**Electronic Supplementary Information**

# Poly(fluoroacrylate)s with tunable surface hydrophobicity *via* radical copolymerization of 2,2,2-trifluoroethyl α-fluoroacrylate and 2-(trifluoromethyl)acrylic acid

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**Table S1** Experimental conditions and results of the conventional radical copolymerization of FATRIFE and MAF.[a]

| Entry | Initiator | FATRIFE (mol%) | | Temp. (°C ) | Time (h) | Yield (%) | $M_{n,NMR}^{c}$ (g mol$^{-1}$ ) |
|---|---|---|---|---|---|---|---|
| | | feed | copolymer[b] | | | | |
| P1 | TBPPi | 100 | 100 | 56 | 2 | 100 | 38,600 |
| P2 | TBPPi | 90 | 93 | 56 | 2 | 94 | 32,400 |
| P3 | TBPPi | 80 | 88 | 56 | 4 | 75 | 26,200 |
| P4 | TBPPi | 70 | 82 | 56 | 4 | 66 | 22,900 |
| P5 | TBPPi | 50 | 73 | 56 | 4 | 65 | 18,700 |
| P6 | TBPPi | 30 | 65 | 56 | 8 | 46 | 14,800 |
| P7 | TBPPi | 20 | 58 | 56 | 24 | 32 | 10,500 |
| P8 | PPFR | 70 | 84 | 80 | 16 | 71 | 63,600[c1] |

Acronyms: FATRIFE: 2,2,2-trifluoroethyl α-fluoroacrylate; MAF: 2-(trifluoromethyl)acrylic acid; TBPPi: *tert*-butyl peroxypivalate; PPFR: perfluoro-3-ethyl-2,4-dimethyl-3-pentyl. Solvent, AcN: acetonitrile. [a]Conditions: Solvent used = 2.0 mL;. $[\mathrm{TBPPi}]_0/([\mathrm{FATRIFE}]_0+[\mathrm{MAF}]_0) = 1$ mol%. [b]Copolymer compositions were determined by $^{19}$F NMR spectroscopy using equation (3). [c]Calculated by $^{1}$H NMR spectroscopy using equation (S3). [c1]Calculated by $^{19}$F NMR spectroscopy using equation (S6).

Because of the presence of the carboxylic acid functions borne by the copolymers, the molar masses (P1-P6, Table 1) could not be determined by SEC. However, attempts to estimate them were achieved by $^{1}$H NMR using equation (6). They were found to decrease (from 51,800 to 16,800 g mol$^{-1}$) when the MAF content in the monomer feed increased (P2-P7, Table 1). This is probably due to the inhibition effect of MAF that does not homopolymerize under radical conditions,[1, 2] leading to decreased conversion (94 to 32%). The average degrees of polymerization of FATRIFE and MAF in poly(FATRIFE-*co*-MAF) copolymers (P2-P6, Table 1), synthesized using *tert*-butyl peroxypivalate (TBPPi) as the initiator,[3] could be calculated by means of $^{1}$H NMR spectroscopy from equations (S1) and (S2), respectively, while their average molecular weights were assessed from eq. (S3).

$$DP_n\ of\ FATRIFE = \frac{\int_{4.1}^{5.1} CH_2 / 2}{\int_{0.9}^{1.5} CH_3 / 18} \qquad (S1)$$

$$DP_n\ of\ MAF = \frac{(\int_{2.45}^{3.35} CH_2 / 2) - (\int_{4.1}^{5.1} CH_2 / 2)}{\int_{0.9}^{1.5} CH_3 / 18} \qquad (S2)$$

$$M_{n,NMR} = 2M_{C(CH3)3} + (M_{FATRIFE} \times DP_n \text{ of FATRIFE}) + M_{MAF} \times (DP_n \text{ of MAF}) \quad (S3)$$

where $M_{C(CH3)3,}$ $M_{FATRIFE}$ and $M_{MAF}$ stand for the molar masses of $–C(CH_3)_3$ (57 g $mol^{-1}$), FATRIFE (172 g $mol^{-1}$) and MAF (140 g $mol^{-1}$), respectively.

In addition, for the poly(FATRIFE-*co*-MAF) copolymers synthesized using PPFR radical as the initiator[4, 5], average degrees of polymerization of FATRIFE and MAF were determined using $^{19}F$ NMR, from equations (S4) and (S5), respectively, while the average molecular weights were calculated from eq. (S6).

$$DP_n\ of\ FATRIFE = \frac{\int_{-73}^{-77} CF_3}{\int_{-65}^{-67} CF_3} \qquad (S4)$$

$$DP_n\ of\ MAF = \frac{\int_{-68}^{-70} CF_3}{\int_{-65}^{-67} CF_3} \qquad (S5)$$

$$M_{n,NMR} = 2M_{CF3} + (M_{FATRIFE} \times DP_n \text{ of FATRIFE}) + M_{MAF} \times (DP_n \text{ of MAF}) \quad (S6)$$

where $M_{CF3}$ stands for the molar masses of $-CF_3$ end-group (69 g mol$^{-1}$). This equation is based on the assumption that the termination of the fluorinated macroradicals occurred by recombination exclusively.[6-8]

**Table S2** Instantaneous Composition of the Copolymer during the Radical Copolymerization of FATRIFE with MAF initiated by *tert*-butyl peroxypivalate (TBPPi).[a]

| entry | FATRIFE mol% | | time (min) | yield (%) |
|---|---|---|---|---|
| | feed | copolymer[b] | | |
| P1 | 90 | 93 | 1 | 5.5 |
| P2 | 80 | 88 | 5 | 6.2 |
| P3 | 70 | 82 | 10 | 9.3 |
| P4 | 50 | 73 | 5 | 4.7 |
| P5 | 30 | 65 | 10 | 8.2 |
| P6 | 20 | 58 | 20 | 2.9 |

Acronyms: FATRIFE: 2,2,2-trifluoroethyl α-fluoroacrylate; MAF: 2-(trifluoromethyl)acrylic acid; TBPPi: *tert*-butyl peroxypivalate; Solvent, AcN: acetonitrile. [a]Conditions: Solvent used = 2.0 mL;. $[TBPPi]_0/[FATRIFE+MAF]_0 = 1$ mol%. Temperature: 56 °C. [b]Copolymer compositions were determined by $^{19}F$ NMR spectroscopy using equation 1.

## Determination of reactivity ratios of FATIRE and MAF by Fineman-Ross and Kelen-Tüdos Method

First the instantaneous copolymer compositions were obtained using $^{1}$H NMR spectroscopy (equations 1 and 2) from copolymerization experiments carried out at various FATRIFE/MAF monomer feeds and stopped at low (< 10 %) monomer conversions (data points are supplied in Table S2).

The following equation was used:

$$G = r_{FATRIFE}H - r_{MAF}$$

$$\text{where, } G = x(X\text{-}1) / X.$$

$$H = x^2/X.$$

where $x = f_{FATRIFE}/f_{MAF}$, and $f_{FATRIFE}$ and $f_{MAF}$ stand for the initial molar fractions of FATRIFE and MAF, respectively.

$X = F_{FATRIFE}/F_{MAF}$, where $F_{FATRIFE}$ and $F_{MAF}$ stand for the FATRIFE and MAF molar fractions, respectively, in the obtained copolymer.

Now,

$$\eta = (r_{FATRIFE} + r_{MAF}/\alpha)\zeta - r_{MAF}/\alpha$$

$$\text{where, } \eta = G/(\alpha + H)$$

$$\zeta = H/(\alpha + H)$$

$$\alpha = (H_{min} \times H_{max})^{1/2}$$

For determination of reactivity ratios by Fineman-Ross method[9], the plot of $G$ versus $H$ led to a straight line, the slope of which yielded $r_{FATRIFE}$ while the intercept worthed minus $r_{MAF}$.

For determination of reactivity ratios by Kelen-Tüdos method[10], a plot of $\eta$ versus $\zeta$ was constructed and the data points were fit linear. The intercepts *at* $\xi = 1$ and *at* $\xi = 0$ of the plot of $\eta$ *versus* $\xi$ gives $r_{FATRIFE}$ and whose intercept is minus $r_{MAF/\alpha}$, *respectively.*

**Table S3** Parameters for Fineman-Ross and Kelen-Tüdos plot for the determination of the reactivity ratios of FATRIFE and MAF in the free radical copolymerization of these comonomers

| $F_{FATTIFE}$ | $F_{MAF}$ | $x$ | $F_{FATRIFE}$ | $F_{MAF}$ | $X$ | $G$ | $H$ | $\eta$ | $\xi$ |
|---|---|---|---|---|---|---|---|---|---|
| 0.2 | 0.8 | 0.250 | 0.58 | 0.42 | 1.381 | 0.069 | 0.045 | 0.121 | 0.079 |
| 0.3 | 0.7 | 0.423 | 0.65 | 0.35 | 1.857 | 0.198 | 0.098 | 0.317 | 0.158 |
| 0.5 | 0.5 | 1.0 | 0.73 | 0.27 | 2.704 | 0.630 | 0.370 | 0.704 | 0.413 |
| 0.7 | 0.3 | 2.333 | 0.82 | 0.18 | 4.556 | 1.821 | 1.195 | 1.059 | 0.695 |
| 0.8 | 0.2 | 4.0 | 0.88 | 0.12 | 7.333 | 3.454 | 2.182 | 1.276 | 0.806 |
| 0.9 | 0.1 | 9.0 | 0.93 | 0.07 | 13.286 | 8.322 | 6.097 | 1.257 | 0.921 |
| 1.0 | 0 | * | 1.00 | 0 | * | * | * | * | * |

*symbol represents undefined values

**Table S4** Comparison of monomer reactivity ratios determined by different methods

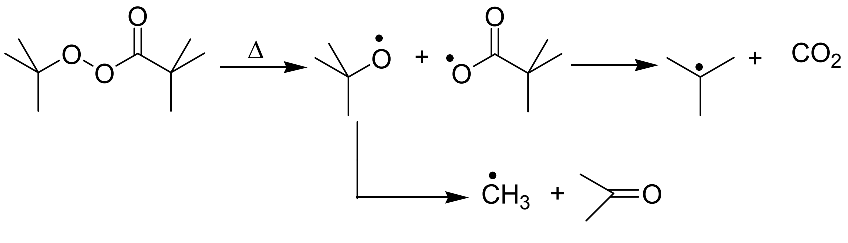
| *Method* | $r_{FATRIFE}$ | $r_{MAF}$ | $r_{FATRIFE} \times r_{MAF}$ |
|---|---|---|---|
| Mayo-Lewis | 1.65 | 0 | 0 |
| Fineman-Ross | 1.39 | 0 | 0 |
| Kelen-Tüdos | 1.50 | 0 | 0 |

