

## Supporting Information

### Multifunctionalization of polybutadiene by RAFT/MADIX technology

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### Table of contents

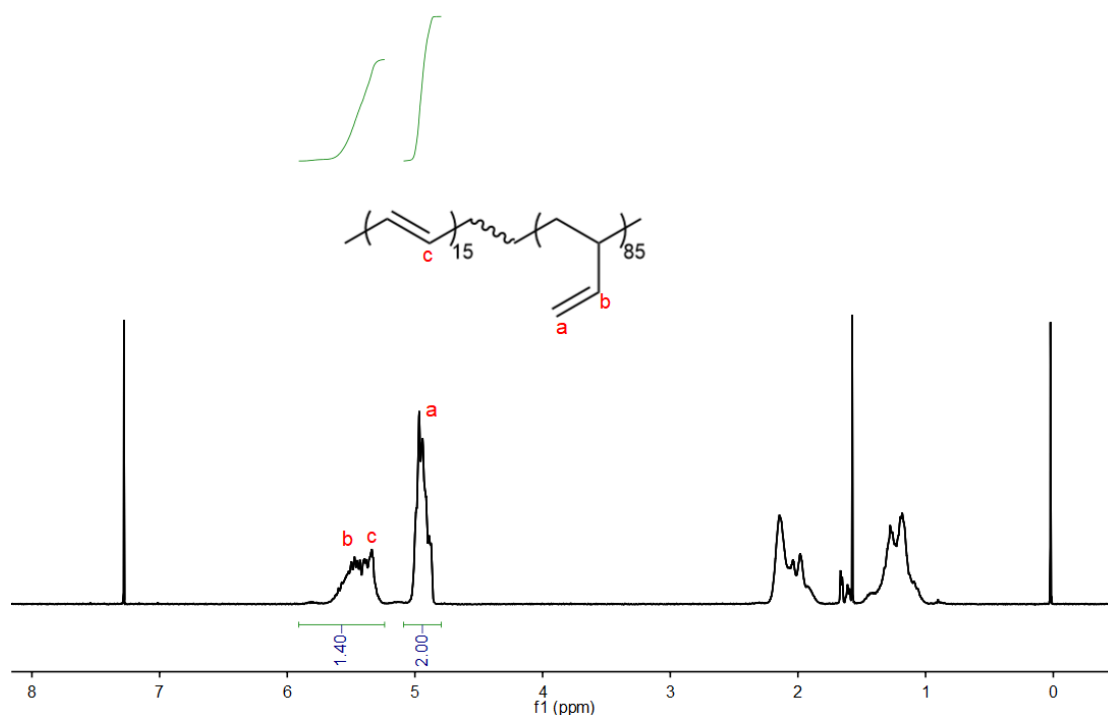
<b>1. Materials and characterization.....</b>	<b>1</b>
<b>2. Experiments and data.....</b>	<b>4</b>
<b>3. NMR spectrum.....</b>	<b>18</b>

## 1. Materials and characterization

Commercial reagents and solvent were purchased from Sigma-Aldrich, Alfa Aesar, Bidepharm, and were used without further purification. **Xan-C2** was obtained from our stockroom. Proton magnetic resonance spectra ( $^1\text{H}$  NMR) were recorded at 400 MHz and coupling constants ( $J$ ) are reported to  $\pm 0.5$  Hz. GPC characterization was performed in tetrahydrofuran (THF) solution at 35 °C with an elution rate of 1.0 mL  $\text{min}^{-1}$  on an Agilent 1260 HPLC system equipped with a G7110B pump and a G7162A refractive index detector. The molecular weights were determined using PLgel MIXED-A column and PLgel MIXED-C column using linear poly(methyl methacrylate) (PMMA) standards. For partially crosslinked polymers, we measure its dissolved components. Polymers were immersed in THF overnight, and then the suspension were centrifuged to recycle the supernatant for testing. The lamp for reaction was Kessil Tuna blue 160W.

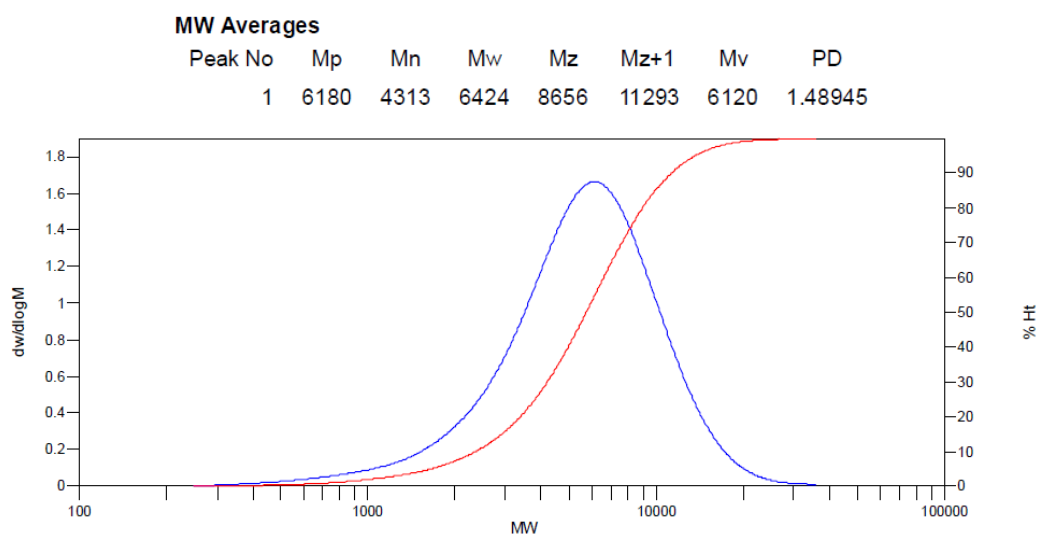
*Materials:*

polybutadiene



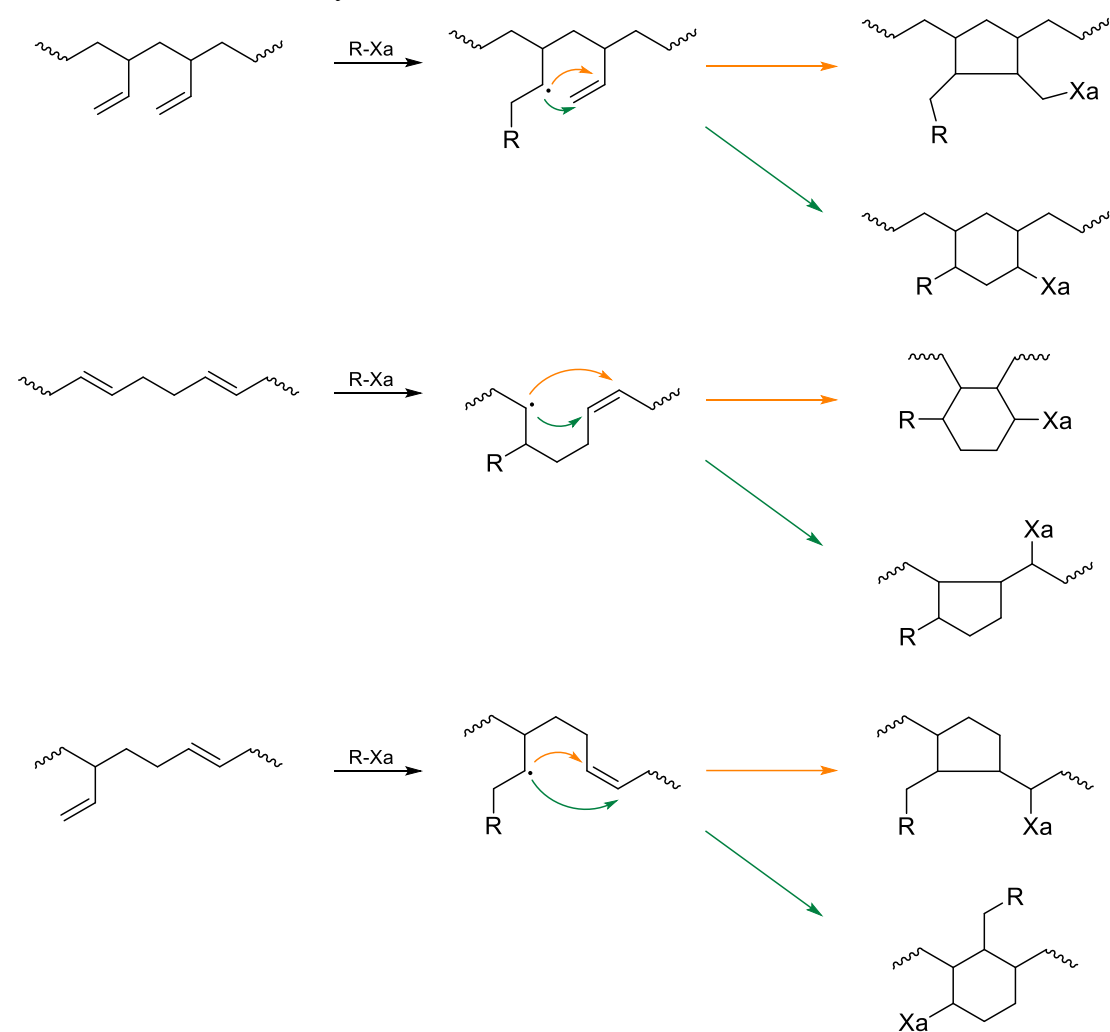
**Figure S1.** <sup>1</sup>H NMR spectrum of polybutadiene used in this work.

