

## Supporting Information

### **CO<sub>2</sub>-Switchable Pickering Emulsion: Efficient and Tunable Interfacial Catalysis for Alcohol Oxidation in Biphasic System**

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## I. Experimental Section

### Materials

2-(dimethylamino) ethyl methacrylate (DMA), methyl methacrylate (MMA), 2,2,6,6-tetramethyl-4-piperidinylmethacrylate (TMPM), azodiisobutyronitrile (AIBN),  $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$ , ethylenediaminetetraacetic acid (EDTA) and  $\text{H}_2\text{O}_2$  were received from Sigma Aldrich. 4-Cyan-4-(dodecylsulfanylthiocarbonyl) sulfanylpentanoic acid (DTTCP) was bought from Chengdu Syns Medical Technology Co. Ltd. 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO), 1,4-dioxane, petroleum ether, toluene, ethyl acetate,  $\text{CH}_2\text{Cl}_2$ , THF, NaClO, NaBr, benzyl alcohol and other substrates were all from Aladdin Co. Ltd. All chemicals were analytical grade and used without further purification.

### Preparation of P((TMPM-*co*-DMA)-*b*-MMA) and P(TMPM-*b*-MMA)

TMPM, DMA, DTTCP, AIBN and 1,4-dioxane were removed into a flask. The sealed reactor was degassed by  $\text{N}_2$  for 0.5 h. The reaction was then proceeded for 15 h (70 °C). Next the mixture of MMA, AIBN and 1,4-dioxane, which have been removed air, was injected into reactor and reacted for 24 h. After finished, the reaction solution was slowly dropped into petroleum ether to precipitate product and repeated 3 times. After dried under vacuum at 60 °C overnight, yellow powder was obtained. Details of the experiment were shown in Table S1. P(TMA-*b*-MMA) was prepared without DMA feeding.

### Preparation of P((TMA-*co*-DMA)-*b*-MMA) and P(TMA-*b*-MMA)

P(TMPM-*b*-MMA)/P((TMPM-*co*-DMA)-*b*-MMA),  $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$ , EDTA and THF were removed into a flask and stirring at 60 °C for 1 h.  $\text{H}_2\text{O}_2$  was added into the mixture in batches, and the color of the reaction solution gradually changed from light yellow to red. After 48 h, the reaction liquid was dropped into petroleum ether. The polymers

were treated by water and dried in vacuum to receive light red powder. Details of the experiment were shown in Table S2.

### **Characterization**

<sup>1</sup>H NMR spectra were measured on Bruker Avance III spectrometer (CDCl<sub>3</sub>-d<sub>6</sub>). FTIR spectra were obtained by Bruker Tensor II FTIR spectrometer. The element content (C, H, N, S) of samples were received by Vario Macro cube elemental analyzer. The surface wettability of samples were measured by DataPhysics OCA-20. The particle sizes and zeta potentials of samples were measured by Malvern ZS90 digital light scattering (DLS). The morphology of polymer-based catalysts were visualized by JEM-1010 transmission electron microscope (80 kV), and uranyl acetate solution as colorant. Pickering emulsions stabilized by polymer-based interfacial catalysts were observed by Olympus BX41 optical microscope.

### **CO<sub>2</sub> response for demulsification**

The stimulus responsiveness of P(TMA-*b*-MMA) and P((TMA-*co*-DMA)-*b*-MMA)-*n* (*n* = 1 - 5) (4 mg) were conducted in the water-oil biphasic system. The samples were added into the bottles with organic solvent (3.5 mL), and ultrasonic treated at room temperature for 2 min. Then equal volume of water was moved into dispersion to afford milky white Pickering emulsion after homogenization 2 min. At room temperature, CO<sub>2</sub> flow (15 mL·min<sup>-1</sup>) was introduced into system for certain time until thorough demulsification. Under the same conditions, CO<sub>2</sub> was removed by N<sub>2</sub> bubbling (15 mL·min<sup>-1</sup>). After vigorous stirring, the emulsification of biphasic system was realized again.

### **Catalytic performance**

In a typical experiment, polymer-based catalyst (0.01mmol TEMPO), substrate (1 mmol) and CH<sub>2</sub>Cl<sub>2</sub> were added into a 25 mL vial and stirred for 1 min to make the

catalyst well dispersed. Then NaBr (0.15 mL, 0.15 mmol) and NaClO (3.35 mL, 1.5 mmol, pH  $\approx$  9.1) aqueous solution were charged into reactor successively. The reaction was stirred at 1400 rpm. In addition, the mixtures of oil and water were homogenized for 3 s (1400 rpm) to estimate the activity under static Pickering emulsion. After reacted for period, the mixture was extracted and removed to a tube containing excessive anhydrous sodium sulfite. The conversion and selectivity of oxidation reaction were detected by Shimadzu GC-2014C (Rtx-5, 30 m $\times$ 0.25 mm $\times$ 0.25 mm). The emulsions were bubbled with CO<sub>2</sub> to demulsify. After deprotonating treatment, oil and aqueous phase were removed and the recycled polymeric interfacial catalysts were directly added into new reaction medium for next cycle.

## II. Supplementary Fig. and tables

4-Cyan-4-(dodecylsulfanylthiocarbonyl) sulfanylpentanoic acid (DTTCP):  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta = 3.37 - 3.26$  (m, 2H),  $2.75 - 2.62$  (m, 2H),  $2.60 - 2.31$  (m, 2H),  $1.84$  (d,  $J=42.3$ , 3H),  $1.69$  (dt,  $J=15.1, 7.5$ , 2H),  $1.46 - 1.33$  (m, 2H),  $1.35 - 1.18$  (m, 16H),  $0.95 - 0.81$  (m, 3H).