**Table S5** Monomer Reactivity of Radical Copolymerization of MAF or MAF-esters with other Monomers.

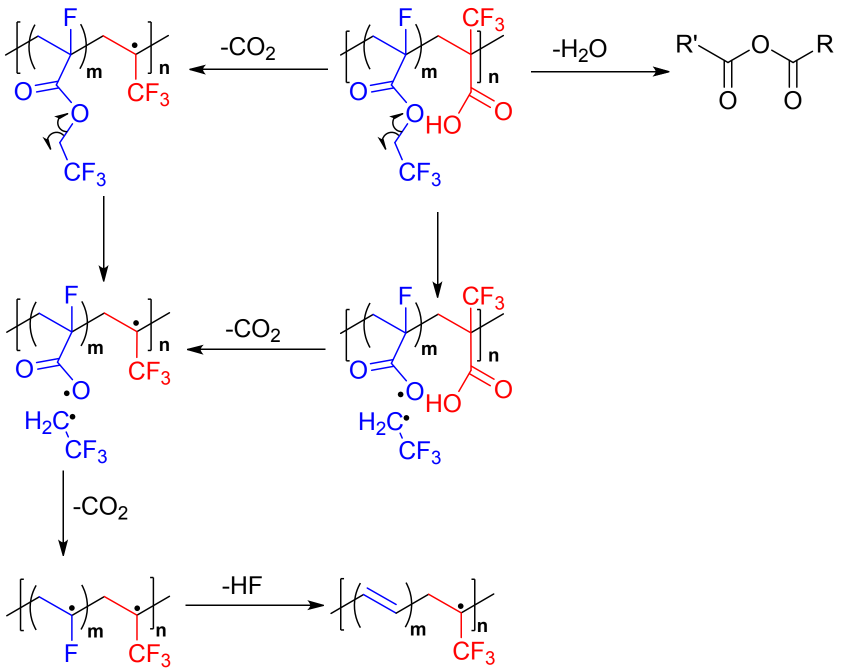
| Monomer (M1) | Monomer (M2) | Temp. ($^{o}$C) | $r_{M1}$ | $r_{M2}$ | Reference |
|---|---|---|---|---|---|
| $H_2C{=}C(CF_3)CO_2C(CH_3)_3$ | MAN | 55 | 0.112 | 5.29 | 11 |
| $H_2C{=}C(CF_3)CO_2C(CH_3)_3$ | FAV8 | 74 | 0 | 0.03 | 12 |
| $H_2C{=}C(CF_3)CO_2C(CH_3)_3$ | FATRIFE | 74 | 0 | 1.71 | 13 |
| $H_2C{=}C(CF_3)CO_2C(CH_3)_3$ | STHFA | 75 | 0.001 | 0.460 | 14 |
| $H_2C{=}C(CF_3)CO_2C(CH_3)_3$ | styrene | 60 | 0.090 | 0.130 | 15 |
| $H_2C{=}C(CF_3)CO_2C(CH_3)_3$ | EVE | 60 | 0.026 | 0 | 16, 17 |
| $H_2C{=}C(CF_3)CO_2C(CH_3)_3$ | DHF | 60 | 0.250 | 0 | 16, 17 |
| $H_2C{=}C(CF_3)CO_2C(CH_3)_3$ | NBHFA | 60 | 0.289 | 0.001 | 16, 17 |
| $H_2C{=}C(CF_3)CO_2C(CH_3)_3$ | NB | 70 | 0.530 | 0 | 16, 17 |
| $H_2C{=}C(CF_3)CO_2C(CH_3)_3$ | TFEMA | 74 | 0.100 | 4.870 | 18 |
| $H_2C{=}C(CF_3)CO_2C(CH_3)_3$ | $H_2C{=}CFCO_2CH_2CF_3$ | 74 | 0.110 | 0.560 | 19 |
| $H_2C{=}C(CF_3)CO_2C(CH_3)_3$ | $H_2C{=}CFCO_2CH_2CF_3$ | 74 | 0.61 | 1.520 | 20 |
| $H_2C{=}C(CF_3)CO_2CH_3$ | $H_2C{=}C(CH_3)CO_2CH_3$ | 60 | 0 | 2.360 | 21 |
| $H_2C{=}C(CF_3)CO_2CH_3$ | $F_2C{=}CF_2$ |  | 0.15 | 3.0 | 22 |
| $H_2C{=}C(CF_3)CO_2H$ | $H_2C{=}CF_2$ | 55 | 0 | 0.330 | 23 |
| $H_2C{=}C(CF_3)CO_2CCH_3$ | MMA | 70 | 0 | 2.360 | 1, 24-26 |
| $H_2C{=}C(CF_3)CO_2CCH_3$ | Styrene | 75 | 0.070 | 0.630 | 25 |
| $H_2C{=}C(CF_3)CO_2CCH_3$ | Styrene | 60 | 0.050 | 0.210 | 27 |
| MAF-ester-($CH_2CF_3$) | CMS | 75 | 0.050 | 0.210 | 10 |
| MAF-ester-($CH(CF_3)_2$) | Styrene | 60 | 0.200 | 0.210 | 28 |

| | | | | | |
|---|---|---|---|---|---|
| $H_2C{=}C(CF_3)CO_2H$ | NBHFA | 60 | 0.0006 | 0.289 | 17 |
| $H_2C{=}C(CF_3)CO_2H$ | NB | 60 | 0 | 0.530 | 17 |
| $H_2C{=}C(CF_3)CO_2H$ | $H_2C{=}CHOC_2H_5$ | 60 | 0.026 | 8.760 | 17 |
| $H_2C{=}C(CF_3)CO_2H$ | $H_2C{=}CFCO_2CH_2CF_3$ | 56 | 0 | 1.650 | This work |

Acronyms: MAN, FAV8, FATRIFE, STHFA, EVE, DHF, NBHFA, NB, TFEMA, MMA, and CMS stand for methacrylonitrile, 1,1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8-heptadecafluoro-10-(vinyloxy)decane, 2,2,2-trifluoroethyl methacrylate, hexafluoroisopropanol [4-(1,1,1,3,3,3-hexafluoro-2-hydroxypropyl)-styrene, ethyl vinyl ether, 3,4-dihydrofurane, 5-(2-trifluoromethyl-1,1,1-trifluoro-2-hydroxylpropyl)-2-norbornene, norbornene, 2,2,2-trifluoroethyl α-fluoroacrylate, methyl methacrylate, and chloromethylstyrene, respectively.



**Scheme S1** Decomposition of TBPPi initiator into *tert*-butoxyl and *tert*-butylcarboxyl that may further releasing *tert*-butyl and methyl radicals



**Scheme S2** Thermolytic pathway for the decarboxylation poly(FATRIFE-*co*-MAF) copolymer and possible formation of anhydride

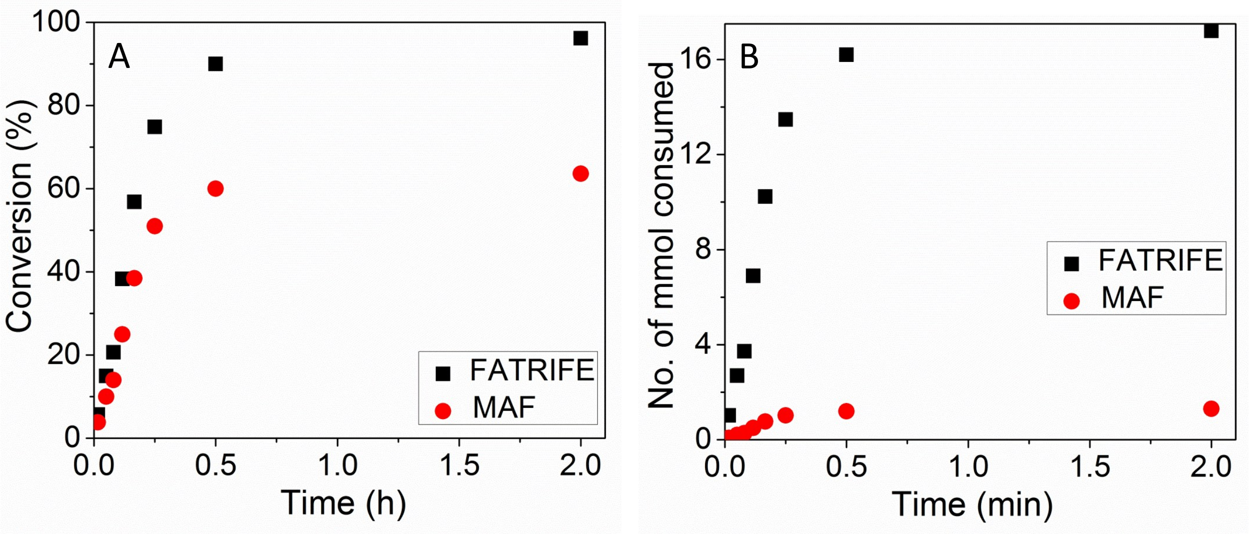


**Fig. S1** Conversion vs. time plot of FATRIFE and MAF in terms of their conversion in % (A) and no. of mmol consumed (B) during their copolymerization ($f_{MAF} = 0.1$) initiated by TBPPi at 56 °C in AcN (P2, Table 1).

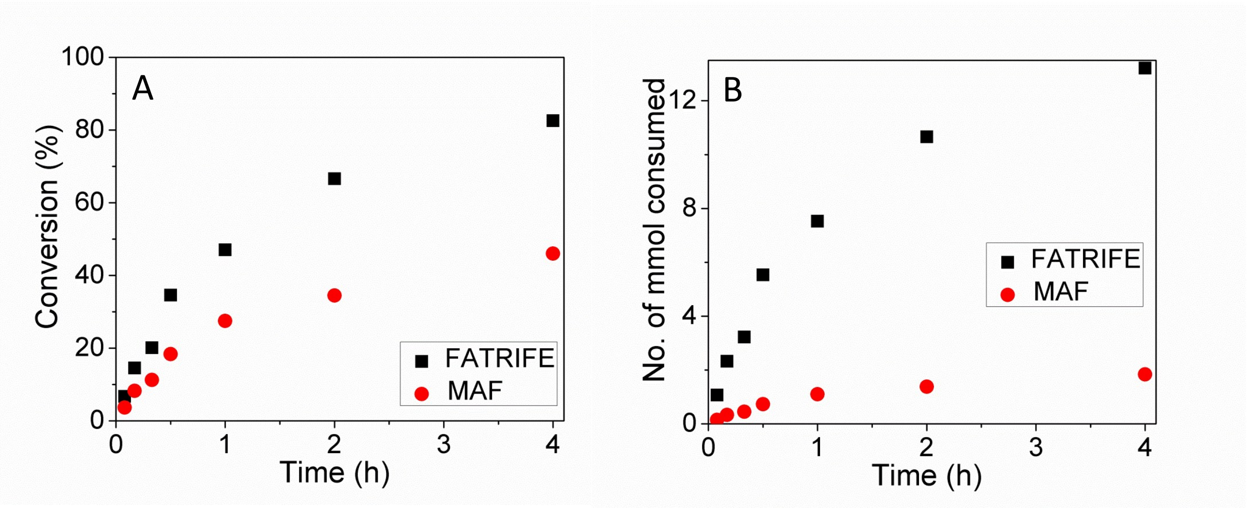


**Fig. S2** Conversion vs. time plot of FATRIFE and MAF in terms of their conversion in % (A) and no. of mmol consumed (B) during their copolymerization ($f_{MAF}$ = 0.2) initiated by TBPPi at 56 °C in AcN (P3, Table 1).