The results were calculated based on the polymer containing 85% double bond on side group and 15% in backbone. Two double bond peaks are remarked as **Peak-a** and **Peak-bc**.

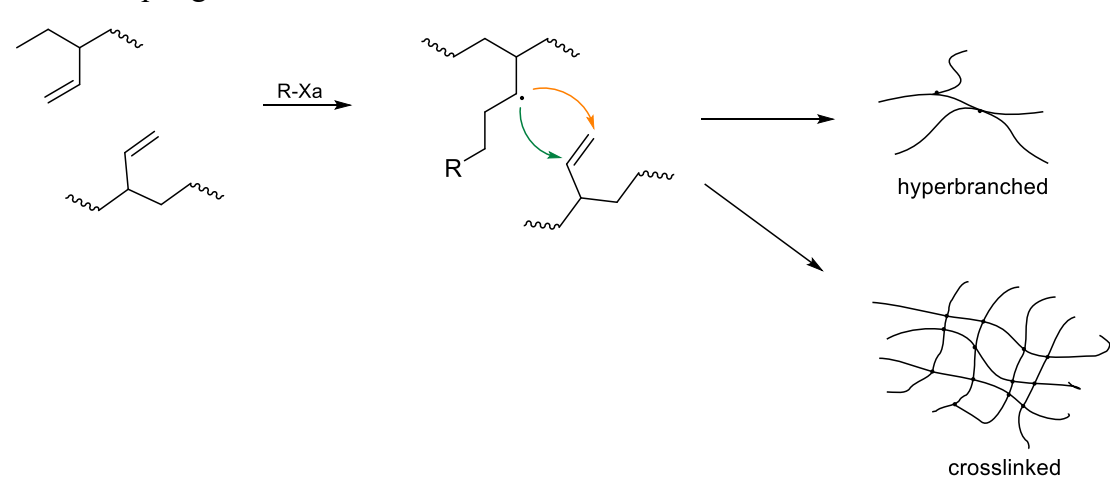


**Figure S2.** GPC analysis for the polybutadiene used (THF GPC, PMMA as standard).

### Possible intramolecular cyclizations



### Chain coupling



**Figure S3.** Possible intramolecular cyclizations during the addition reactions.

## 2. Experiments and data

### Part one: The modification of PBD by xanthate

#### Model addition reaction initiated by DLP

A model reaction initiated by DLP was performed as below. Polybutadiene (100 mg), xanthate (0.3 mmol, 1 equiv.) and DLP (0.03 mmol, 0.1 equiv.) was dissolved in pre-degassed DCE (3 mL). Before the reactions start, the original solution was tested for  $^1\text{H}$  NMR (**NMR-before**). The solution was filled in a 4 mL sealed vial, and stirred at 85 °C for 4 h. The reacted solution was conducted  $^1\text{H}$  NMR test (**NMR-after**). Reaction solution was concentrated in *vacuo* and precipitated in MeOH (15 mL). After centrifugation, the MeOH was removed and the obtained polymer was dissolved (or dispersed) in EA (1 mL). The polymer solution was then precipitated in MeOH, and this operation was repeated for 3 times. The final target polymer was dry in *vacuo* and characterized by  $^1\text{H}$  NMR in  $\text{CDCl}_3$  (**NMR-product**).

#### Model addition reaction initiated by blue light

A model reaction initiated by tuna was performed as below. Polybutadiene (100 mg), xanthate (0.3 mmol) was dissolved in pre-degassed DCE (3 mL). The solution was filled in a 4 mL sealed vial, and stirred at room temperature for 24 h under the Kessil Tuna blue lamp. A fan is installed a to ensure that the reaction system remain at room temperature. Three  $^1\text{H}$  NMR characterizations (**NMR-before**, **NMR-after**, **NMR-product**) and after-treatment were the same as above.

#### Model addition reaction initiated by $\text{Et}_3\text{B}/\text{O}_2$

A model reaction initiated by  $\text{Et}_3\text{B}/\text{O}_2$  was performed as below. Polybutadiene (100 mg), xanthate (0.3 mmol) was dissolved in pre-degassed THF. The total amount of solvent including  $\text{Et}_3\text{B}$  THF solution was 3 mL (0.3 equiv.). The solution was filled in a 5 mL vial and a needle was equipped to connect to the atmosphere.  $\text{Et}_3\text{B}$  solution was injected quickly and the solution was stirred at room temperature for 4 h. Three  $^1\text{H}$  NMR characterizations (**NMR-before**, **NMR-after**, **NMR-product**) and after-treatment were the same as above.

The calculation method for the data in below sheet

#### Conversion of double bond on side group (vinyl)

The **vinyl** was calculated from the changing in the integral value of **Peak-a** in **NMR-before** and **NMR-after**. Total amount of xanthate (both reacted and unreacted) can be used as a benchmark.

$$\mathbf{vinyl} = \frac{(\text{Integration proton a before}) - (\text{Integration proton a after})}{(\text{Integration proton a before})} \times 100\%$$

#### Conversion of double bond on backbone (vinylene)

The **vinylene** was calculated from the changing in the integral value of **Peak-bc** in **NMR-before** and **NMR-after**. But it needs to be considered that **Peak-bc** contains one proton from side group.

$$\mathbf{vinylene} = \frac{\left(\text{bc before} - \frac{\text{a before}}{2}\right) - \left(\text{bc after} - \frac{\text{a after}}{2}\right)}{\left(\text{bc before} - \frac{\text{a before}}{2}\right)} \times 100\%$$

#### Calculation of grafting yield

$$\mathbf{grafting\ yield} = \frac{\text{Integration proton on xanthate (NMR - product)}}{\text{Integration proton on xanthate (NMR - before)}} \times 100\%$$

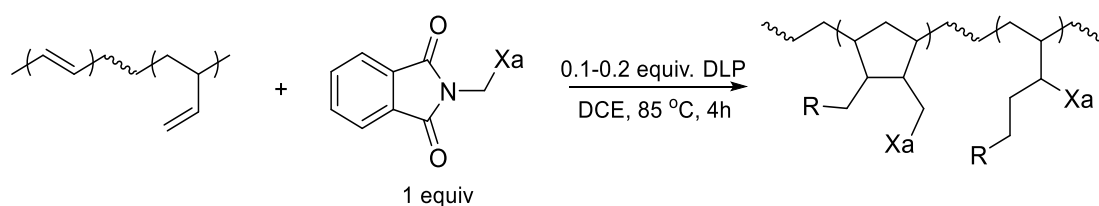
#### Calculation of degree of functionalization (f)

$$\mathbf{f} = \mathbf{grafting\ yield} / ([\text{DB}] / [\text{Xa}])$$

Calculation of average consumption of double bonds (**Con. of DB**)

$$\mathbf{Con.\ of\ DB} = \frac{0.85 \times \mathbf{vinyl} + 0.15 \times \mathbf{vinylene}}{\mathbf{f}}$$

