Novel Hydrolytically Stable Lewis Acidic Ionic Liquid Catalyst system For Polybutylene Succinate (PBS) Synthesis

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General protocol for the synthesis of PBS:

Procedure 1:

In a clean three neck RB flask fitted with distillation assembly, Succinic acid (20 g, 0.17 mol) and butane diol (16 g, 0.18 mol) was added followed by a catalyst (100-300 ppm) and the resultant mixture was heated to 175 °C till the completion of the esterification reaction (water collected 5.6 mL). After completion of the esterification reaction, polycondensation was carried out at 180-230 °C under vacuum to give the corresponding polymer (Refer table 1). After completion of the polycondensation reaction, reaction mixture was poured into water and the polymer thus obtained was filtered, washed with acetone and dried under nitrogen to afford the corresponding polymer in good yield (92%). IR (neat):1714, 1157 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.71 (m, 4H), 1.71 (m, 4H), 1.71 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.3, 64.2, 29.0, 25.2.

Procedure 2:

In a clean three neck RB flask fitted with distillation assembly, Succinic acid (20 g, 0.17 mol) and butane diol (16 g, 0.18 mol) was added and the resultant mixture was heated to 175 °C till the completion of the esterification reaction (water collected 5.6 mL). After completion of the esterification reaction, catalyst (200 ppm) was added and the polycondensation was carried out at 200 - 230 °C under vacuum to give the corresponding polymer (Refer table 2, Entry 9 – 11). After completion of the polycondensation reaction, reaction mixture was poured into water and the polymer thus obtained was filtered, washed with acetone and dried under nitrogen to afford the corresponding polymer in good yield (91%).

General procedure for the preparation of Task specific Lewis acidic ionic liquids:

Lewis Acidic Ionic Liquid	Ratio	BMIMCl (g)	$ZnCl_2$ (g)	$SnCl_2$ (g)
BMIMCl:$ZnCl_2$	(1:1)	5	3.9	
	(1:2)		7.8	
BMIMCL:$SnCl_2$	(1:1)	5		5.43
	(1:2)			10.86
ChCl:$ZnCl_2$	(1:1)	5	4.88	

	(1:2)		9.76	
$ChCl:SnCl_2$	(1:1)	5		6.79
	(1:2)			13.58
	(1:3)			20.372

In a clean two-neck RB flask, corresponding quaternary ammonium salt and metal chloride in required quantity was taken and the resultant mixture was heated to 130 °C and stirred at that temperature for 3 h until the colorless solution is obtained. Reaction mixture was then cooled to room temperature and catalyst thus obtained was used for PBS synthesis.