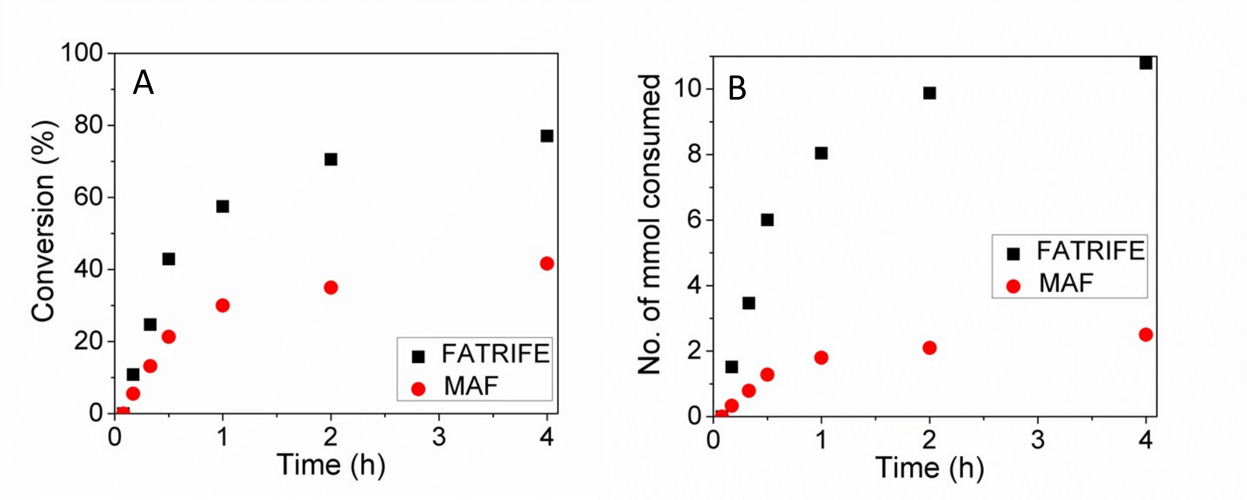


**Fig. S3** Conversion vs. time plot of FATRIFE and MAF in terms of their conversion in % (A) and no. of mmol consumed (B) during their copolymerization ($f_{MAF}$ = 0.3) initiated by TBPPi at 56 °C in AcN (P4, Table 1).

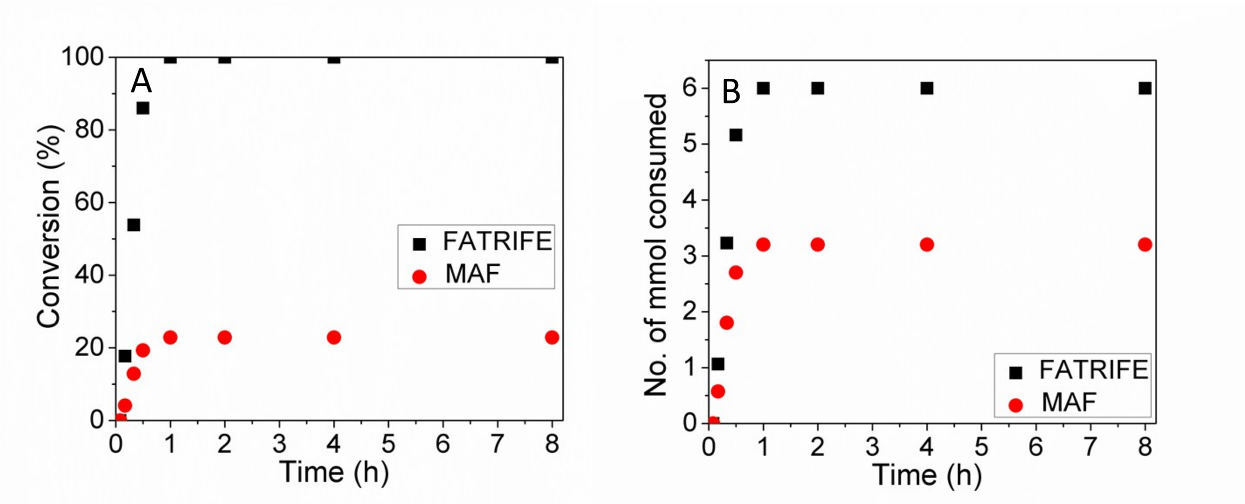


**Fig. S4** Conversion vs. time plot of FATRIFE and MAF in terms of their conversion in % (A) and no. of mmol consumed (B) during their copolymerization ($f_{MAF}$ = 0.7) initiated by TBPPi at 56 °C in AcN (P6, Table 1).

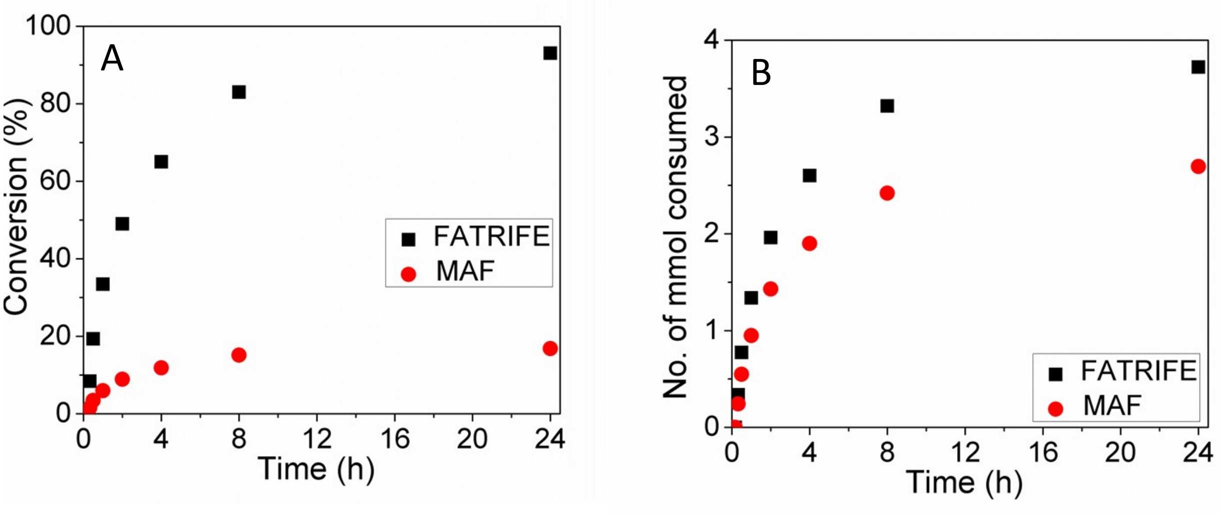


**Fig. S5** Conversion vs. time plot of FATRIFE and MAF in terms of their conversion in % (A) and no. of mmol consumed (B) during their copolymerization ($f_{MAF}$ = 0.8) initiated by TBPPi at 56 °C in AcN (P7, Table 1).

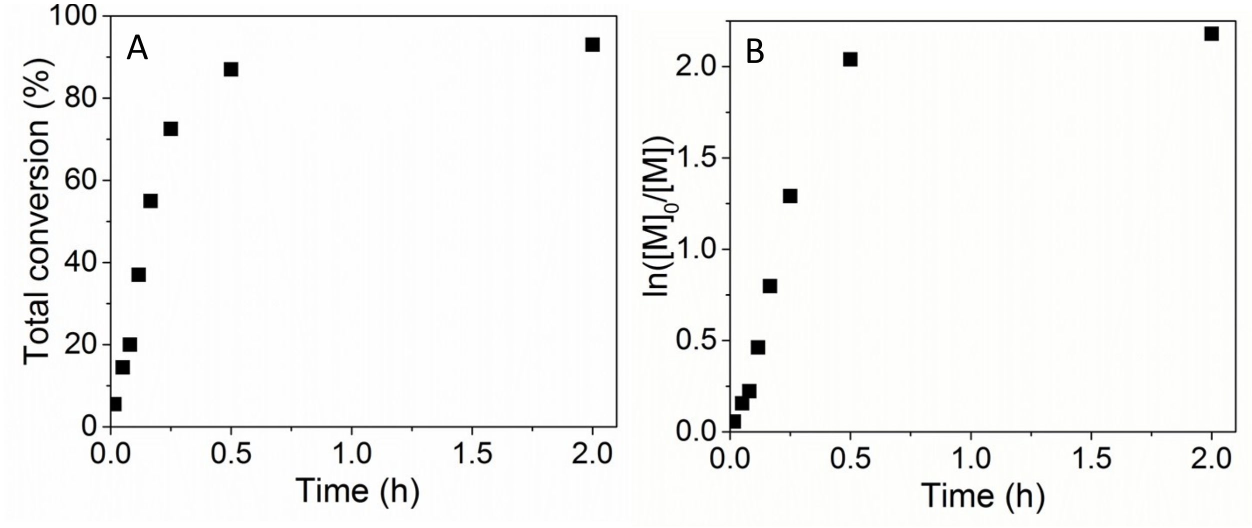


**Fig. S6** Total monomer conversion vs. time (A) and ln([M]$_0$/[M]) vs. time (B) plots during conventional radical copolymerization of FATRIFE with MAF ($f_{MAF}$ = 0.1) initiated by TBPPi at 56 °C in AcN (P2, Table 1).

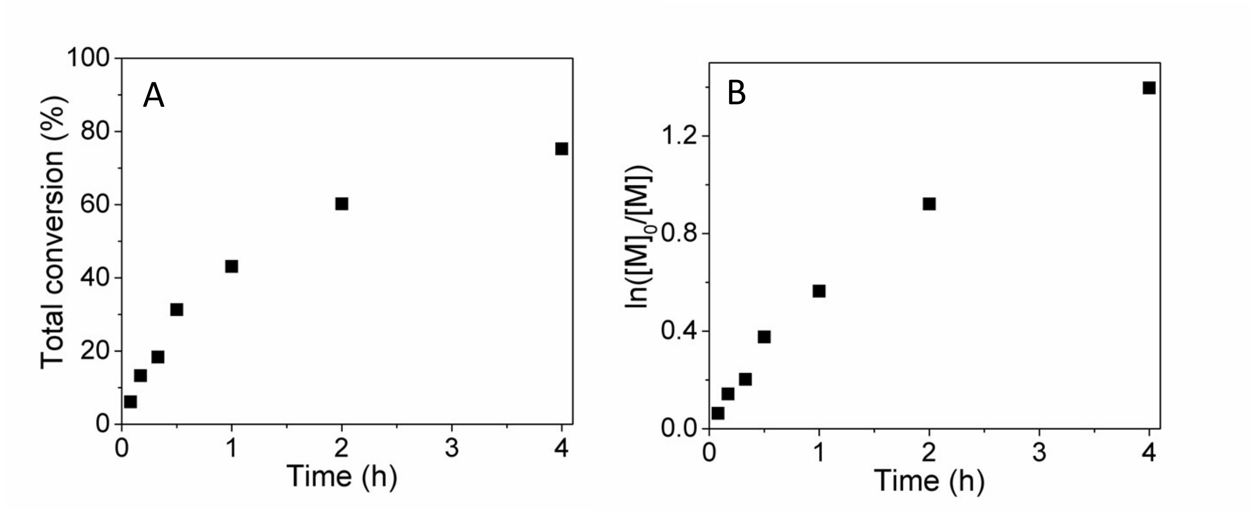


**Fig. S7** Total monomer conversion vs. time (A) and ln([M]$_0$/[M]) vs. time (B) plots during conventional radical copolymerization of FATRIFE with MAF ($f_{MAF}$ = 0.2) initiated by TBPPi at 56 °C in AcN (P3, Table 1).