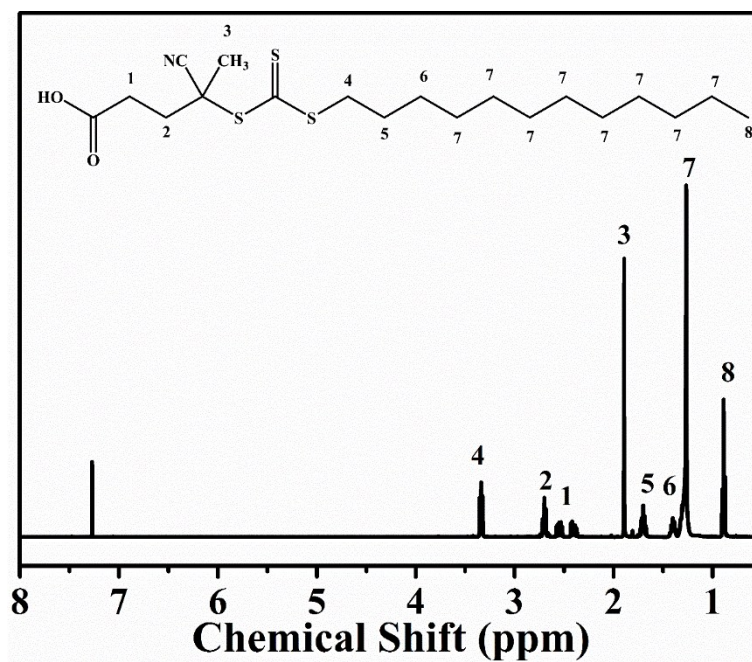
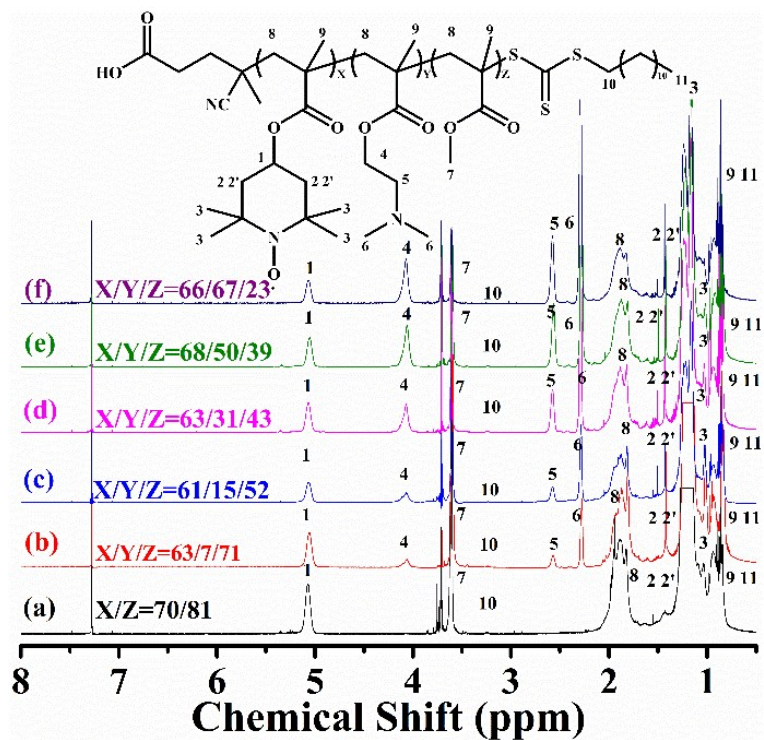
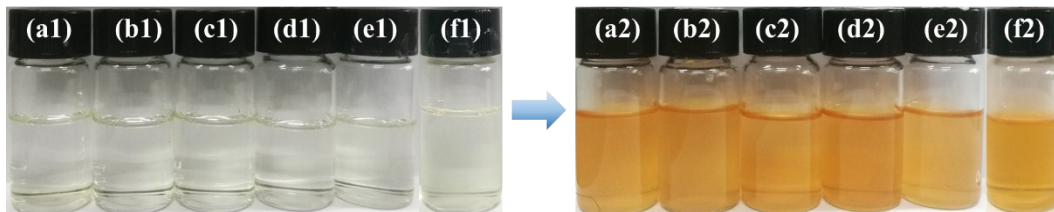


Fig. S1  $^1\text{H}$  NMR spectrum of DTTCP

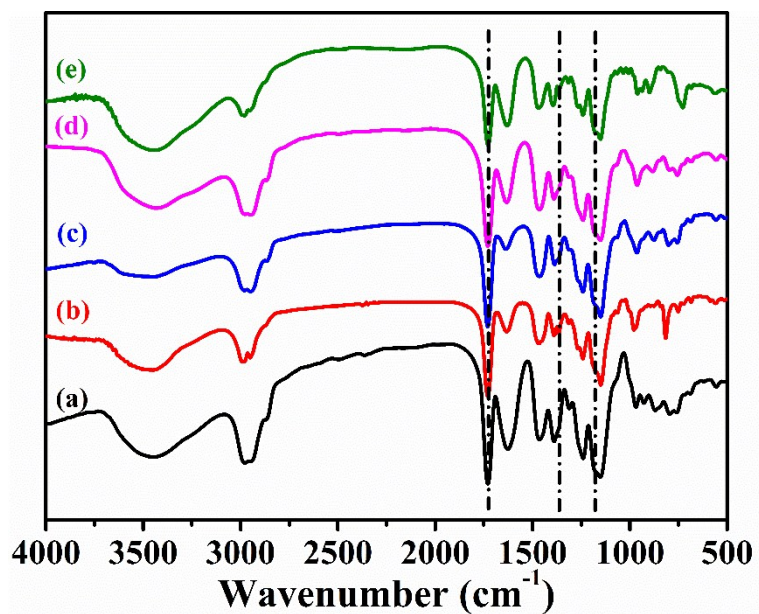




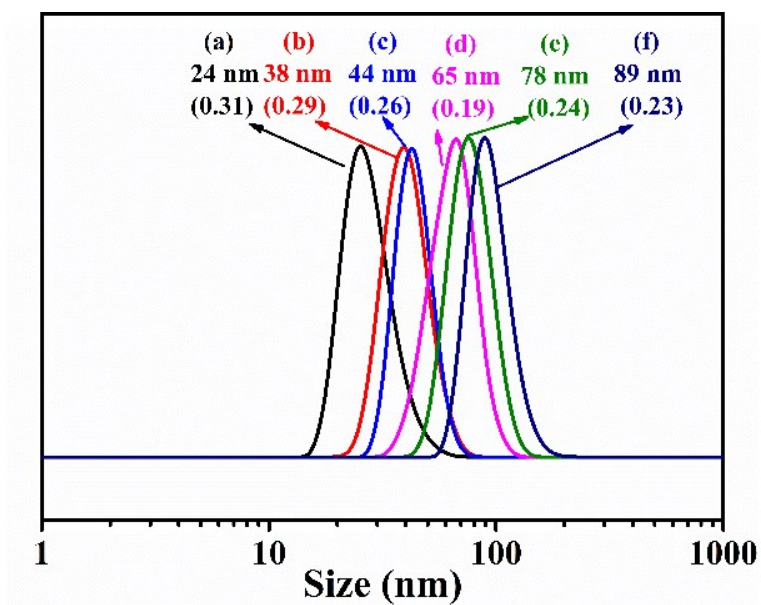
**Fig. S2**  $^1\text{H}$  NMR spectra of (a) P(TMA-*b*-MMA), (b) P((TMA-*co*-DMA)-*b*-MMA)-1, (b) P((TMA-*co*-DMA)-*b*-MMA)-2, (c) P((TMA-*co*-DMA)-*b*-MMA)-2, (d) P((TMA-*co*-DMA)-*b*-MMA)-3, (e) P((TMA-*co*-DMA)-*b*-MMA)-4 and (f) P((TMA-*co*-DMA)-*b*-MMA)-5