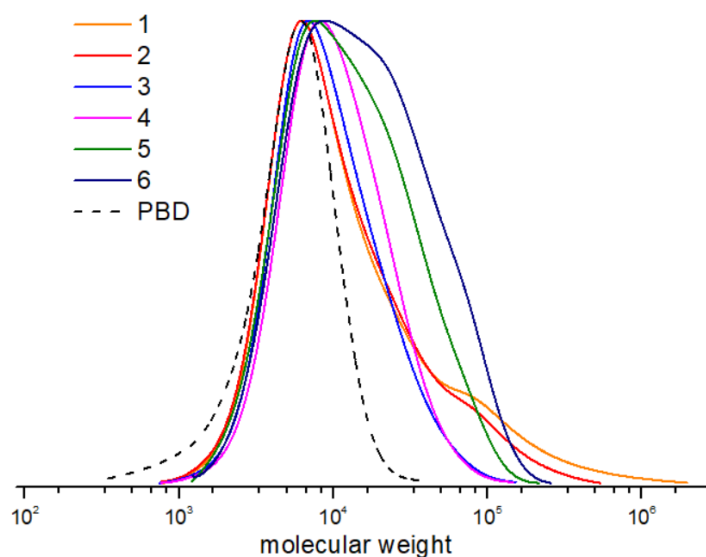
## Addition reaction of **Xan-1** initiated by DLP



**Table S1<sup>a</sup>**

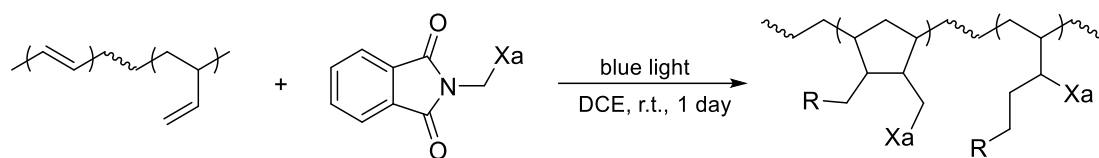
No	[DB]/[Xa] <sup>a</sup>	grafting yield (%) <sup>b</sup>	<i>f</i> (%) <sup>c</sup>	vinyl (%) <sup>d</sup>	vinylene (%) <sup>e</sup>	Con. DB <sup>f</sup>	<i>M<sub>n</sub></i> (k) <sup>g</sup>	<i>M<sub>w</sub></i> (k) <sup>g</sup>	<i>M<sub>w</sub></i> / <i>M<sub>n</sub></i> <sup>g</sup>
1	12.8/1	6.0	0.5	7.0	27.3	20.1	7.3	31.82	4.42
2	6.1/1	16.2	2.6	15.7	31.8	7.0	7.0	20.37	2.91
3	2.6/1	20.1	7.7	32.0	87.1	5.2	6.8	12.31	1.81
4	1.2/1	17.3	14.2	51.5	>99 <sup>h</sup>	4.1	7.6	13.38	1.76
5 <sup>i</sup>	6.7/1	33.5	5.0	23.6	61	5.8	8.2	18.94	2.31
6 <sup>i</sup>	2.6/1	42.7	16.7	54.8	>99 <sup>h</sup>	3.7	9.4	24.44	2.60

<sup>a</sup>Reaction conditions: 0.5 M PBD (double bond) in DCE, heated at 85 °C for 4 h, entry 1-4: 0.1 equiv. DLP (to Xan), entry 5,6: 0.2 equiv. DLP. <sup>b</sup>Ratio of total double bonds to added xanthate. <sup>c</sup>Xanthate proportion grafted onto polymer. <sup>d</sup>Degree of functionalization: grafted xanthate to polymer segment ratio. <sup>e</sup>Conversion of vinyl group. <sup>f</sup>Conversion of backbone double bonds. <sup>g</sup>Average double bonds consumed per grafted xanthate. <sup>h</sup>GPC analysis in THF with PMMA standard.



**Figure S4.** GPC traces of the addition in Table S1 (THF GPC, PMMA as standard).

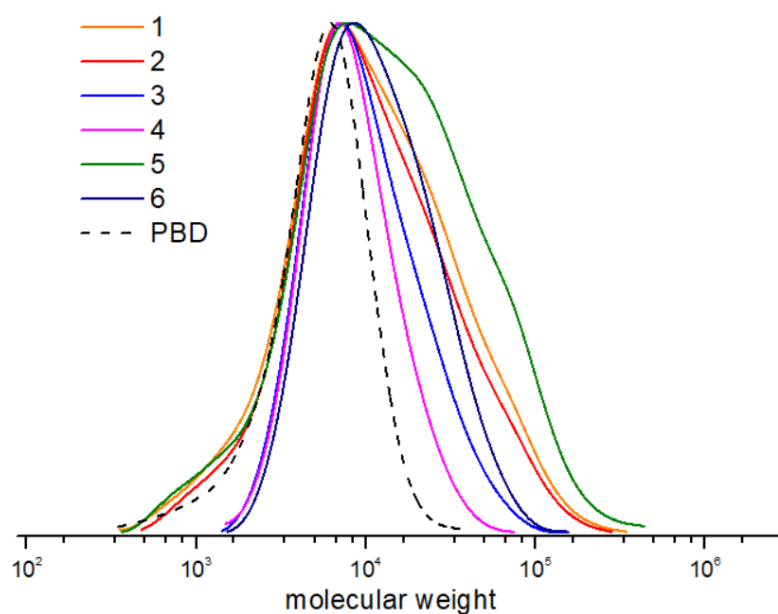
### Addition reaction of **Xan-1** initiated by blue light



**Table S2<sup>a</sup>**

No	[DB]/[Xa]	grafting yield (%)	<i>f</i> (%)	vinyl (%)	vinylene (%)	Con. DB	<i>M<sub>n</sub></i> (k)	<i>M<sub>w</sub></i> (k)	<i>M<sub>w</sub>/M<sub>n</sub></i>
1	12.2/1	58.1	4.7	23.8	54.6	6.0	6.2	19.03	3.07
2	6.2/1	33.2	5.3	27.9	67.6	6.4	6.3	18.46	2.93
3	2.5/1	15.0	5.9	22.7	85.1	5.4	7.3	12.41	1.70
4	1.2/1	3.0	2.5	12.6	54.8	7.6	6.6	9.57	1.45
5 <sup>a</sup>	6.7/1	80.0	10.0	46.6	>99	5.5	7.2	25.78	3.58
6 <sup>b</sup>	2.6/1	49.4	19.1	58.3	>99	3.4	8.6	15.22	1.77

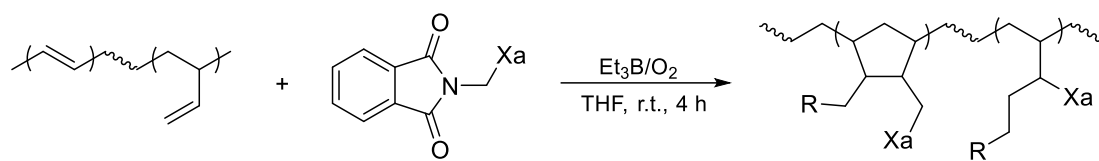
<sup>a</sup>Reaction conditions: 0.5 M PBD (double) in DCE, degassed, irradiated at room temperature for 24 h, <sup>b</sup>for 2 days, <sup>c</sup>for 4 days. <sup>d</sup>Ratio of total double bonds to added xanthate. <sup>e</sup>Xanthate proportion grafted onto polymer. <sup>f</sup>Degree of functionalization: grafted xanthate to polymer segment ratio. <sup>g</sup>Conversion of vinyl group. <sup>h</sup>Conversion of backbone double bonds. <sup>i</sup>Average double bonds consumed per grafted xanthate. <sup>j</sup>GPC analysis in THF with PMMA standard.