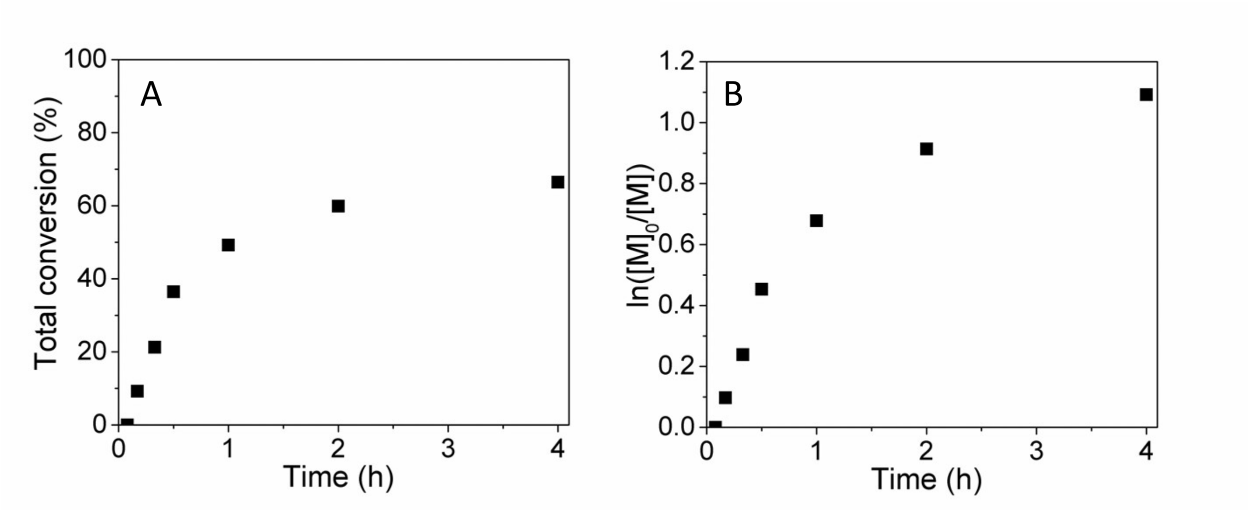


**Fig. S8** Total monomer conversion vs. time (A) and $\ln([M]_0/[M])$ vs. time (B) plots during conventional radical copolymerization of FATRIFE with MAF ($f_{MAF} = 0.3$) initiated by TBPPi at 56 °C in AcN (P4, Table 1).

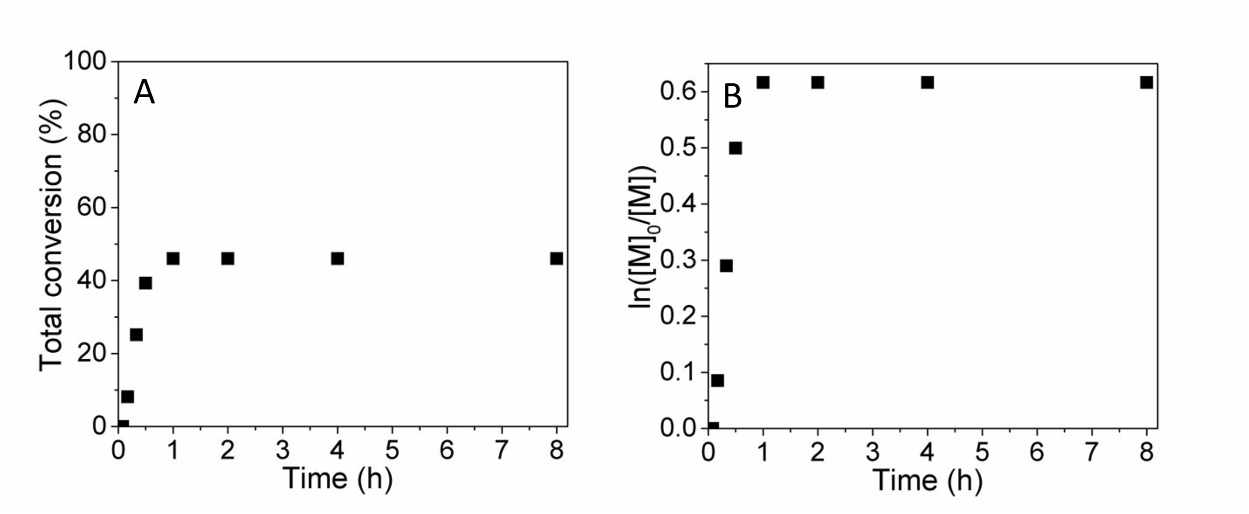


**Fig. S9** Total monomer conversion vs. time (A) and $\ln([M]_0/[M])$ vs. time (B) plots during conventional radical copolymerization of FATRIFE with MAF ($f_{MAF} = 0.7$) initiated by TBPPi at 56 °C in AcN (P6, Table 1).

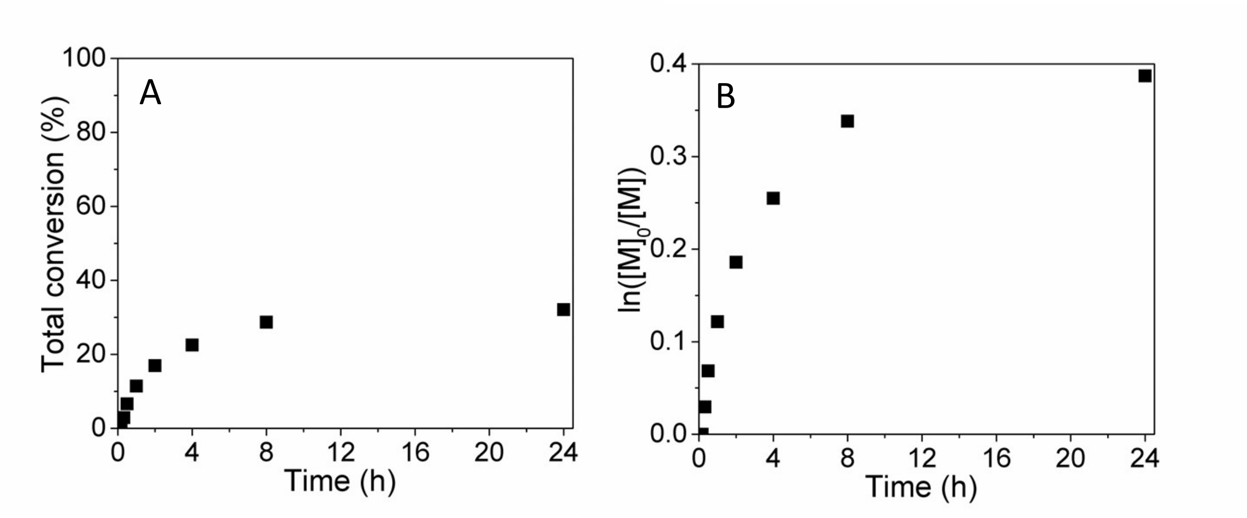


**Fig. S10** Total monomer conversion vs. time (A) and ln([M]$_0$/[M]) vs. time (B) plots during conventional radical copolymerization of FATRIFE with MAF ($f_{MAF}$ = 0.8) initiated by TBPPi at 56 °C in AcN (P7, Table 1).

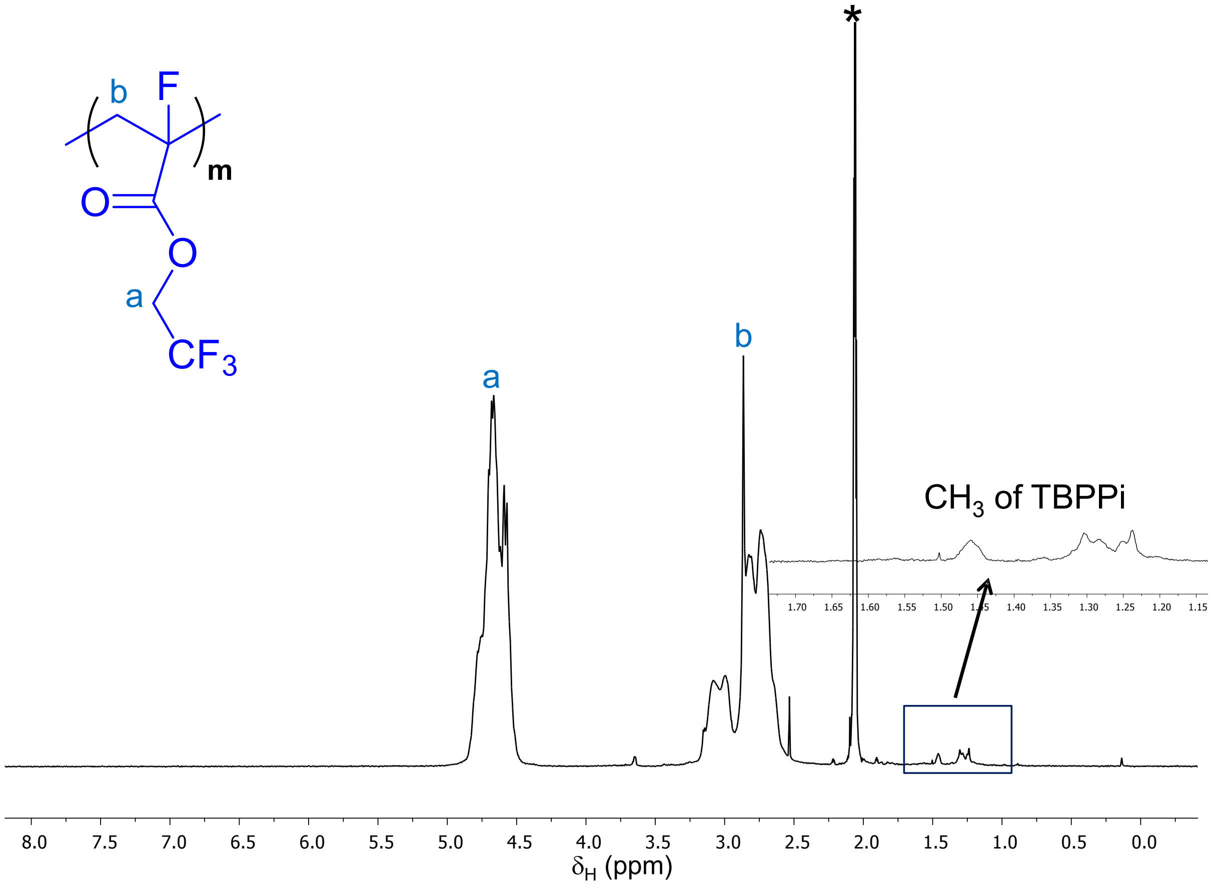


**Fig. S11** $^1$H NMR spectrum of PFATRIFE prepared by conventional radical copolymerization of FATRIFE using TBPPi in AcN at 56 °C (P1, Table 1), recorded in acetone-$d_6$ at 20 °C. (*) Solvent (acetone) peak.

