim]⁺ theoretical value is 205.2438, cationic mode detection value is 205.0710, HSO₄⁻ theoretical value is 97.0705, anion mode detection value is 96.9614, indicating that the MW difference of the theoretical value and the actual detection value of [PSmim]HSO₄ is very small. It proved that the synthesized ionic liquid (IL) is the target product, and the results of ESI-MS are presented on Figure S2 and S3.



Figure S2 The ESI-MS cationic model of [PSmim]HSO4



Figure S3 The ESI-MS anionic model of [PSmim]HSO₄

[PSmim][pTSA]

The MW of [PSmim]⁺ theoretical value is 205.2438, cationic mode detection value is 205.0638, [pTSA]⁻ theoretical value is 171.1888, anion mode detection value is 171.0172, indicating that the MW difference of the theoretical value and the actual detection value of [PSmim][pTSA] is very small. It proved that the synthesized IL is the target product, and the results of ESI-MS are presented on Figure S4 and S5.



Figure S4 The ESI-MS cationic model of [PSmim][pTSA]



Figure S5 The ESI-MS anionic model of [PSmim][pTSA]

[PSPy]HSO₄

The MW of $[PSPy]^+$ theoretical value is 202.2422, cationic mode detection value is 202.0566, HSO_4^- theoretical value is 97.0705, anion mode detection value is 96.9623, indicating that the MW difference of the theoretical value and the actual detection value of $[PSPy]HSO_4$ is very small. It proved that the synthesized IL is the target product, and the results of ESI-MS are presented on Figure S6 and S7.







Figure S7 The ESI-MS anionic model of [PSPy]HSO4

[PSPy][pTSA]

The MW of [PSPy]⁺theoretical value is 202.2422, cationic mode detection value is 202.0537, [pTSA]⁻ theoretical value is 171.1888, anion mode detection value is 171.0119, indicating that the MW difference of the theoretical value and the actual detection value of [PSPy][pTSA] is very small. It proved that the synthesized IL is the target product, and the results of ESI-MS are presented on Figure S8 and S9.



Figure S8 The ESI-MS cationic model of [PSPy][pTSA]



Figure S9 The ESI-MS anionic model of [PSPy][pTSA]

4. The decomposition temperature of four SFILs are measured by thermal gravimetric analyzer (TGA Q500 V3.15 Build 263) by heating samples from 298.15 K to 873.15 K at a heating rate 10 K/min with N_2 atmosphere at a flow of 25 mL/min. The TGA results exhibit the decomposition temperature are over 523 K of four SFILs, indicating four SFILs have well thermal stability, and the TGA results are displayed on Figure S10.



Figure S10 The thermogravimetric trace of four SFILs