**Figure S5.** GPC traces of the addition in Table S2 (THF GPC, PMMA as standard).



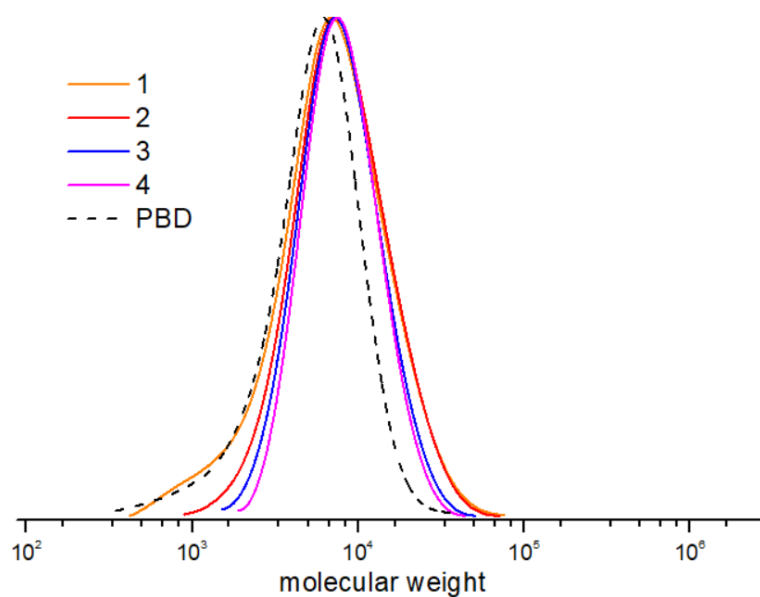
Addition reaction of **Xan-1** initiated by Et<sub>3</sub>B/O<sub>2</sub>



**Table S3<sup>a</sup>**

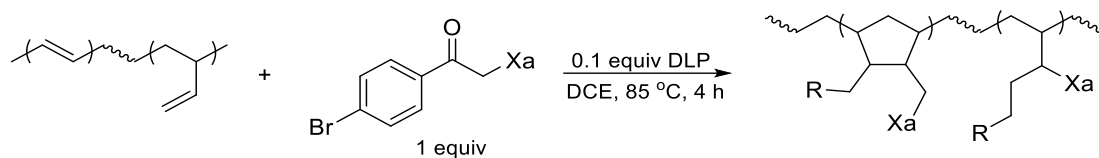
No	[DB]/[Xa]	grafting yield (%)	<i>f</i> (%)	vinyl (%)	vinylene (%)	Con. DB	<i>M<sub>n</sub></i> (k)	<i>M<sub>w</sub></i> (k)	<i>M<sub>w</sub></i> / <i>M<sub>n</sub></i>
1	12.3/1	48.1	3.9	16.0	60.7	5.8	5.0	8.95	1.79
2	6.2/1	40.1	6.4	17.6	>99	4.7	6.3	9.51	1.51
3	2.5/1	32.3	12.8	19.7	>99	2.5	6.5	8.91	1.37
4	1.2/1	27.0	21.4	23.6	>99	1.5	6.8	8.91	1.31

<sup>a</sup>Reaction conditions: 0.5 M PBD (double) in DCE, stirred at room temperature and ambient atmosphere, Et<sub>3</sub>B: 1 mol/L in THF. <sup>b</sup>Ratio of total double bonds to added xanthate. <sup>c</sup>Xanthate proportion grafted onto polymer. <sup>d</sup>Degree of functionalization: grafted xanthate to polymer segment ratio. <sup>e</sup>Conversion of vinyl group. <sup>f</sup>Conversion of backbone double bonds. <sup>g</sup>Average double bonds consumed per grafted xanthate. <sup>h</sup>GPC analysis in THF with PMMA standard.



**Figure S6.** GPC traces of the addition in Table S3 (THF GPC, PMMA as standard).

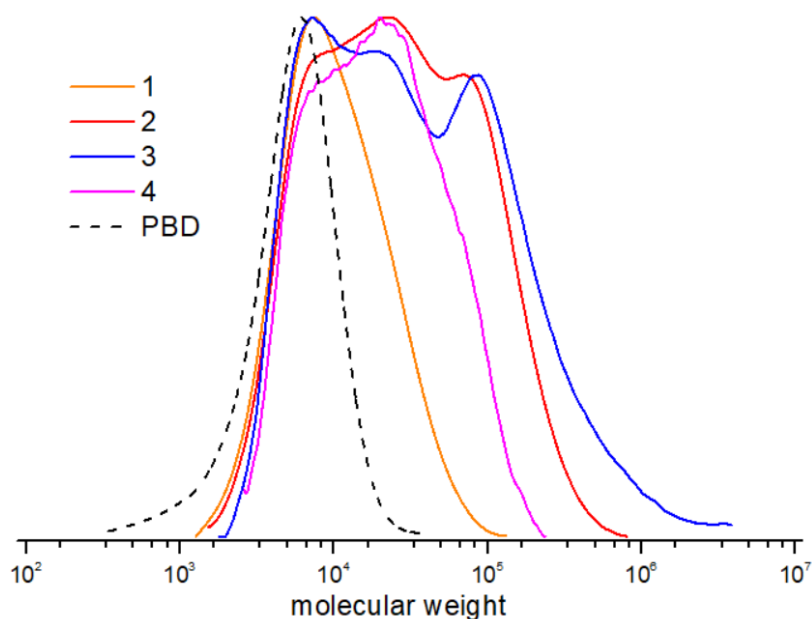
Addition reaction of **Xan-2** initiated by DLP (various dosage of Xan)



**Table S4<sup>a</sup>**

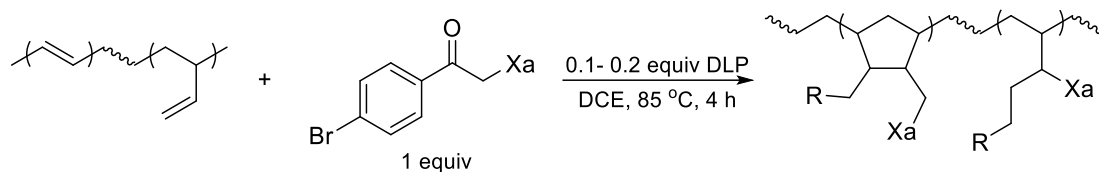
No	[DB]/[Xa]	grafting yield (%)	<i>f</i> (%)	vinyl (%)	vinylene (%)	Con. DB	<i>M<sub>n</sub></i> (k)	<i>M<sub>w</sub></i> (k)	<i>M<sub>w</sub></i> / <i>M<sub>n</sub></i>
1 <sup>a</sup>	6.9/1	20.0	2.6	15.6	17.5	6.1	7.4 <sup>b</sup>	14.43 <sup>b</sup>	1.95
2 <sup>a</sup>	2.7/1	19.2	7.0	31.7	21.8	4.3	12.9	49.67	3.85
3 <sup>a</sup>	1.5/1	19.4	12.3	52.7	51.1	4.3	14.2	81.22	5.72
4	0.4/1	14.0	35.0	82.0	61.1	2.3	12.1	31.10	2.57

<sup>a</sup>Reaction conditions: 0.5 M PBD (double bond) in DCE, heated at 85 °C for 4 h, 0.1 equiv. DLP (to Xan). <sup>b</sup>Ratio of total double bonds to added xanthate. <sup>c</sup>Xanthate proportion grafted onto polymer. <sup>d</sup>Degree of functionalization: grafted xanthate to polymer segment ratio. <sup>e</sup>Conversion of vinyl group. <sup>f</sup>Conversion of backbone double bonds. <sup>g</sup>Average double bonds consumed per grafted xanthate. <sup>h</sup>GPC analysis in THF with PMMA standard. <sup>i</sup>Dissolved part was taken for GPC analysis.



**Figure S7.** GPC traces of the addition in Table S4 (THF GPC, PMMA as standard).

Addition reaction of **Xan-2** initiated by DLP (various concentration of PBD)

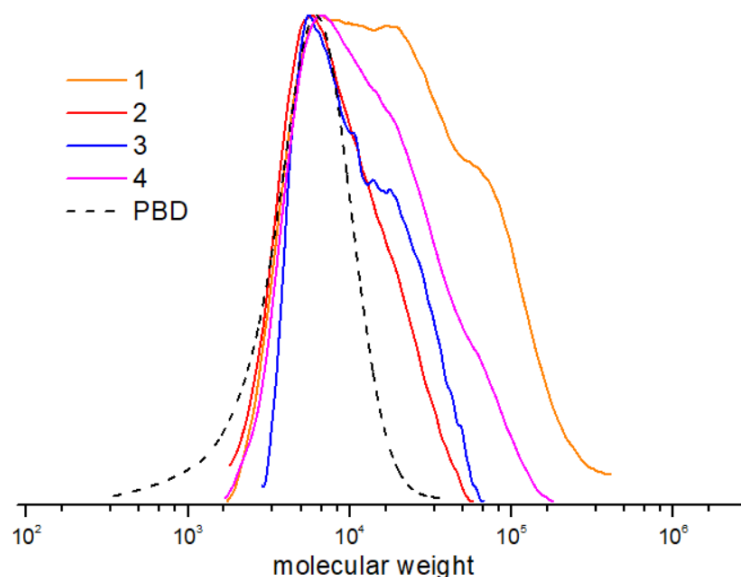


**Table S5<sup>a</sup>**

No	[DB]/[Xa]/[C]	grafting yield (%)	<i>f</i> (%)	vinyl (%)	vinylene (%)	Con. DB	<i>M<sub>n</sub></i> (k)	<i>M<sub>w</sub></i> (k)	<i>M<sub>w</sub>/M<sub>n</sub></i>
1 <sup>a</sup>	1.22/1 (0.05M)	16.0	13.0	62.5	36.1	4.50	10.5	38.96	3.71
2 <sup>a</sup>	1.27/1 (0.1M)	15.0	12.0	63.0	41.1	4.96	6.4	10.24	1.60
3 <sup>a</sup>	1.25/1 (0.25M)	16.0	13.0	62.3	33.1	4.43	8.4	14.78	1.76
4 <sup>a</sup>	1.22/1 (0.5M)	16.0	13.0	54.0	33.0	3.98	8.7	18.79	2.16
5 <sup>b</sup>	1.25/1 (0.05M)	18.0	14.4	92.5	84.2	6.33	/	/	/
6 <sup>b</sup>	1.22/1 (0.1M)	19.0	15.6	95.1	83.9	5.98	/	/	/
7 <sup>b</sup>	1.20/1 (0.25M)	19.0	15.8	92.2	78.6	5.71	/	/	/