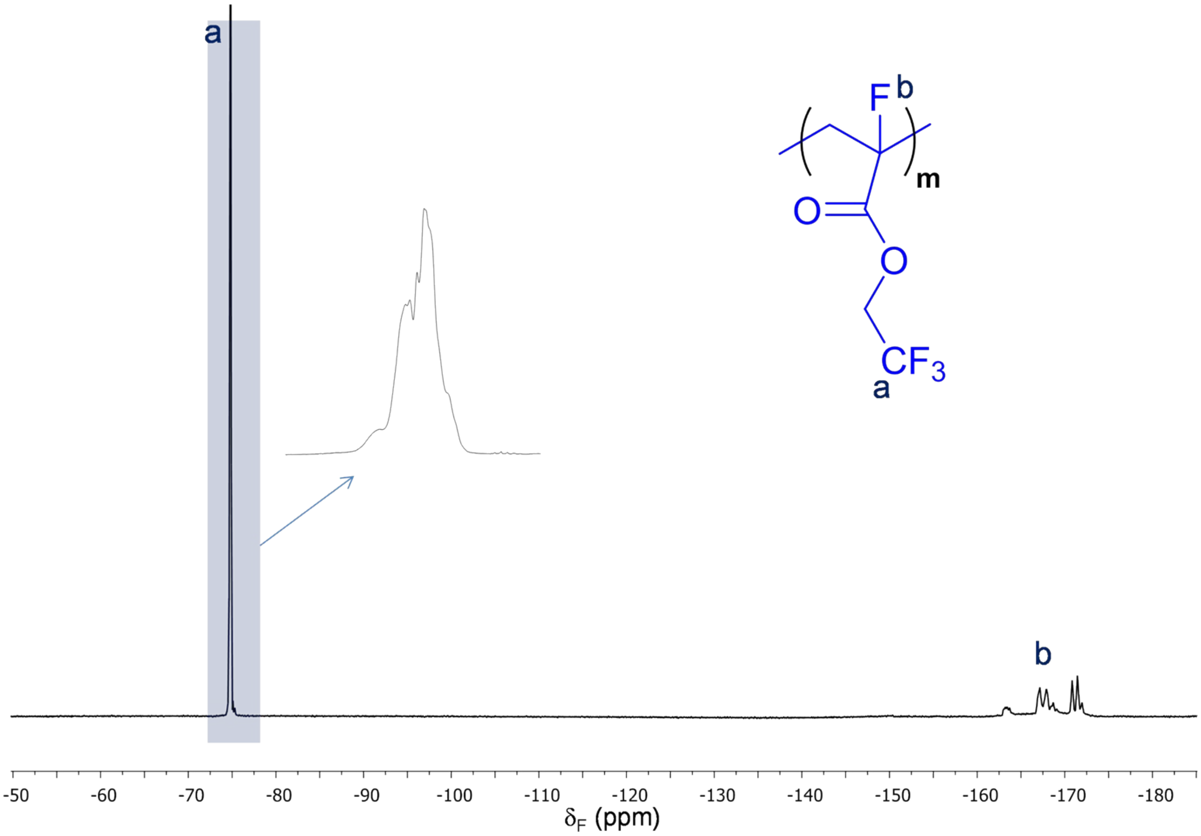


**Fig. S12** $^{19}$F NMR spectrum of PFATRIFE prepared by conventional radical copolymerization of FATRIFE using TBPPi in AcN at 56 °C (P1, Table 1), recorded in acetone-$d_6$ at 20 °C.

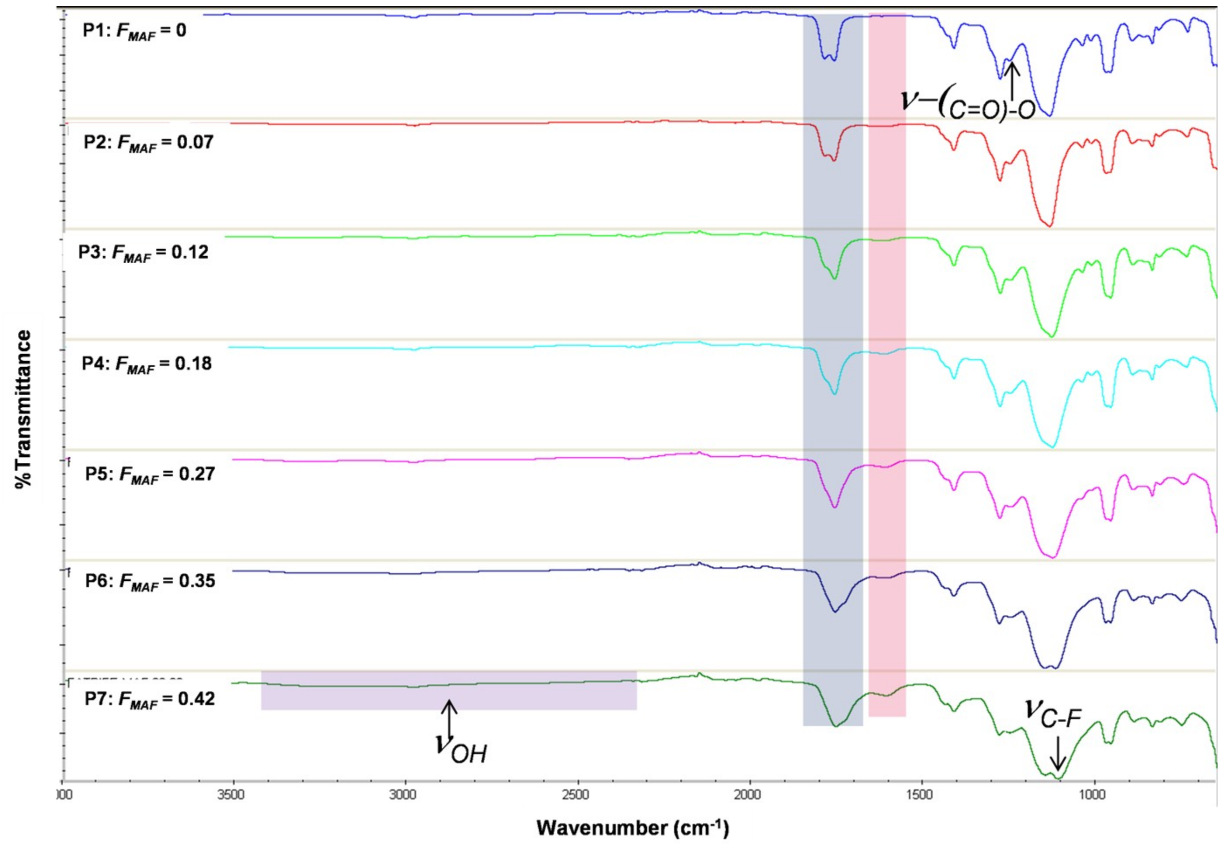


**Fig. S13** ATR-IR spectra of PFATRIFE and poly(FATRIFE-*co*-MAF) copolymers (P1-P7, Table 1). The pink zone indicates the C=O stretching frequency of MAF, while the blue shade indicates the C=O stretching frequency of FATRIFE.

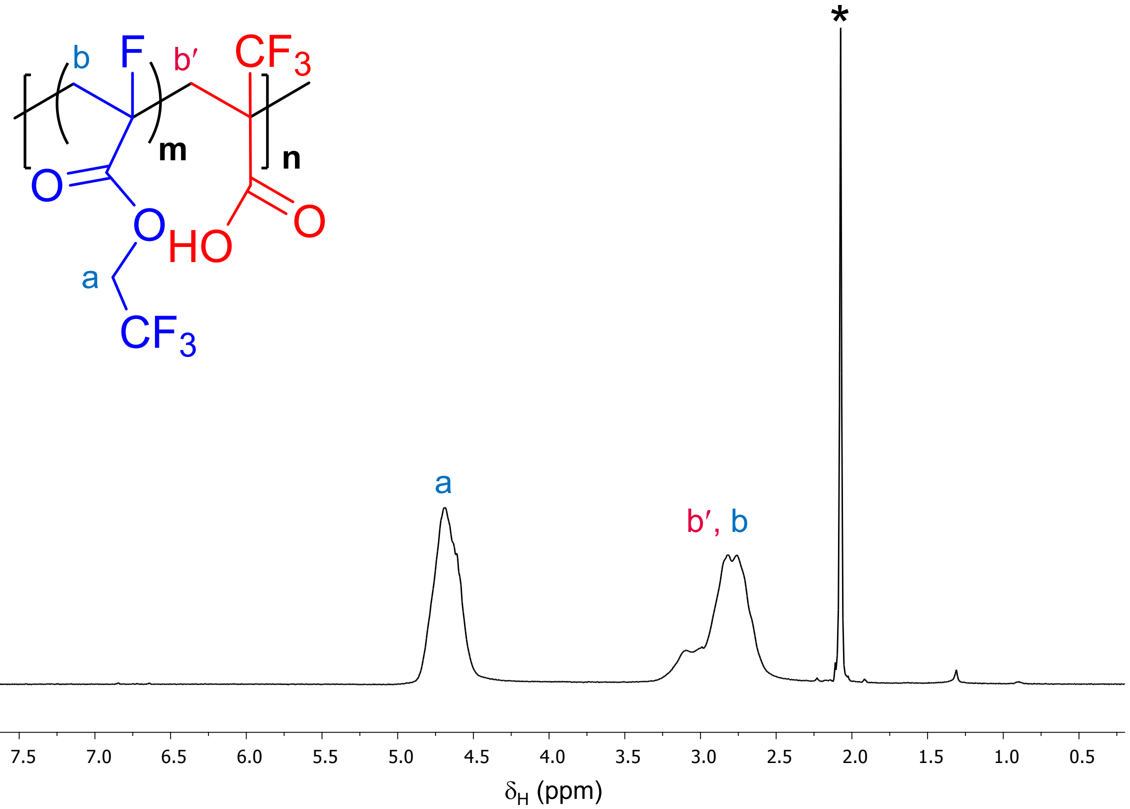


**Fig. S14** $^{1}$H NMR spectrum of poly(FATRIFE-*co*-MAF) copolymer prepared by conventional radical copolymerization of FATRIFE and MAF using PPFR radical in AcN at 80 °C (P8, Table 1), recorded in acetone-$d_6$ at 20 °C. (*) Solvent (acetone) peak.