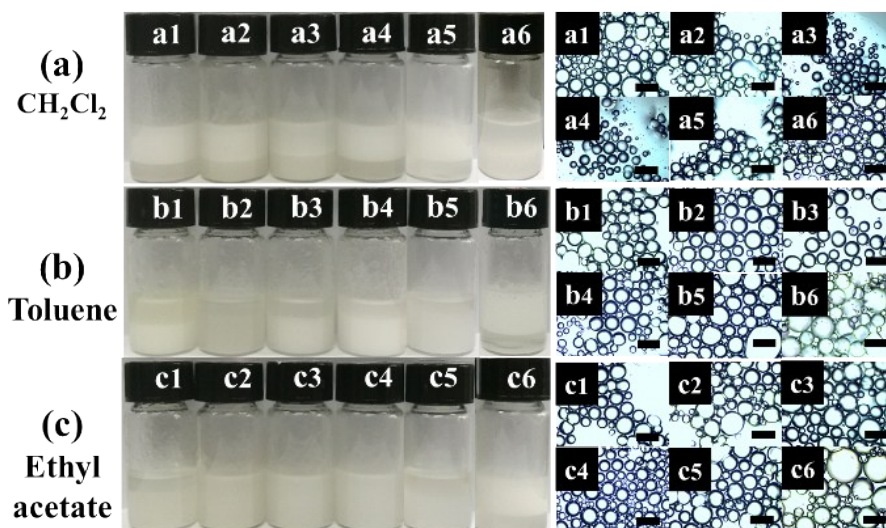
**Fig. S3** Digital photos of (a1) P(TMPM-*b*-MMA), (b1) P((TMPM-*co*-DMA)-*b*-MMA)-1, (c1) P((TMPM-*co*-DMA)-*b*-MMA)-2, (d1) P((TMPM-*co*-DMA)-*b*-MMA)-3, (e1) P((TMPM-*co*-DMA)-*b*-MMA)-4 and (f1) P((TMPM-*co*-DMA)-*b*-MMA)-5; (a2) P(TMA-*b*-MMA), (b2) P((TMA-*co*-DMA)-*b*-MMA)-1, (c2) P((TMA-*co*-DMA)-*b*-MMA)-2, (d2) P((TMA-*co*-DMA)-*b*-MMA)-3, (e2) P((TMA-*co*-DMA)-*b*-MMA)-4 and (f2) P((TMA-*co*-DMA)-*b*-MMA)-5 dispersed in THF



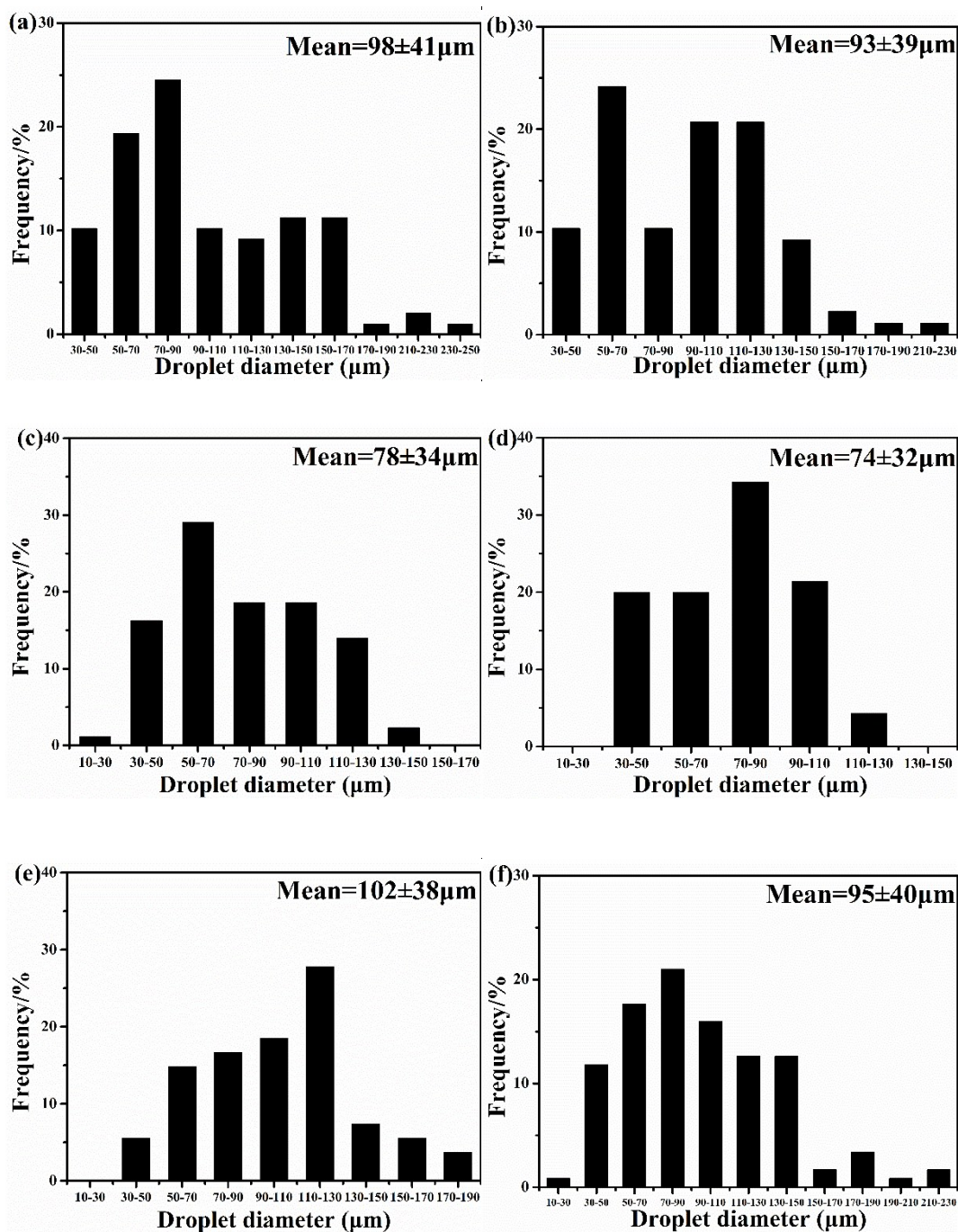
**Fig. S4** FTIR spectra of (a) P(TMA-*b*-MMA), (b) P((TMA-*co*-DMA)-*b*-MMA)-1, (c) P((TMA-*co*-DMA)-*b*-MMA)-2, (d) P((TMA-*co*-DMA)-*b*-MMA)-3 and (e) P((TMA-*co*-DMA)-*b*-MMA)-5