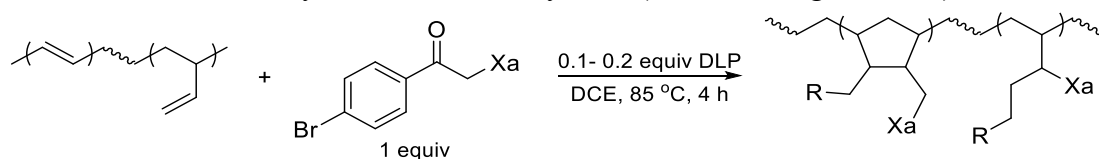
<sup>a</sup>Reaction conditions: heated at 85 °C for 4 h, entry 1-5: 0.1 equiv. DLP (to Xan), entry 6,7: 0.2 equiv. DLP.

<sup>b</sup>Concentration of PBD (double bond) in DCE. <sup>c</sup>Ratio of total double bonds to added xanthate. <sup>d</sup>Xanthate proportion grafted onto polymer. <sup>e</sup>Degree of functionalization: grafted xanthate to polymer segment ratio. <sup>f</sup>Conversion of vinyl group. <sup>g</sup>Conversion of backbone double bonds. <sup>h</sup>Average double bonds consumed per grafted xanthate. <sup>i</sup>GPC analysis in THF with PMMA standard. <sup>j</sup>Dissolved part was taken for GPC analysis. <sup>k</sup>Crosslinked gel was obtained finally.



**Figure S8.** GPC traces of the addition in Table S5 (THF GPC, PMMA as standard).

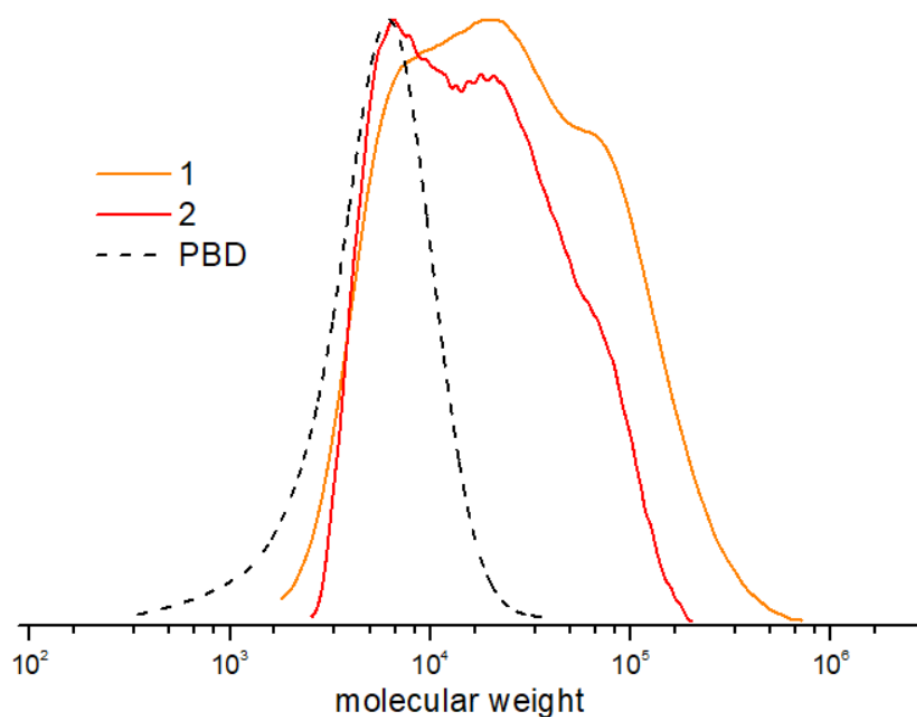
Modification of PBD by **Xan-2** initiated by DLP (various dosage of DLP)



**Table S6<sup>a</sup>**

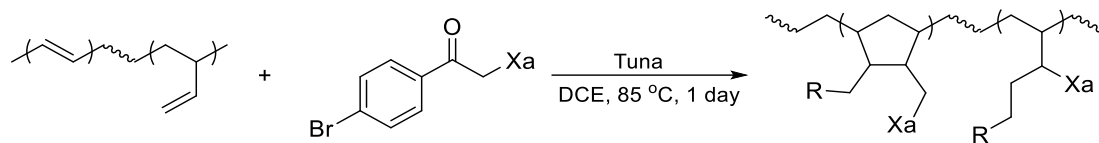
No	[DB]/[Xa]/[I]	grafting yield (%)	f (%)	vinyl (%)	vinylene (%)	Con. DB	$M_n$ (k)	$M_w$ (k)	$M_w/M_n$
1	1.33/1/0.05	13.0	9.8	39.8	30.3	3.92	12.4	46.50	3.75
2	1.33/1/0.1	20.0	15.0	59.3	38.5	3.75	11.1	25.53	2.30
3 <sup>a</sup>	1.33/1/0.2	29.1	21.8	78.1	42.2	3.34	/	/	/

<sup>a</sup>Reaction conditions: 0.5 M PBD (double bond) in DCE, heated at 85 °C for 4 h. <sup>b</sup>Ratio of total double bonds to added xanthate and DLP. <sup>c</sup>Xanthate proportion grafted onto polymer. <sup>d</sup>Degree of functionalization: grafted xanthate to polymer segment ratio. <sup>e</sup>Conversion of vinyl group. <sup>f</sup>Conversion of backbone double bonds. <sup>g</sup>Average double bonds consumed per grafted xanthate. <sup>h</sup>GPC analysis in THF with PMMA standard. <sup>i</sup>Crosslinked gel was obtained finally.



**Figure S9.** GPC traces of the addition in Table S6 (THF GPC, PMMA as standard).

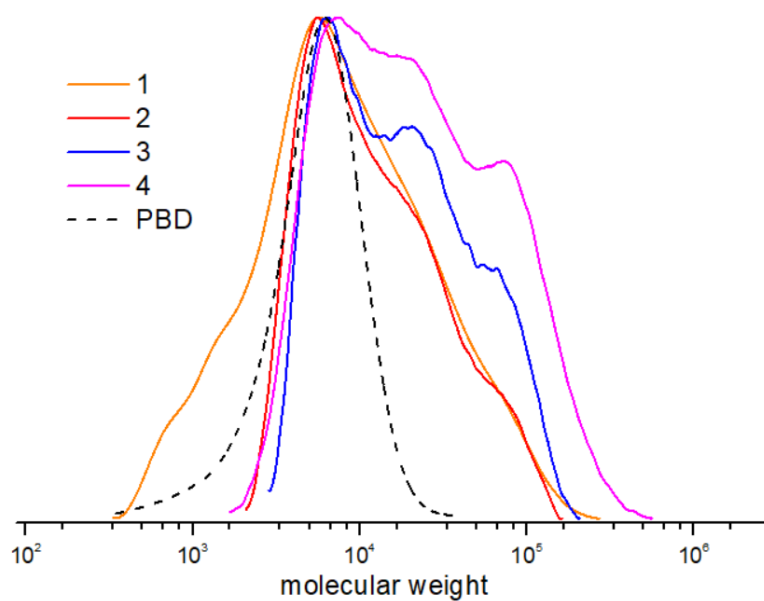
### Addition reaction of **Xan-2** initiated by blue light