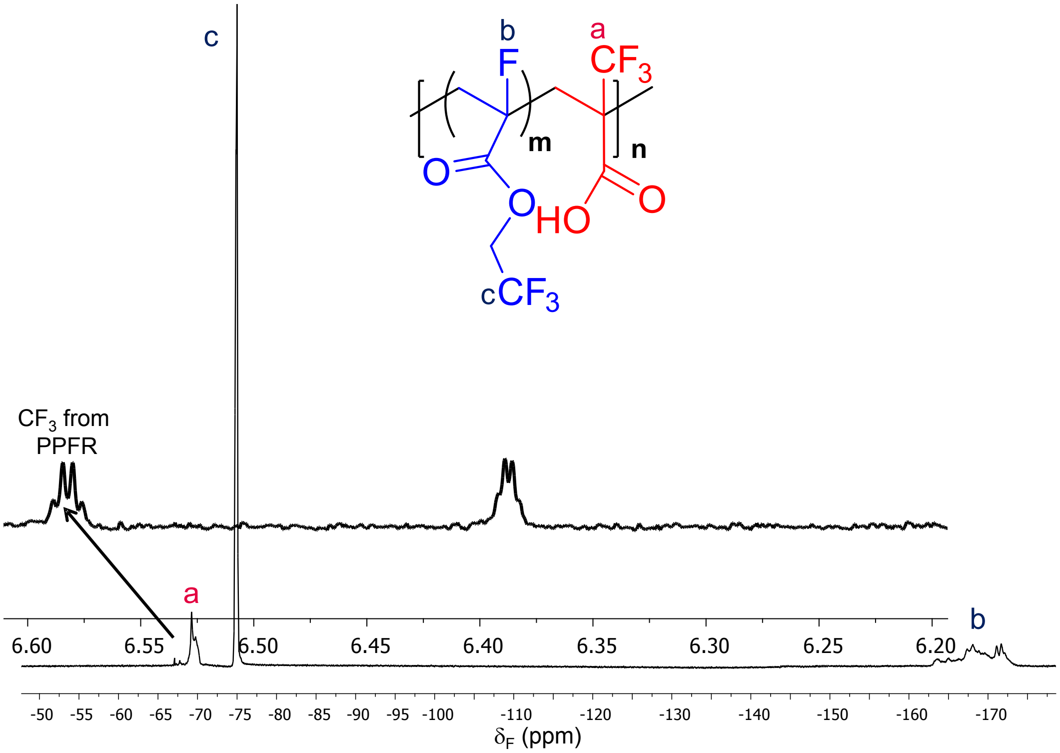


**Fig. S15** $^{19}$F NMR spectrum of poly(FATRIFE-*co*-MAF) copolymer prepared by conventional radical copolymerization of FATRIFE and MAF using PPFR in AcN at 80 °C (P8, Table 1), recorded in acetone-$d_6$ at 20 °C.

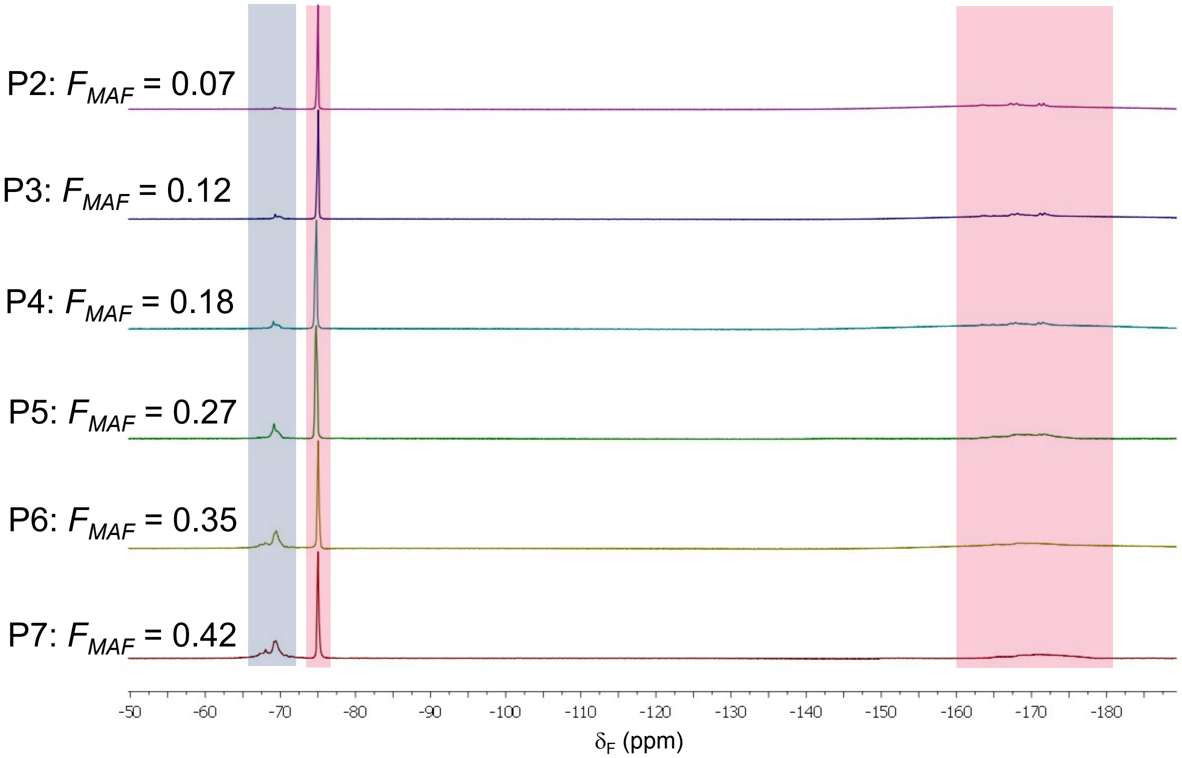
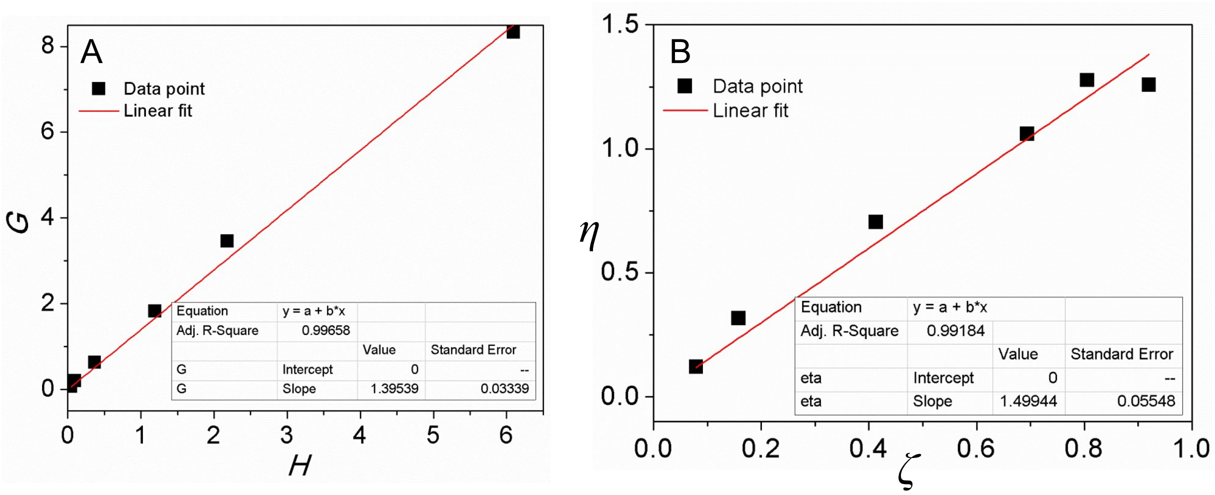


**Fig. S16.** $^{19}$F NMR spectra of poly(FATRIFE-*co*-MAF) copolymers at low conversion (<10%) (P2-P7, Table 1) prepared by conventional radical copolymerization of FATRIFE and MAF using TBPPi in AcN at 56 °C, recorded in acetone-$d_6$ at 20 °C. The instantaneous composition of the copolymers determined from these spectra were used to construct copolymer-monomer composition curve (Fig. 5).



| Equation | y = a + b*x | | |
|---|---|---|---|
| Adj. R-Square | 0.99658 | | |
| | | Value | Standard Error |
| G | Intercept | 0 | -- |
| G | Slope | 1.39539 | 0.03339 |

| Equation | y = a + b*x | | |
|---|---|---|---|
| Adj. R-Square | 0.99184 | | |
| | | Value | Standard Error |
| eta | Intercept | 0 | -- |
| eta | Slope | 1.49944 | 0.05548 |

**Fig. S17** (A) Fineman-Ross and (B) Kelen-Tüdos linearization plots for the radical polymerization of FATRIFE with MAF using initiated by TBPPi in AcN at 56 °C.

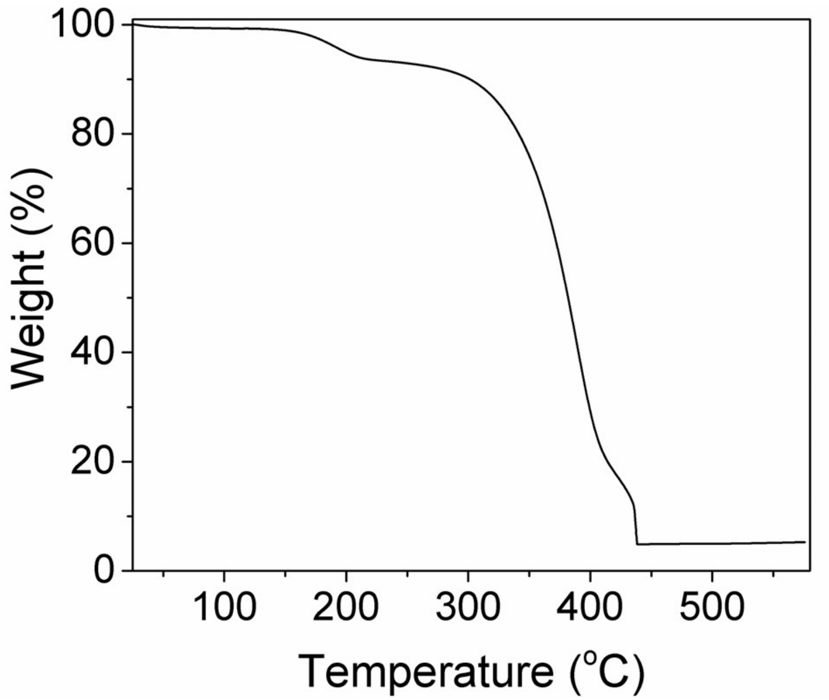


**Fig. S18** TGA thermogram of poly(FATIRFE-*co*-MAF) copolymer prepared by conventional radical copolymerization of FATRIFE and MAF using PPFR radical in AcN at 80 °C (84 mol% FATRIFE in the copolymer, P8, Table 1) heated at 10 °C $\text{min}^{-1}$ under air.