**Fig. S5** DLS results of (a) P(TMA-*b*-MMA), (b) P((TMA-*co*-DMA)-*b*-MMA)-1, (c) P((TMA-*co*-DMA)-*b*-MMA)-2, (d) P((TMA-*co*-DMA)-*b*-MMA)-3, (e) P((TMA-*co*-DMA)-*b*-MMA)-4 and (f) P((TMA-*co*-DMA)-*b*-MMA)-5

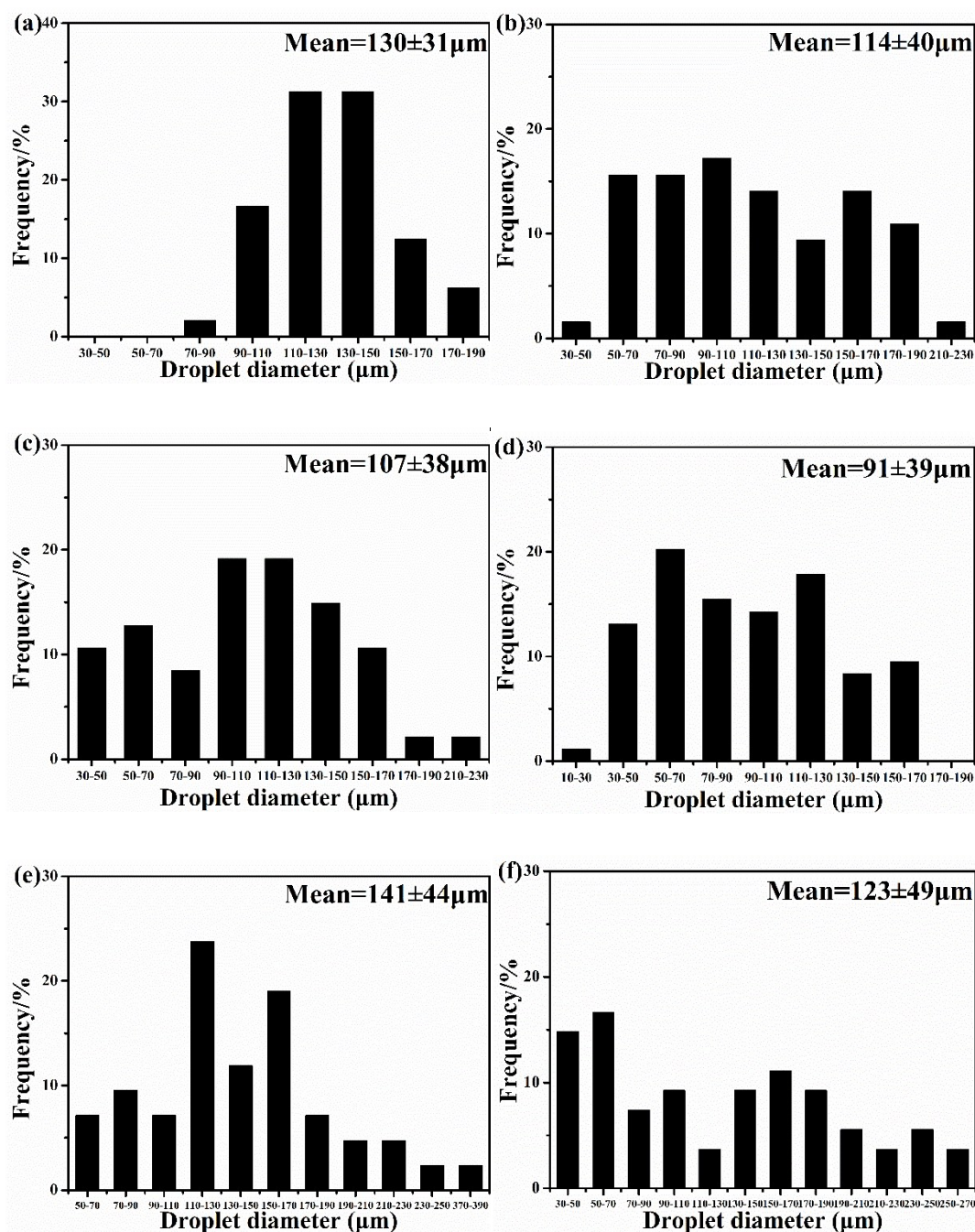


**Fig. S6** Pickering emulsion stabilized by P(TMA-*b*-MMA) and P((TMA-*co*-DMA)-*b*-MMA)-*n* (*n* = 1 - 5) with (a) CH<sub>2</sub>Cl<sub>2</sub>, (b) toluene and (c) ethyl acetate as oil phase (standing for 30 min, scale bar: 200 μm).



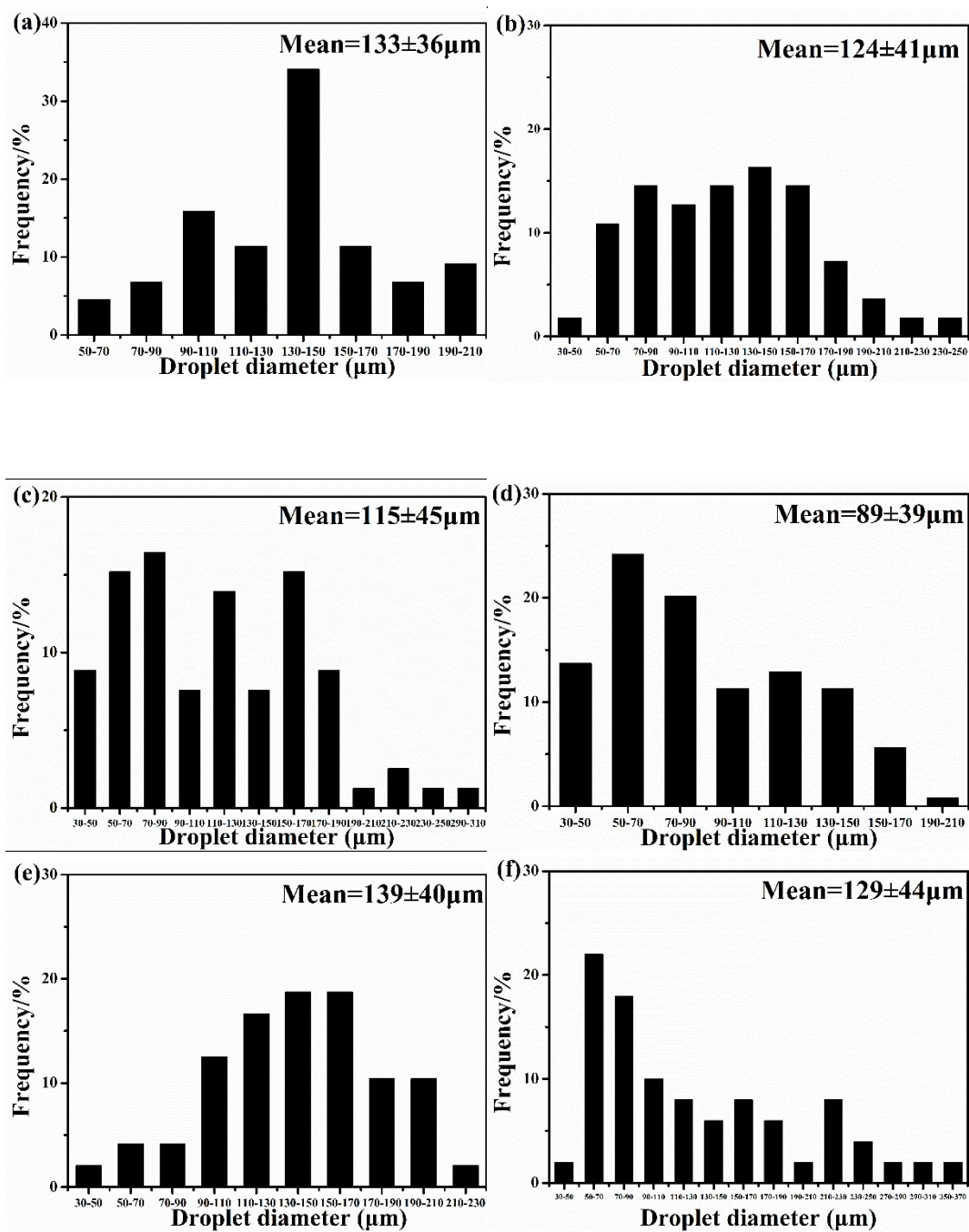
**Fig. S7** Statistical droplet diameters of Pickering emulsions stabilized by (a) P(TMA-*b*-MMA), (b) P((TMA-*co*-DMA)-*b*-MMA)-1, (c) P((TMA-*co*-DMA)-*b*-MMA)-2, (d) P((TMA-*co*-DMA)-*b*-MMA)-3, (e) P((TMA-*co*-DMA)-*b*-MMA)-4 and (f) P((TMA-