**Table S7<sup>a</sup>**

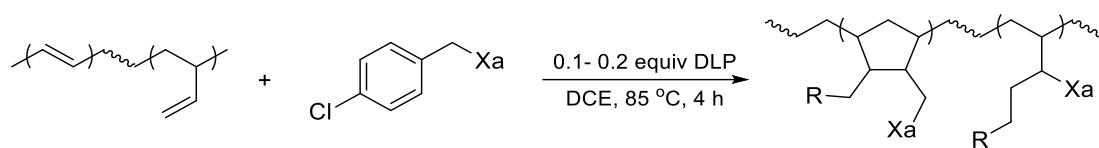
No	[DB]/[Xa] ]	grafting yield (%)	f (%)	vinyl (%)	vinylene (%)	Con. DB	$M_n$ (k)	$M_w$ (k)	$M_w/M_n$
1	11.0/1	93.4	8.5	31.9	29.7	3.7	4.4	17.29	3.93
2	5.3/1	46.2	8.7	53.2	31.1	5.7	8.2	18.61	2.27
3	2.5/1	38.2	15.0	61.4	28.6	3.8	11.1	29.30	2.64
4	1.2/1	22.1	17.2	68.8	35.1	3.7	11.4	43.89	3.85

<sup>a</sup>Reaction conditions: 0.5 M PBD (double) in DCE, degassed, irradiated at room temperature for 24 h. <sup>b</sup>Ratio of total double bonds to added xanthate. <sup>c</sup>Xanthate proportion grafted onto polymer. <sup>d</sup>Degree of functionalization: grafted xanthate to polymer segment ratio. <sup>e</sup>Conversion of vinyl group. <sup>f</sup>Conversion of backbone double bonds. <sup>g</sup>Average double bonds consumed per grafted xanthate. <sup>h</sup>Dissolved part was taken for GPC analysis in THF with PMMA standard.



**Figure S10.** GPC traces of the addition in Table S7 (THF GPC, PMMA as standard).

### Addition reaction of **Xan-3** initiated by DLP

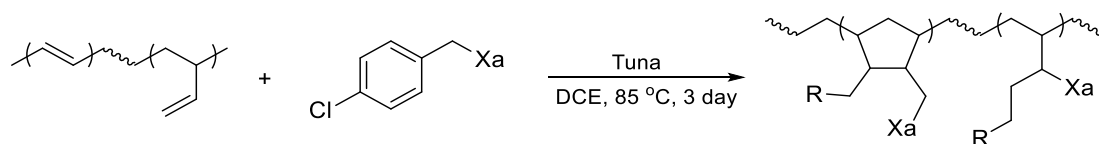


**Table S8<sup>a</sup>**

No	[DB]/[Xa]	grafting yield (%)	f (%)	vinyl (%)	vinylene (%)	Con. DB	$M_n$ (k)	$M_w/M_n$
1 <sup>a</sup>	14.0/1	0	0	0	0	/	/	/
2 <sup>a</sup>	7.2/1	0	0	< 1% <sup>c</sup>	< 1%	/	/	/
3 <sup>a</sup>	3.0/1	/	< 2%	< 2%	< 2%	/	/	/
4 <sup>a</sup>	1.4/1	/	< 5%	< 5%	< 5%	/	/	/
5 <sup>b</sup>	15.3/1	/	< 2%	< 5%	< 5%	/	/	/
6 <sup>b</sup>	7.2/1	/	< 3%	< 5%	< 5%	/	/	/
7 <sup>b</sup>	2.99/1	/	< 5%	< 5%	< 5%	/	/	/
8 <sup>b</sup>	1.4/1	3	2	4	3	1.9	5.7	1.30

<sup>a</sup>Reaction conditions: 0.5 M PBD (double bond) in DCE, heated at 85 °C for 4 h, entry 1-4: 0.1 equiv. DLP (to Xan), entry 5-8: 0.2 equiv. DLP. <sup>b</sup>Ratio of total double bonds to added xanthate. <sup>c</sup>Xanthate proportion grafted onto polymer. <sup>d</sup>Degree of functionalization: grafted xanthate to polymer segment ratio. <sup>e</sup>Conversion of vinyl group. <sup>f</sup>Conversion of backbone double bonds. <sup>g</sup>Average double bonds consumed per grafted xanthate. <sup>h</sup>GPC analysis in THF with PMMA standard.

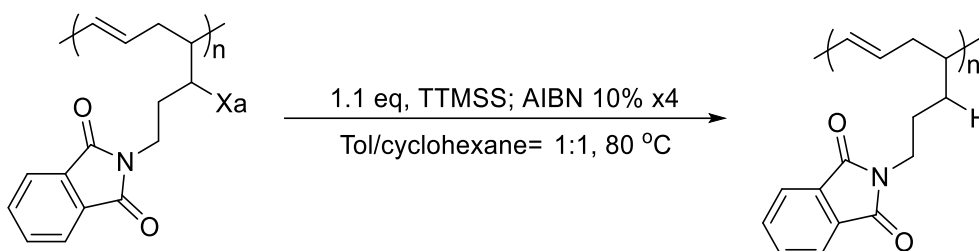
### Addition reaction of **Xan-3** initiated by Tuna



Blue light turned out to be not suitable for this modification. No conversion was observed and part of xanthate was damaged.

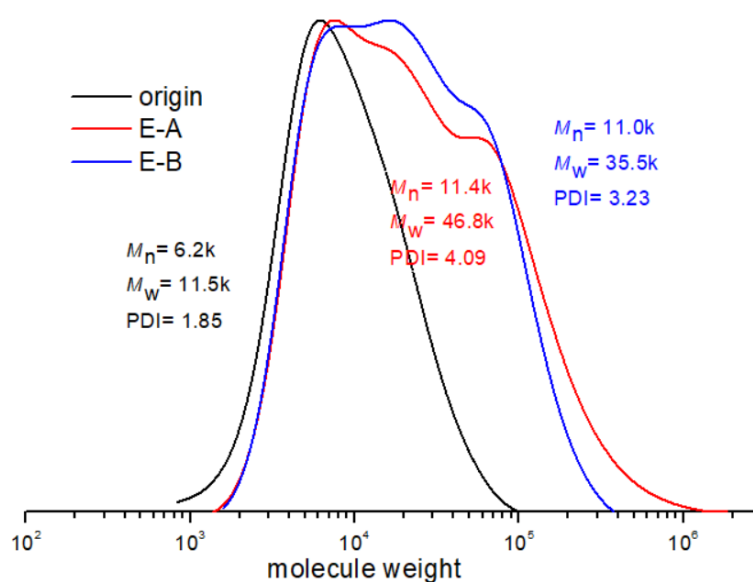
## Part two: The dexanthylation of functionazlied polybutadiene

### (1) Dexanthylation with tris(trimethylsilyl)silane



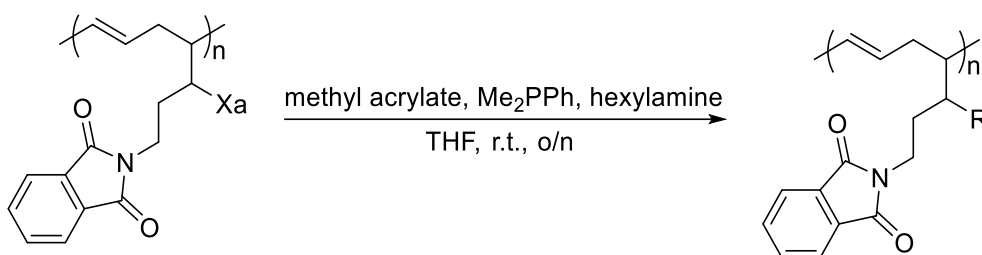
Experiment A (E-A): To a solution of **Xan-1** functionalized polybutadiene (100 mg) in mixed solvent (2 mL, Tol:cyclohexane = 1:1), excessive tris(trimethylsilyl)silane (248 mg, 1 mmol, TTMSS) and AIBN (16.4 mg, 0.1 mmol) were added. The solution was put at 80 °C, and AIBN (16.4 mg, 0.1 mmol) was added every one hour. After being stirred for 4 h (AIBN added for another 3 times), the solution was cooled down to room temperature and taken for analysis.

Experiment B (E-B): The steps are the same as above, but increase the usage of DCE to 6 mL.