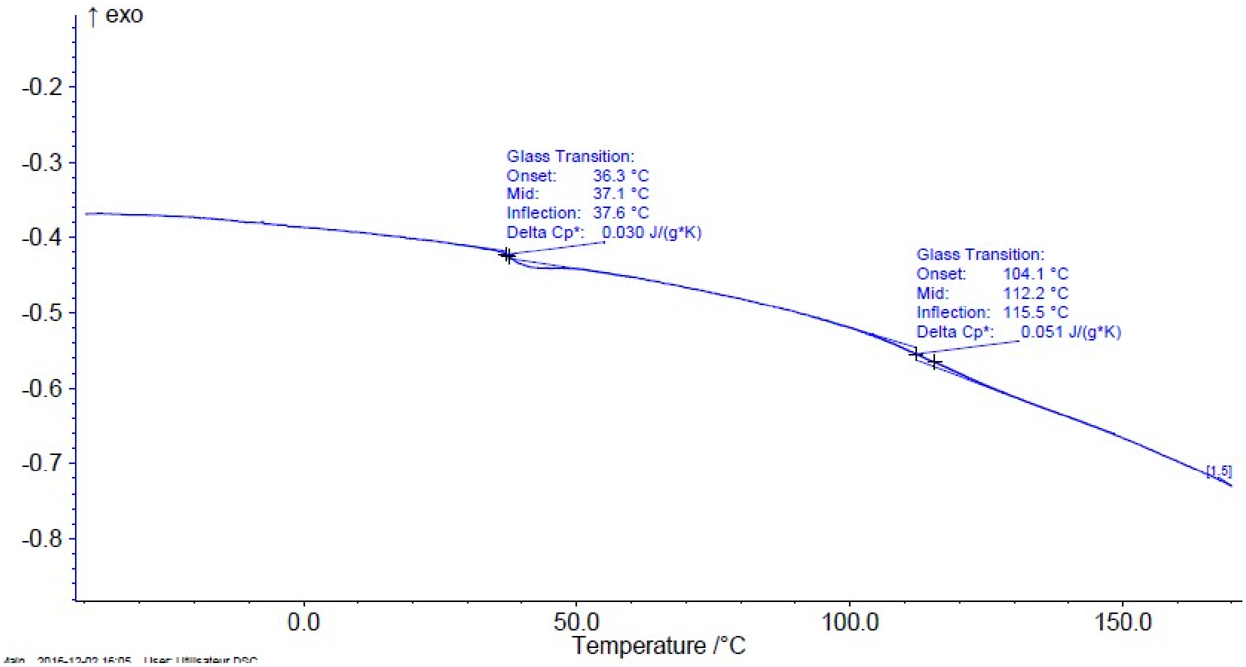


**Fig. S19** DSC thermogram of PFATRIFE (P1, Table 1).

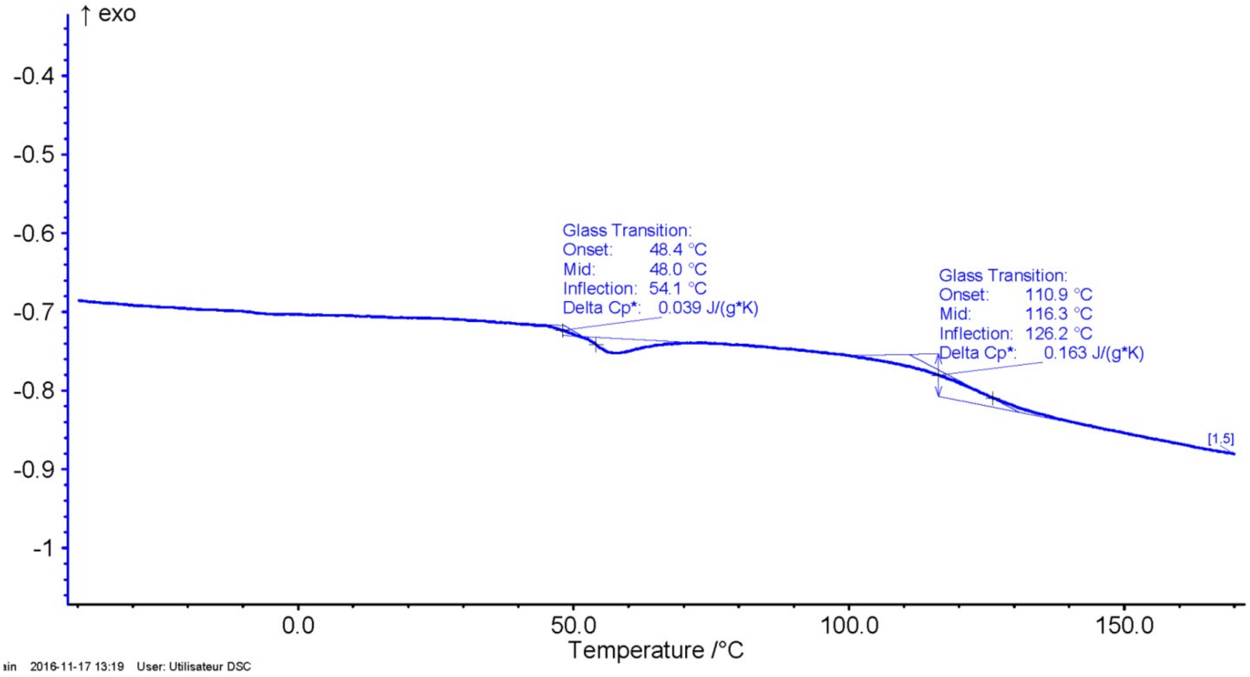


**Fig. S20** DSC thermogram of poly(FATRIFE-*co*-MAF) copolymer containing 93 mol% FATRIFE (P2, Table 1).

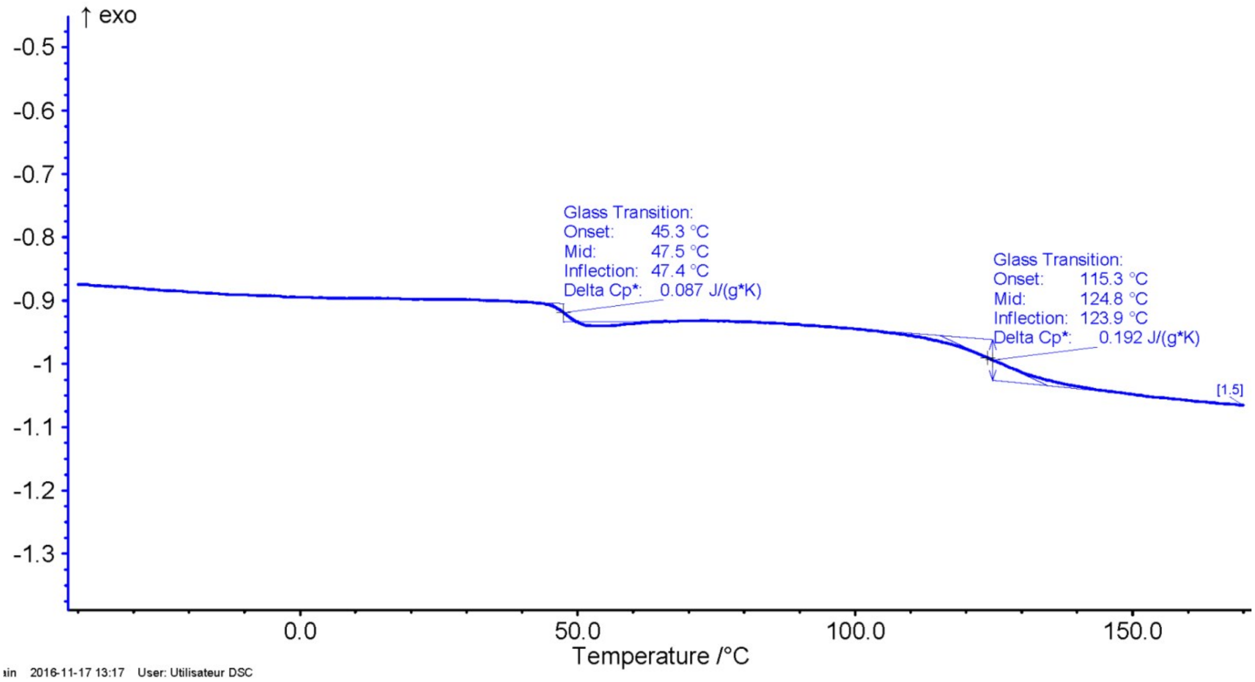


**Fig. S21** DSC thermogram of poly(FATRIFE-*co*-MAF) copolymer containing 88 mol% FATRIFE (P3, Table 1).

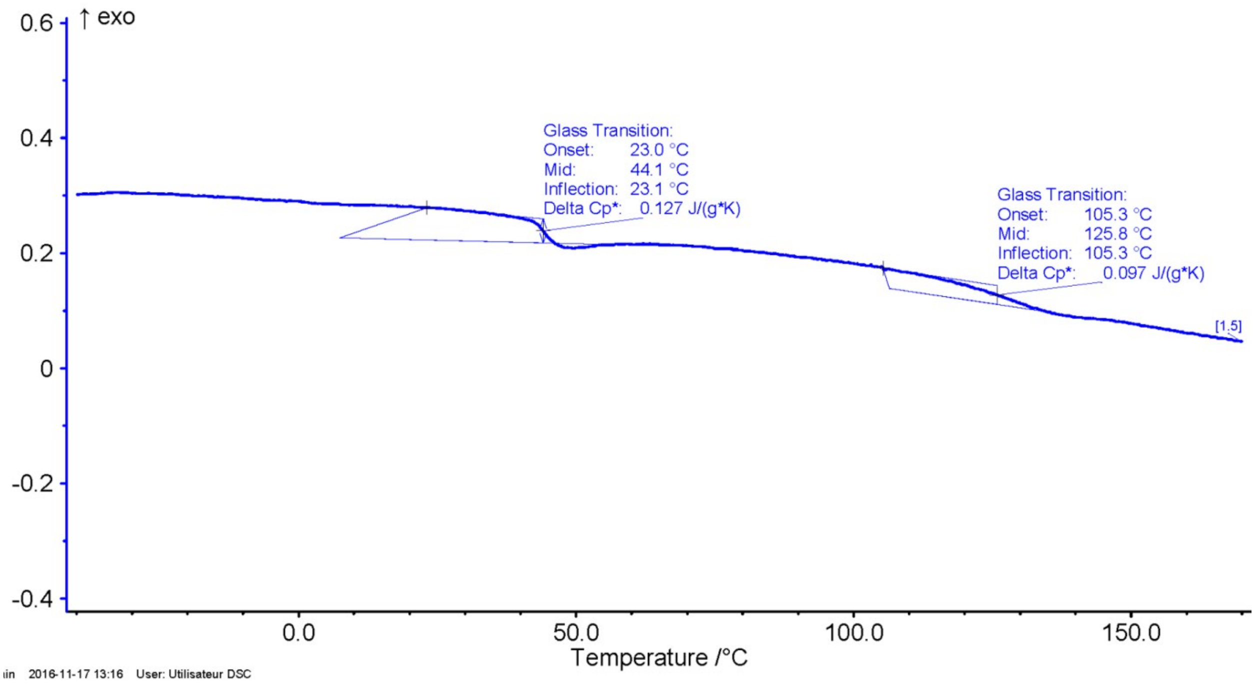


**Fig. S22** DSC thermogram of poly(FATRIFE-*co*-MAF) copolymer containing 82 mol% FATRIFE (P4, Table 1).

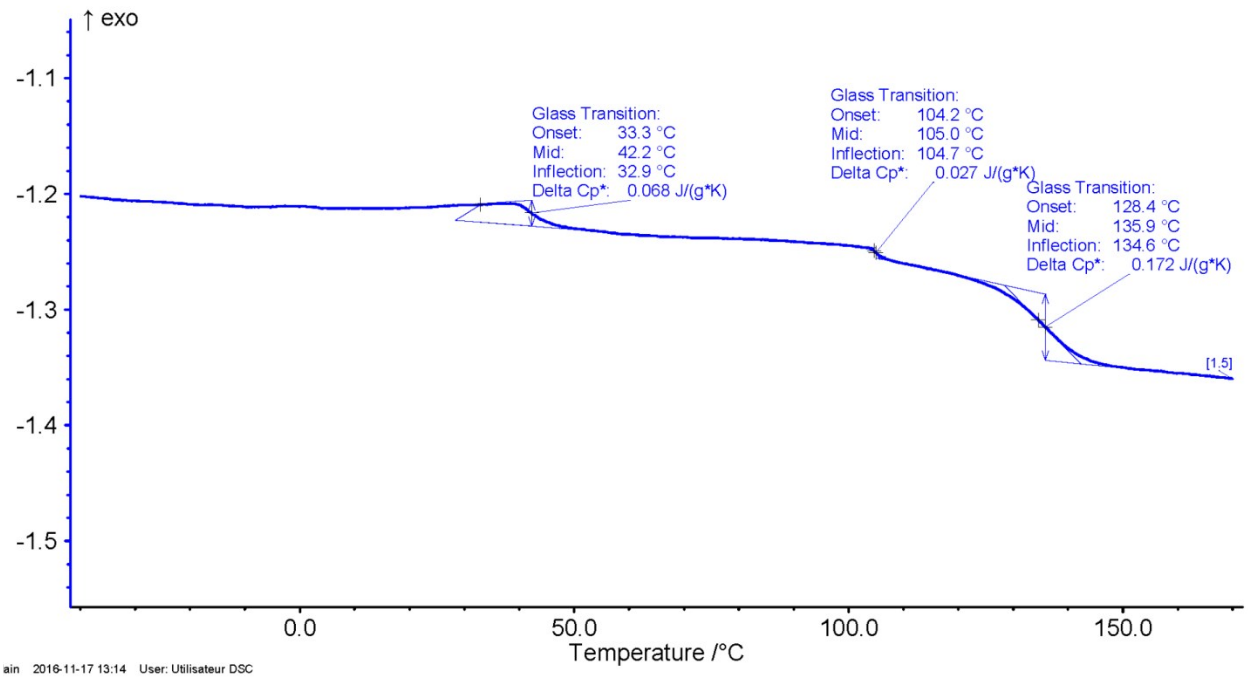


**Fig. S23** DSC thermogram of poly(FATRIFE-*co*-MAF) copolymer containing 73 mol% FATRIFE (P5, Table 1).

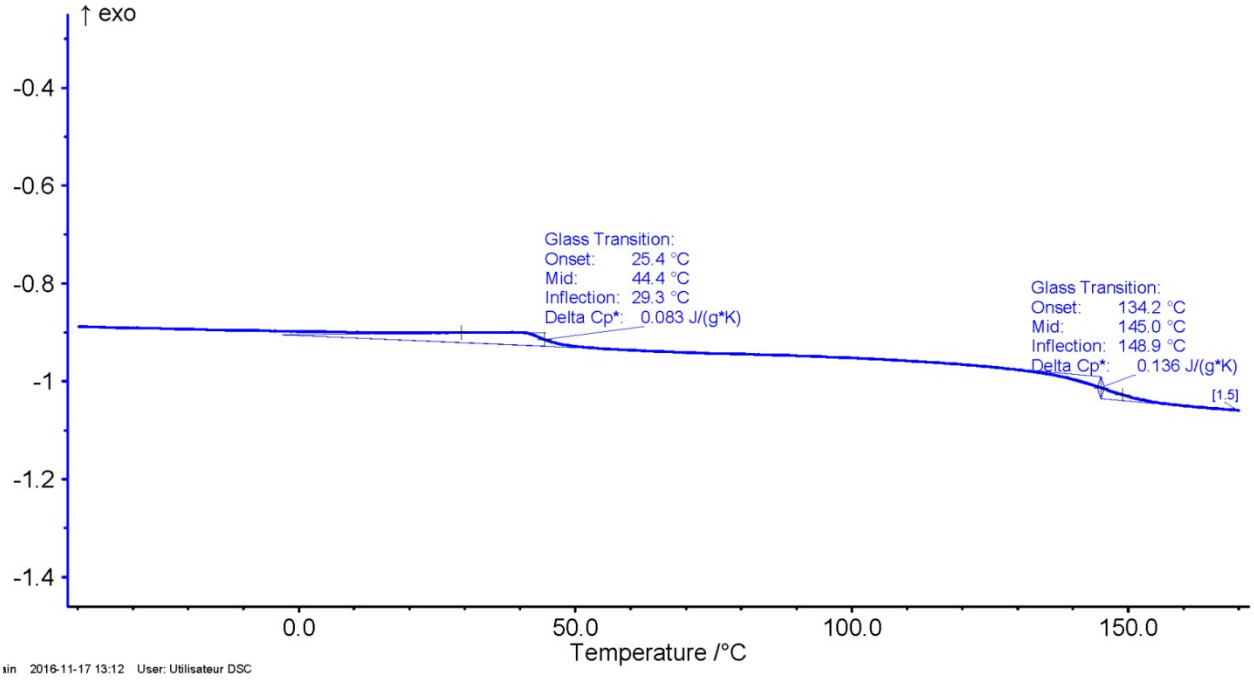