*co*-DMA)-*b*-MMA)-5. (Conditions: water 3.5 mL, CH<sub>2</sub>Cl<sub>2</sub> 3.5 mL, polymer 4 mg, standing for 30 min)



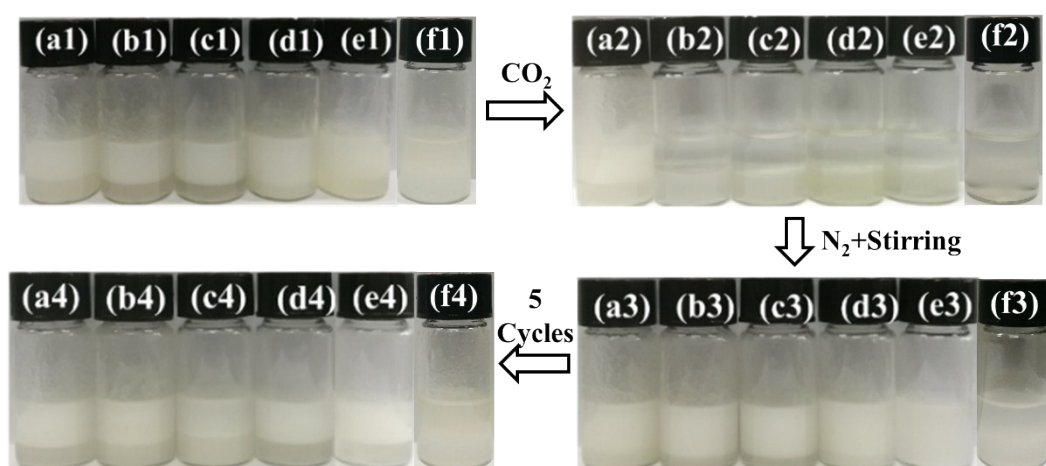
**Fig. S8** Statistical droplet diameters of Pickering emulsions stabilized by (a) P(TMA-*b*-MMA), (b) P((TMA-*co*-DMA)-*b*-MMA)-1, (c) P((TMA-*co*-DMA)-*b*-MMA)-2, (d) P((TMA-*co*-DMA)-*b*-MMA)-3, (e) P((TMA-*co*-DMA)-*b*-MMA)-4 and (f) P((TMA-*co*-DMA)-*b*-MMA)-5

*co*-DMA)-*b*-MMA)-5. (Conditions: water 3.5 mL, toluene 3.5 mL, polymer 4 mg, standing for 30 min)



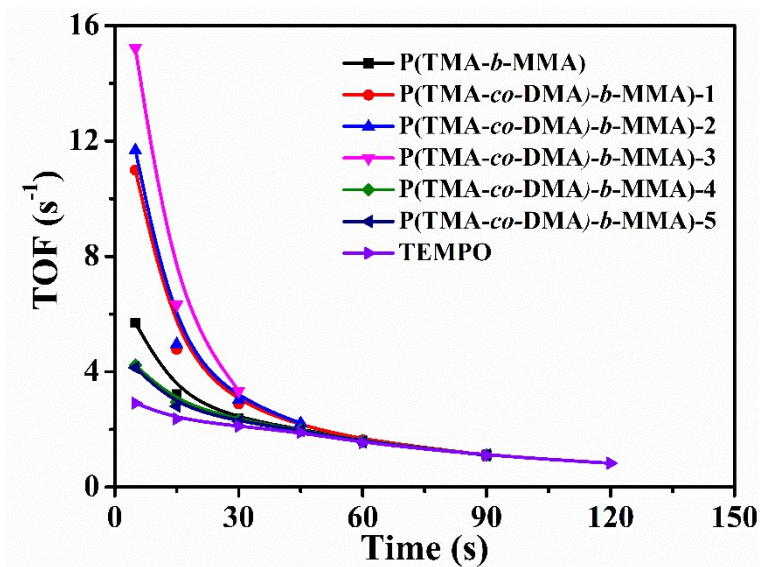


**Fig. S9** Statistical droplet diameters of Pickering emulsions stabilized by (a) P(TMA-*b*-MMA), (b) P((TMA-*co*-DMA)-*b*-MMA)-1, (c) P((TMA-*co*-DMA)-*b*-MMA)-2, (d) P((TMA-*co*-DMA)-*b*-MMA)-3, (e) P((TMA-*co*-DMA)-*b*-MMA)-4 and (f) P((TMA-*co*-DMA)-*b*-MMA)-5. (Conditions: water 3.5 mL, ethyl acetate 3.5 mL, polymer 4 mg, standing for 30 min)