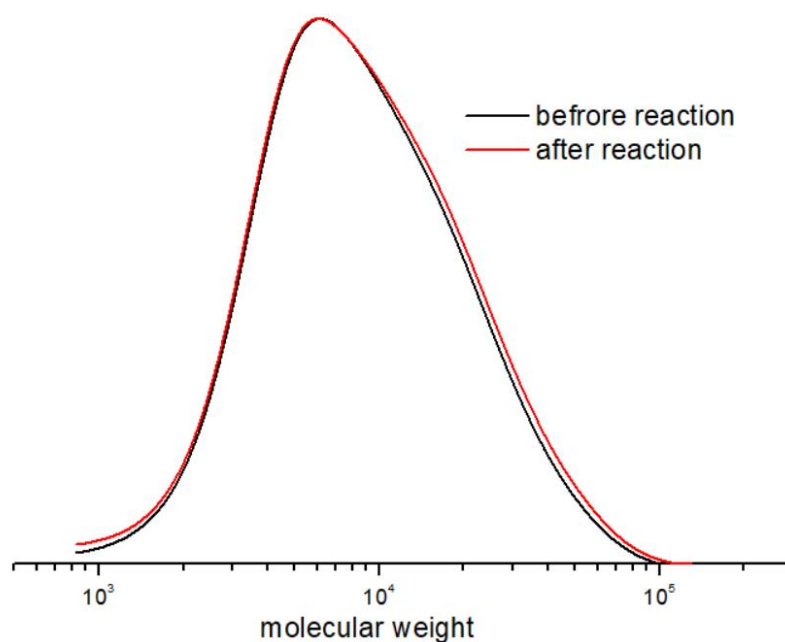


**Figure S11.** GPC traces of the product obtained from Experiment-A and Experiment-B (THF GPC, PMMA as standard).

(2) Dexanthylation by Aminolysis-Michael addition



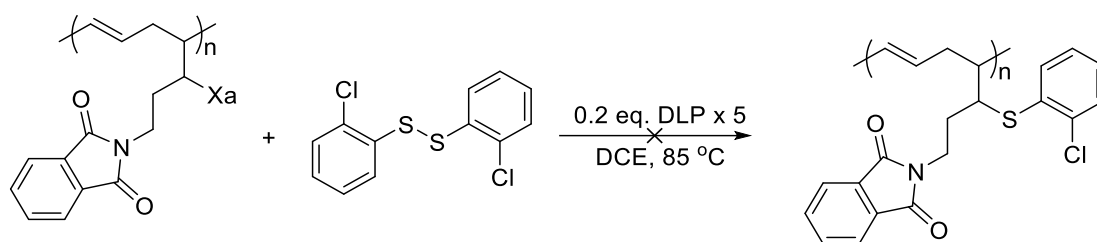
**Xan-1** functionalized polybutadiene (100 mg), hexylamine (50 mg, 0.5 mmol), dimethylphenylphosphine (0.69 mg, 0.005 mmol,  $Me_2PPh$ ) and methyl acrylate (43 mg, 0.5 mmol) were dissolved in 3 mL THF. The solution was stirred at room temperature for overnight. The polymer solution was then precipitated in MeOH, and this operation was repeated for 3 times. The obtained polymer was taken for NMR and GPC analysis.  $^1H$  NMR is shown in **4. NMR spectrum** and the GPC trace is below.



**Figure S12.** GPC traces of the polymer before and after dexanthylation (THF GPC, PMMA as standard).



### (3) Dexanthylation with *o*-chlorophenyl thioethers

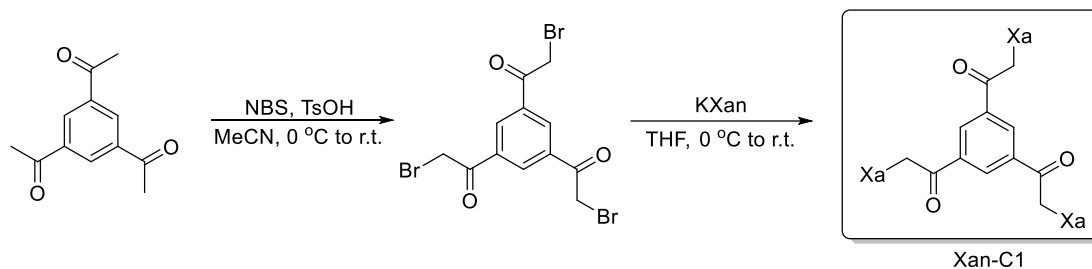


**Xan-1** functionalized polybutadiene (100 mg), *o*-chlorophenyl thioethers (72 mg, 0.4 mmol) and DLP (20 mg, 0.05 mmol) were dissolved in 3 mL DCE. The solution was deoxygenated by pumping into N<sub>2</sub> for 5 min, followed by being stirred at 85 °C. Another 0.05 mmol DLP was added each one hour until the reaction was completed in 6 hours. The colour of solution changed from colorless to orange at last. The solution was precipitated in MeOH to obtain product as brown powder.

<sup>1</sup>H NMR is shown in **4. NMR spectrum**.

### Part three: Crosslinker for PBD

#### 1. Synthesis of crosslinker **Xan-C1**



1,3,5-triacetylbenzene (2.04 g, 10 mmol) and *p*-toluenesulfonic acid (8.6 g, 50 mmol) were dissolved in 30 mL MeCN. The solution was cooled to 0 °C, and then *N*-bromosuccinimide (5.87 g, 33 mmol) was added slowly. After being stirred for overnight at ice-bath, the solution was evaporated to remove the MeCN. The obtained white solid was extracted by EA/H<sub>2</sub>O, and organic was evaporated to get crude product as white powder.

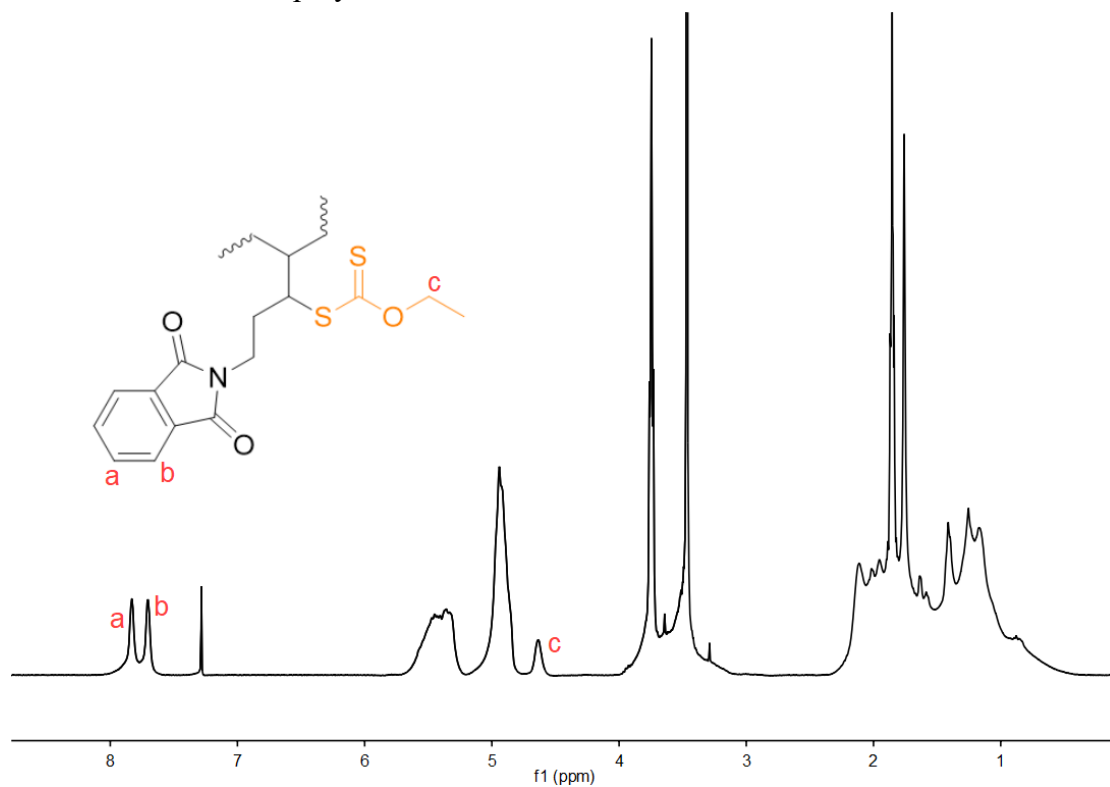
Obtained crude product was dissolved in 80 mL THF, and the solution was cooled to 0 °C. Added potassium ethylxanthate (4.8 g, 350 mmol) slowly and stirred the suspension at ice-bath for 3 h. Remove the acetone in vacuo and extract the mixture by EA/H<sub>2</sub>O. Recycle the EA layer and dry by Na<sub>2</sub>SO<sub>4</sub>. (Do not use MgSO<sub>4</sub>). The target product was obtained as white (4.79 g, 85%).

