Supporting Information

Fe(III)–Mediated ICAR ATRP in *p*-Xylene/PEG-200 Biphasic System: Facile and Highly Efficient Separation and Recycling of Iron Catalyst

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Table S1. Recycling experiments for Fe(III)-mediated TPSC-based ICAR ATRP of MMA.

Entry	Recycling times[a]	Monomer conversion (%)	$M_{n,th}$[b] (g/mol)	$M_{n,GPC}$ (g/mol)	M_w/M_n
1	1	23.5	5000	9000	1.27
2	2	73.8	15000	16500	1.21
3	3	82	16600	16000	1.28
4	4	80.2	16300	17300	1.19
5	5	84.1	17000	16800	1.28
6	6	81.6	16600	16800	1.30
7	7	77.8	15900	17700	1.34
8	8	83.9	17000	16500	1.45
9	9	83.6	17000	18000	1.39
10	10	80.2	16300	20100	1.45

Polymerization conditions: $[monomer]_0:[EBPA]_0:[FeCl_3{\cdot}6H_2O]_0:[TBABr]_0:[AIBN]_0$ = 200:1:1:3:1, $V_{monomer}$ = 1.0 mL, $V_{p\text{-xylene}}$ = 2.0 mL and $V_{PEG\text{-}200}$ = 1.0 mL, temperature = 70 °C. [a]Polymerization was conducted using the iron catalyst solution in PEG-200 after every recycling experiment without adding any fresh $FeCl_3{\cdot}6H_2O$ and TBABr. [b]$M_{n,th} = ([M]_0/[EBPA]_0) \times M_{w,MMA} \times$ conv.%.

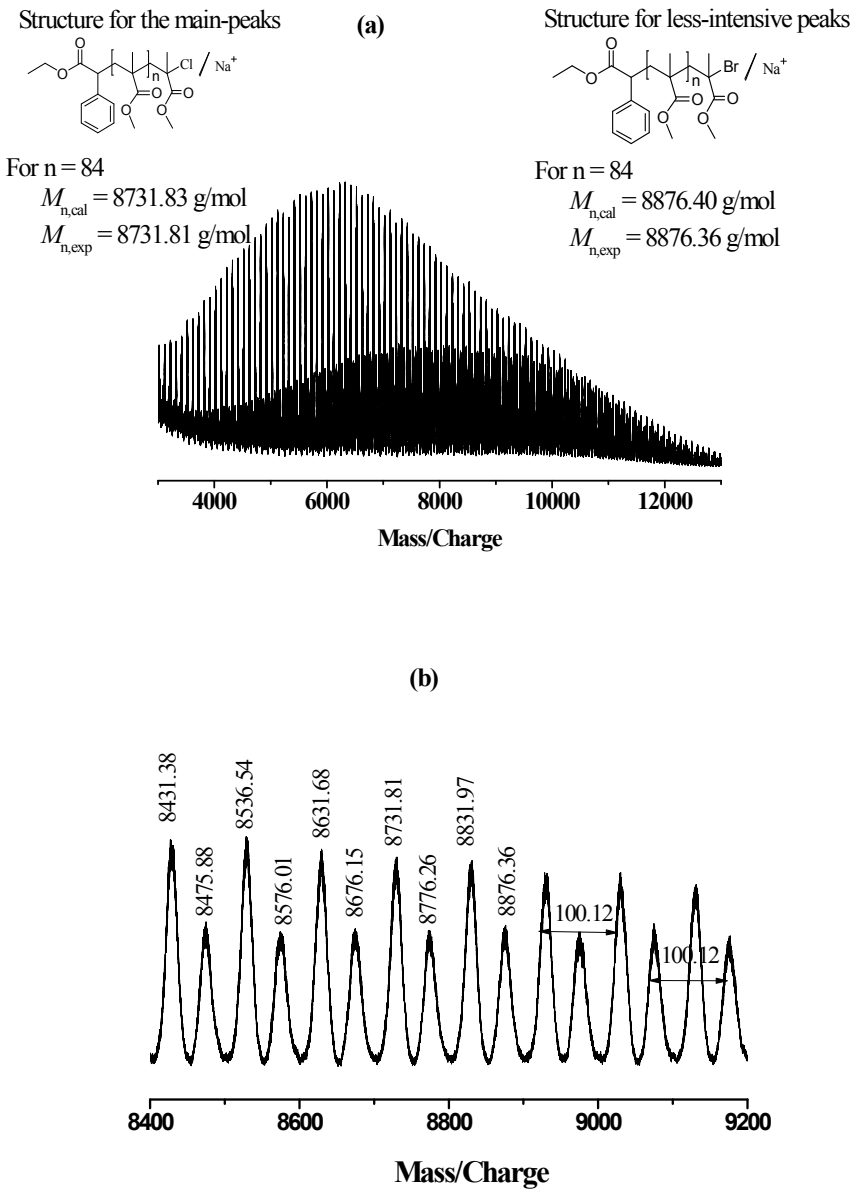

Figure S1. MALDL-TOF-MS in the linear mode of PMMA-Br and PMMA-Cl ($M_{n,GPC}$ = 8700 g/mol, M_w/M_n = 1.25) (a) and enlargement of the MALDL-TOF-MS from m/z 8400 to 9200 of PMMA-Br and PMMA-Cl (b).

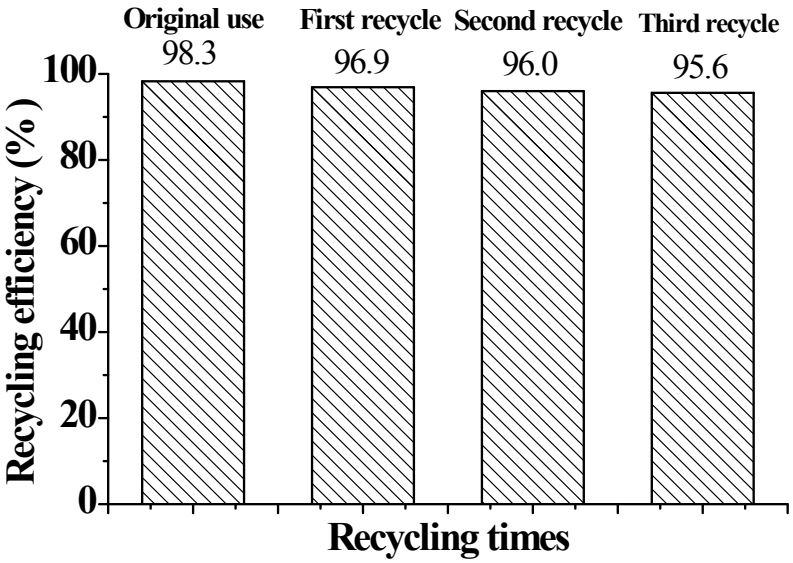

Figure S2. Recycling efficiency of iron catalyst at different recycle times. Polymerization conditions : $[MMA]_0$:$[EBPA]_0$:$[FeCl_3{\cdot}6H_2O]_0$:$[TBABr]_0$: $[AIBN]_0$ = 200:1:1:3:1, V_{MMA} = 1.0 mL, $V_{p\text{-xylene}}$ = 2.0 mL and $V_{PEG\text{-}200}$ = 1.0 mL, temperature = 70 °C. Recycling efficiency is the percentage of residual iron catalyst in PEG-200 phase to the original iron catalyst, which was determined by inductively coupled plasma (ICP) of Vista MPX.