**Fig. S24** DSC thermogram of poly(FATRIFE-*co*-MAF) copolymer containing 65 mol% FATRIFE (P6, Table 1).

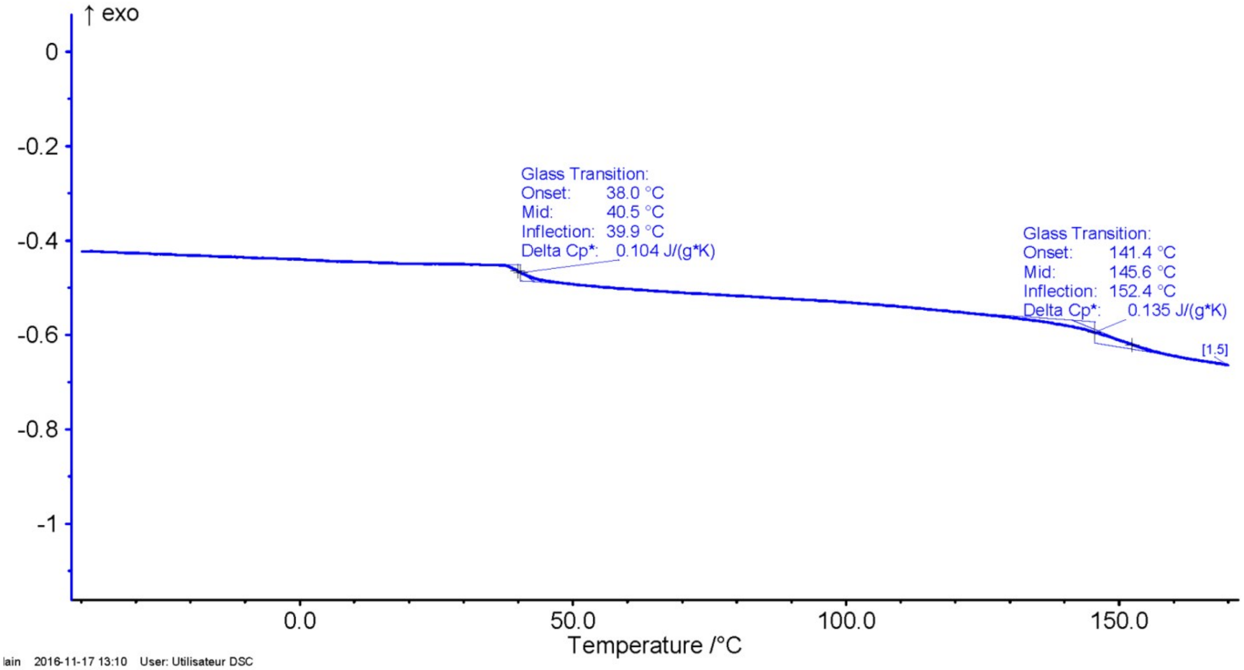


**Fig. S25** DSC thermogram of poly(FATRIFE-*co*-MAF) copolymer containing 58 mol% FATRIFE (P7, Table 1).

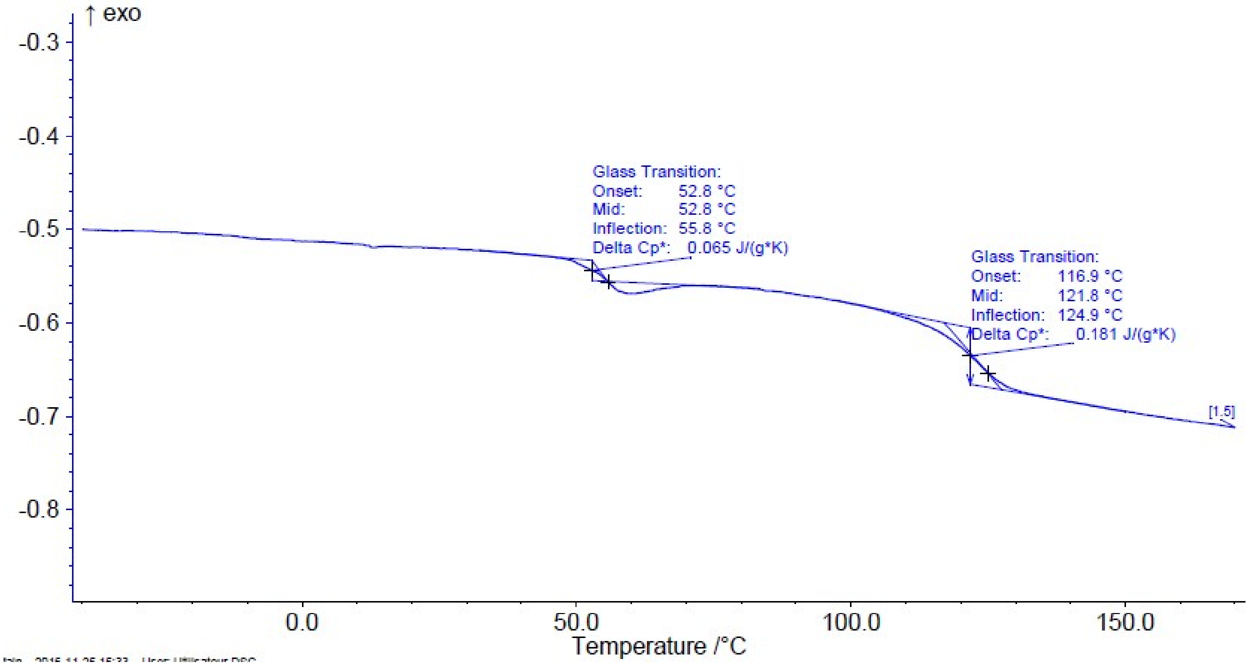


**Fig. S26** DSC thermogram of poly(FATRIFE-*co*-MAF) copolymer containing 58 mol% FATRIFE (P7, Table 1).

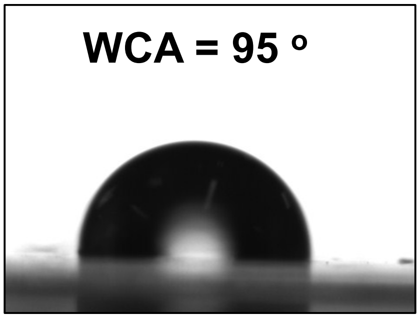


**Fig. S27** Image of water droplets deposited on the surface of poly(FATRIFE-*co*-MAF) copolymer containing 58 mol% FATRIFE (P7, Table 1).

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## References and notes

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