#### 2. Crosslinking reaction

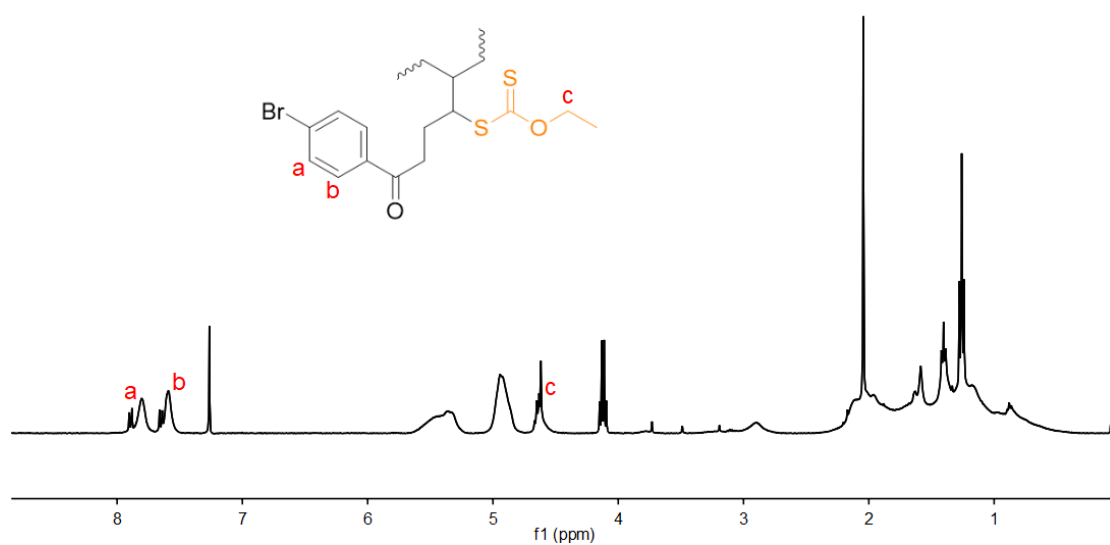
A model crosslinking reaction could be conducted as below. In a transplant 5 mL vial equipped with a stirring bar was charged with polybutadiene (200 mg) and **Xan-C1** (20 mg) in 3 mL DCE. The vial was sealed and stirred under the Kessil Tuna blue light for overnight. A fan was used to cool down the lamp and vial.

### 3. NMR spectrum

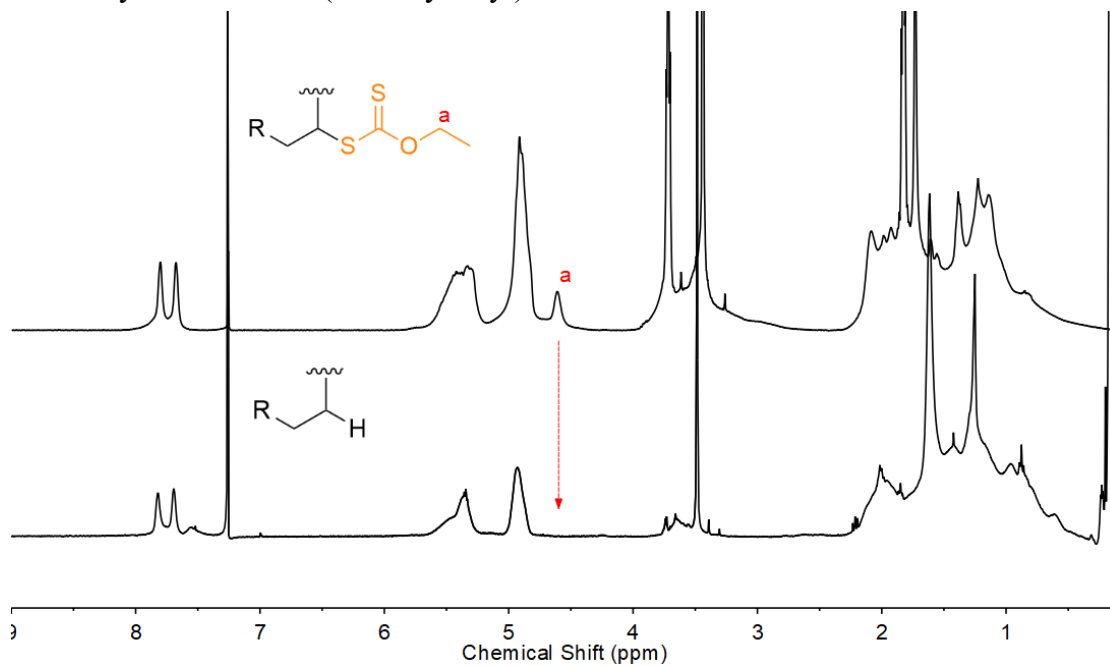
#### Xan-1 functionalized polybutadiene



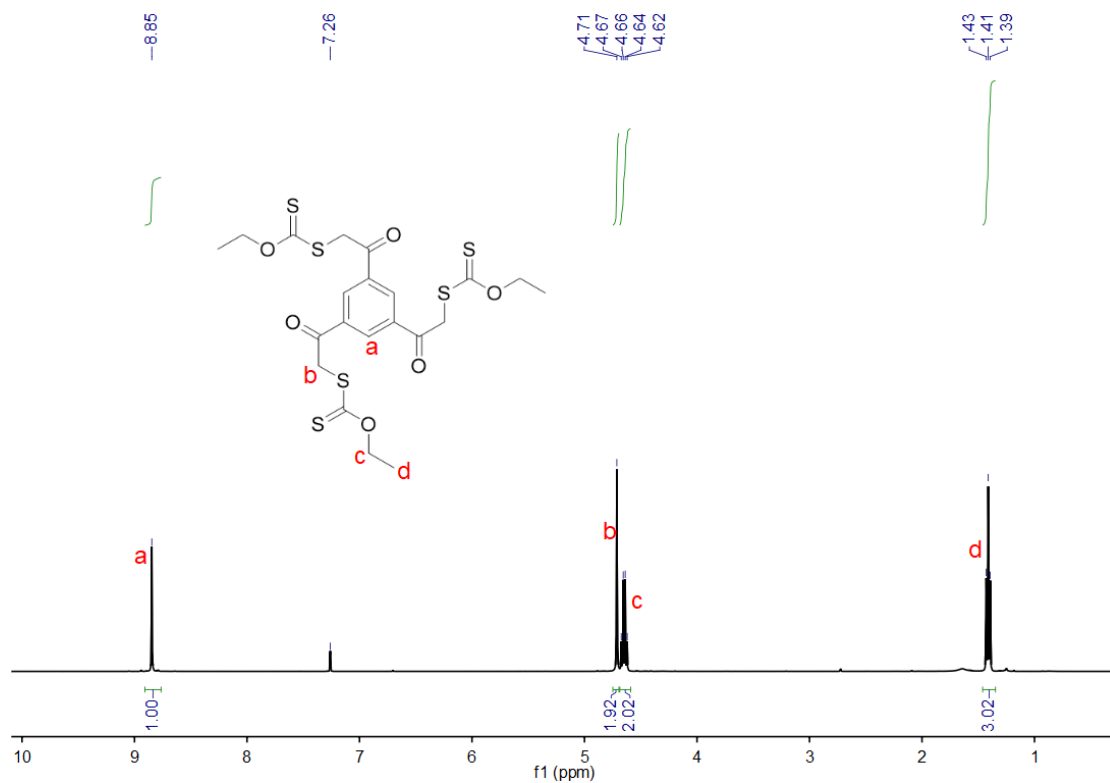
#### Xan-2 functionalized polybutadiene



### Dexanthylation with tris(trimethylsilyl)silane



### <sup>1</sup>H NMR spectrum of Xan-C1



<sup>13</sup>C NMR spectrum of Xan-C1