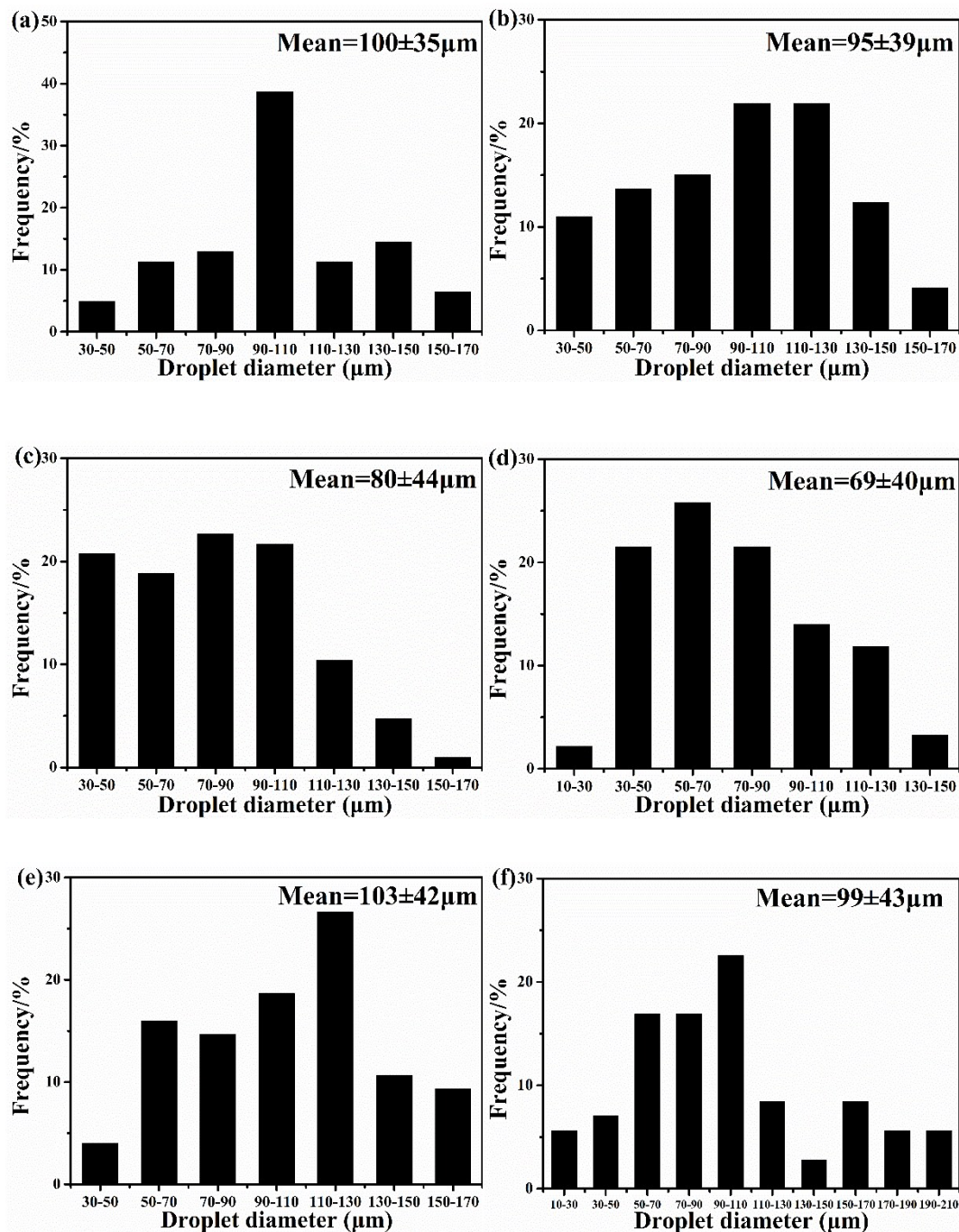


**Fig. S10** The CO<sub>2</sub> responsiveness of Pickering emulsion stabilized by (a) P(TMA-*b*-MMA), (b) P((TMA-*co*-DMA)-*b*-MMA)-1, (c) P((TMA-*co*-DMA)-*b*-MMA)-2, (d) P((TMA-*co*-DMA)-*b*-MMA)-3, (e) P((TMA-*co*-DMA)-*b*-MMA)-4 and (f) P((TMA-*co*-DMA)-*b*-MMA)-5



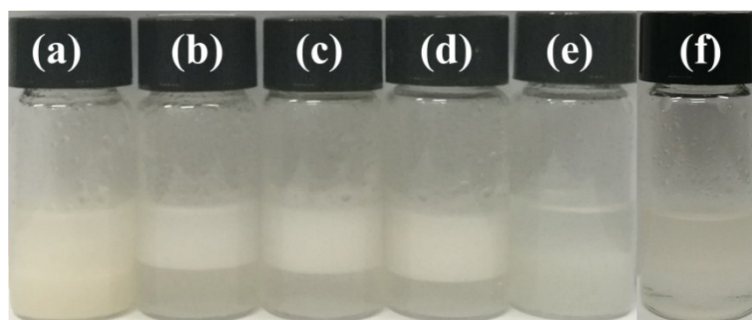


**Fig. S11** The TOF value of P(TMA-*b*-MMA), P((TMA-*co*-DMA)-*b*-MMA)-*n* (*n* = 1 - 5) and TEMPO

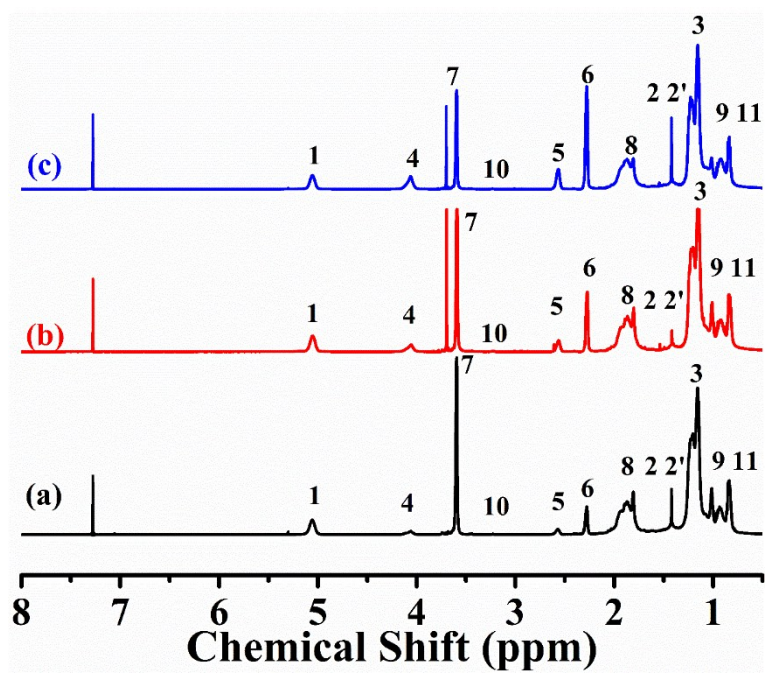


**Fig. S12** Statistical droplet diameters of Pickering emulsions stabilized by (a) P(TMA-*b*-MMA), (b) P((TMA-*co*-DMA)-*b*-MMA)-1, (c) P((TMA-*co*-DMA)-*b*-MMA)-2, (d) P((TMA-*co*-DMA)-*b*-MMA)-3, (e) P((TMA-*co*-DMA)-*b*-MMA)-4 and (e) P((TMA-*co*-DMA)-*b*-MMA)-5. (Conditions: NaClO 3.35 mL, NaBr 0.15 mL, CH<sub>2</sub>Cl<sub>2</sub> 3.5 mL,

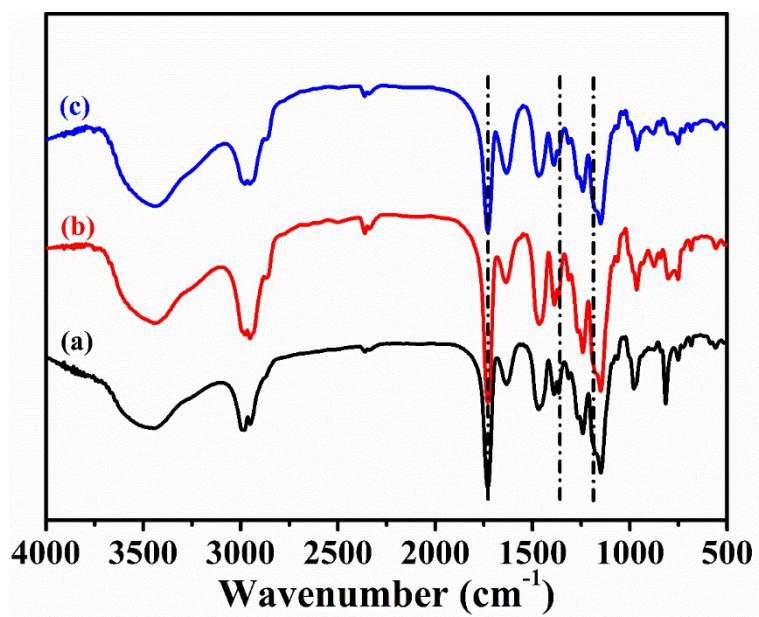
polymer-based catalyst 0.01mmol TEMPO, standing for 30 min)



**Fig. S13** Pickering emulsions stabilized by (a) P(TMA-*b*-MMA), (b) P((TMA-*co*-DMA)-*b*-MMA)-1, (c) P((TMA-*co*-DMA)-*b*-MMA)-2, (d) P((TMA-*co*-DMA)-*b*-MMA)-3, (e) P((TMA-*co*-DMA)-*b*-MMA)-4 and (f) P((TMA-*co*-DMA)-*b*-MMA)-5 standing for one week at room temperature



**Fig. S14**  $^1\text{H}$  NMR spectra of (a) P((TMA-*co*-DMA)-*b*-MMA)-1, (b) P((TMA-*co*-DMA)-*b*-MMA)-2 and (c) P((TMA-*co*-DMA)-*b*-MMA)-3 recycled 5 times



**Fig. S15** FTIR spectra of (a) P((TMA-*co*-DMA)-*b*-MMA)-1, (b) P((TMA-*co*-DMA)-*b*-MMA)-2 and (c) P((TMA-*co*-DMA)-*b*-MMA)-3 after 5 recycling



**Table S1** Experiment details of preparing P(TMPM-*b*-MMA)/P((TMPM-*co*-DMA-*b*-MMA)-*n* (n = 1 - 5)

Entry	TMPM	DMA	MMA	RAFT reagent	AIBN	1,4-dioxane	Yield
1	7.2 g	0	4.8g	0.16 g	26 mg	30 mL	79%
2	7.2 g	0.63 g	4.4 g	0.16 g	26 mg	30 mL	73%
3	7.2 g	1.26 g	4.0 g	0.16 g	26 mg	30 mL	68%
4	7.2 g	2.51g	3.2 g	0.16 g	26 mg	30 mL	72%
5	7.2 g	3.71 g	2.4 g	0.16 g	26 mg	30 mL	81%

**Table S2** Experiment details of preparing P(TMA-*b*-MMA), P((TMA-*co*-DMA-*b*-MMA)-*n* (*n* = 1 - 5)

Entry	P(TMPM- <i>b</i> -MMA)/P((TMPM- <i>co</i> -DMA- <i>b</i> -MMA)	Na <sub>2</sub> WO <sub>4</sub> ·2H <sub>2</sub> O	EDTA	H <sub>2</sub> O <sub>2</sub>	THF	Yield
1	4 g	0.90 g	0.45 g	10 mL	60 mL	89%
2	4 g	0.90 g	0.45 g	10 mL	60 mL	91%
3	4 g	0.90g	0.45 g	10 mL	60 mL	88%
4	4 g	0.90g	0.45 g	10 mL	60 mL	90%
5	4 g	0.90g	0.45 g	10 mL	60 mL	92%

**Table S3.** Elemental analysis of P(TMPM-*b*-MMA), P(TMA-*b*-MMA), P((TMPM-*co*-DMA)-*b*-MMA)-*n* (*n* = 1 - 5) and P((TMA-*co*-DMA)-*b*-MMA)-*n* (*n* = 1 - 5)

Samples	Elements (wt%)				TEMPO	DMA
	C	N	H	S	loading (mmol·g <sup>-1</sup> )	loading (mmol·g <sup>-1</sup> )
P(TMPM- <i>b</i> -MMA)	66.01	4.09	9.571	0.396	-	-
P(TMA- <i>b</i> -MMA)	63.26	3.93	7.586	0.379	2.71	0
P((TMPM- <i>co</i> -DMA)- <i>b</i> -MMA)-1	65.80	4.36	9.407	0.421	-	-
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-1	63.18	4.19	7.244	0.404	2.70	0.26
P((TMPM- <i>co</i> -DMA)- <i>b</i> -MMA)-2	65.97	4.96	9.364	0.443	-	-
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-2	63.30	4.77	8.342	0.425	2.65	0.71
P((TMPM- <i>co</i> -DMA)- <i>b</i> -MMA)-3	65.75	5.59	9.553	0.404	-	-
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-3	63.23	5.38	8.262	0.389	2.59	1.22
P((TMPM- <i>co</i> -DMA)- <i>b</i> -MMA)-4	65.48	6.06	9.582	0.35	-	-
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-4	63.13	5.85	8.485	0.337	2.39	1.75
P((TMPM- <i>co</i> -DMA)- <i>b</i> -MMA)-5	65.31	6.67	9.496	0.34	-	-
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-5	63.09	6.45	8.154	0.329	2.27	2.29

$$T = \frac{\left(N_1 - \frac{S_1 \times 14}{3 \times 32}\right) - \left(N_2 - \frac{S_2 \times 14}{3 \times 32}\right)}{\left(N_2 - \frac{S_2 \times 14}{3 \times 32}\right) \times 15} \quad (\text{S1})$$

$$D = \frac{\left(N_2 - \frac{S_2 \times 14}{3 \times 32}\right)}{14 \times 100} - T \quad (\text{S2})$$

$T$  is the TEMPO loading of P(TMA-*b*-MMA) and P((TMA-*co*-DMA)-*b*-MMA)- $n$  ( $n = 1 - 5$ ).  $D$  is the DMA loading of P(TMA-*b*-MMA) and P((TMA-*co*-DMA)-*b*-MMA)- $n$  ( $n = 1 - 5$ ).  $N_1$  and  $S_1$  are the  $N$  and  $S$  content of P(TMPM-*b*-MMA) and P((TMPM-*co*-DMA)-*b*-MMA)- $n$  ( $n = 1 - 5$ ), respectively.  $N_2$  and  $S_2$  are the  $N$  and  $S$  content of P(TMA-*b*-MMA) and P((TMA-*co*-DMA)-*b*-MMA)- $n$  ( $n = 1 - 5$ ), respectively.

**Table S4.** The preparation of P(TMA-*b*-MMA)/P((TMA-*co*-DMA)-*b*-MMA)- $n$  ( $n = 1 - 5$ )

Samples	Feed ratio <sup>[a]</sup>	TEMPO loading (mmol·g <sup>-1</sup> ) <sup>[b]</sup>	DMA loading (mmol·g <sup>-1</sup> ) <sup>[b]</sup>
P(TMA- <i>b</i> -MMA)	80/0/120/1	2.76	0
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-1	80/10/110/1	2.65	0.29
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-2	80/20/100/1	2.69	0.66
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-3	80/40/80/1	2.55	1.25
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-4	80/60/60/1	2.38	1.75
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-5	80/80/40/1	2.26	2.30

[a] Feed molar ratio of TMA/DMA/MMA/DTTCP; [b] calculated from <sup>1</sup>H NMR.

**Table S5.** The zeta potentials of samples in water

Sample	Zeta potential (mV)	Zeta potential (mV)
	(before CO <sub>2</sub> bubbling)	(after CO <sub>2</sub> bubbling)
P(TMA- <i>b</i> -MMA)	6.91 ± 0.17	9.67 ± 0.31
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-1	7.49 ± 0.34	12.13 ± 0.30
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-2	7.73 ± 0.11	17.53 ± 0.04
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-3	8.10 ± 0.12	20.56 ± 0.15
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-4	8.14 ± 0.14	25.43 ± 0.08
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-5	8.41 ± 0.12	33.07 ± 0.07

**Table S6.** The comparison of this work with other system for oxidation

Entry	Catalyst	Substrate	Oxidant	Time	Temp.	Conv.	Select.	TOF	Ref.
1	[C <sub>12</sub> ] <sub>3</sub> [PW <sub>12</sub> O <sub>40</sub> ]	cyclooctene	H <sub>2</sub> O <sub>2</sub>	0.6 h	65 °C	>95%	>99%	283.2 h <sup>-1</sup>	1
2	PPO-TEMPO	benzyl alcohol	NaClO	3 min	0 °C	>99%	98%	0.56 s <sup>-1</sup>	2
3	H <sub>3</sub> [PW <sub>4</sub> O <sub>24</sub> ]	cyclooctene	H <sub>2</sub> O <sub>2</sub>	3 h	65 °C	61%	-	58.6 h <sup>-1</sup>	3
4	PS-P(MMA-b-TMA)	benzyl alcohol	NaClO	-	0 °C	>99%	>99%	0.17 s <sup>-1</sup>	4
5	TEMPO-CMPs	benzyl alcohol	O <sub>2</sub>	0.5 h	rt	99%	98%	40 h <sup>-1</sup>	5
6	[Au <sub>25-x</sub> (PET) <sub>18-x</sub> Ni] <sub>3</sub> /NH <sub>3</sub> /CeO <sub>2</sub>	benzyl alcohol	O <sub>2</sub>	16 h	80 °C	81%	89%	2.37 h <sup>-1</sup>	6
7	Pd	1,2-propanediol	O <sub>2</sub>	72 h	28 °C	-	-	4.92 h <sup>-1</sup>	7
8	iron(III) complexes	diphenylmethanol	tert-butylhydroperoxide	18 h	27 °C	99%	99%	5.45 h <sup>-1</sup>	8
9	Fe <sub>3</sub> O <sub>4</sub> @SiO <sub>2</sub> @P TMA	benzyl alcohol	NaClO	45 s	10 °C	99%	>99%	2.61 s <sup>-1</sup>	9
10	Fe <sub>3</sub> O <sub>4</sub> @SiO <sub>2</sub> @P (TMA-DEA)	benzyl alcohol	NaClO	30 s	10 °C	>99%	>99%	3.33 s <sup>-1</sup>	10
11	P((TMA-co-DMA)-b-MMA)	benzyl alcohol	NaClO	15 s	10 °C	95%	>99%	6.33 s <sup>-1</sup>	This work



**Table S7.** Catalytic activity of P((TMA-*co*-DMA)-*b*-MMA)-3 in Anelli oxidation system

Entry	Substrates	Time (s)	Conversion (%)	Selectivity (%)
1	1-phenylethanol	30	>99	>99
2	2-phenylethanol	15	>99	>99
3	diphenylmethanol	90	98	>99
4	4-nitrobenzyl alcohol	15	>99	>99
5	3-pyridinemethanol	15	>99	>99
6	cyclohexanol	15	>99	>99
7	1-nonanol	15	97	>99
8	1-hexanol	15	>99	>99
9	1-octanol	30	>99	>99
10	benzyl alcohol <sup>a</sup>	30	99	>99

[a] Catalyzed by P((TMA-*co*-DMA)-*b*-MMA)-3 after 5 cycles without stirring.





**Table S8.** Elemental analysis results of P((TMA-*co*-DMA)-*b*-MMA)-1, P((TMA-*co*-DMA)-*b*-MMA)-2 and P((TMA-*co*-DMA)-*b*-MMA)-3 recycled 5 times

Samples	Elements (wt%)			
	C	N	H	S
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-1 (fresh)	63.18	4.19	7.244	0.404
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-1 (recycled 5 times)	63.10	4.17	7.253	0.400
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-2 (fresh)	63.30	4.77	8.342	0.425
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-2 (recycled 5 times)	63.35	4.73	8.356	0.421
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-3 (fresh)	63.23	5.38	8.262	0.389
P((TMA- <i>co</i> -DMA)- <i>b</i> -MMA)-3 (recycled 5 times)	63.31	5.35	8.297	0.385



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