Supporting information

A fast and mild closed-loop recycling of anhydride-cured epoxy through microwave-assisted catalytic degradation by trifunctional amine and subsequent reuse without separation

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Number of pages: 9

Number of figures: 11

1. **Degradation mixture reused to prepare new epoxy materials**.

Table S1 The preparation formulation of the new epoxy material.

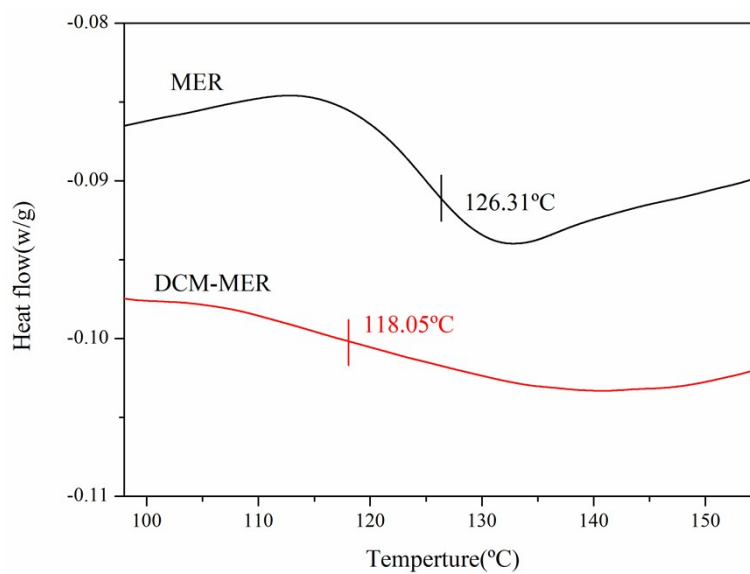| sample	Degradation mixture(g)	Uncured epoxy(g)
9 wt% rER	1.0	10
17 wt% rER	2.0	10
23 wt% rER	3.0	10
30 wt% rER	4.3	10
40 wt% rER	6.7	10

2. **Characterization of DCM-MER**

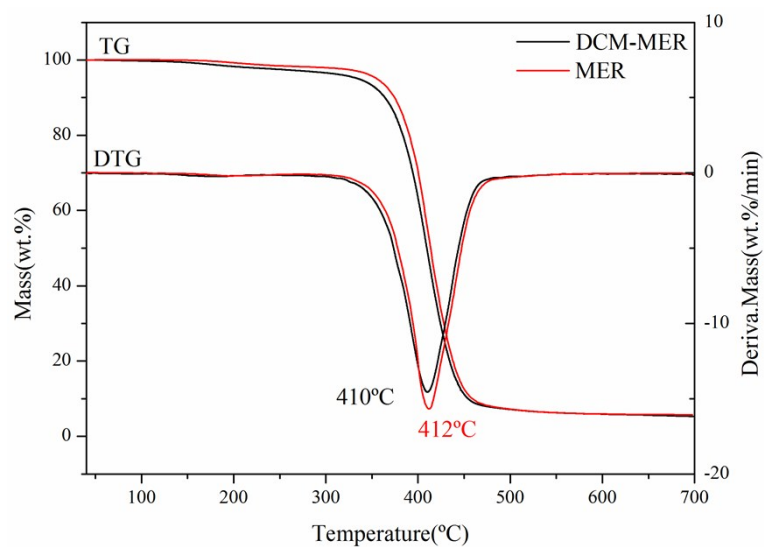Figure S1 DSC curves of MER and DCM-MER.

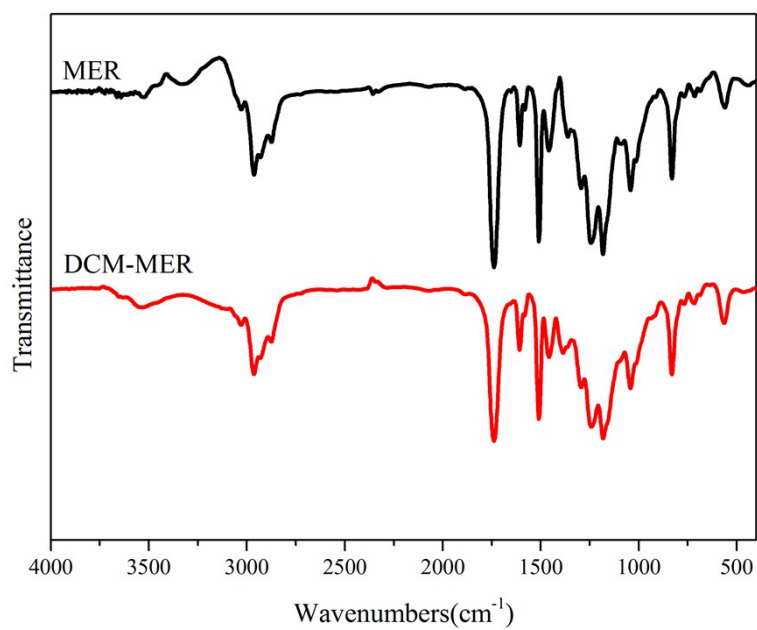Figure S2 TG curves of MER and DCM-MER

Figure S3 FTIR spectra of MER and DCM-MER.
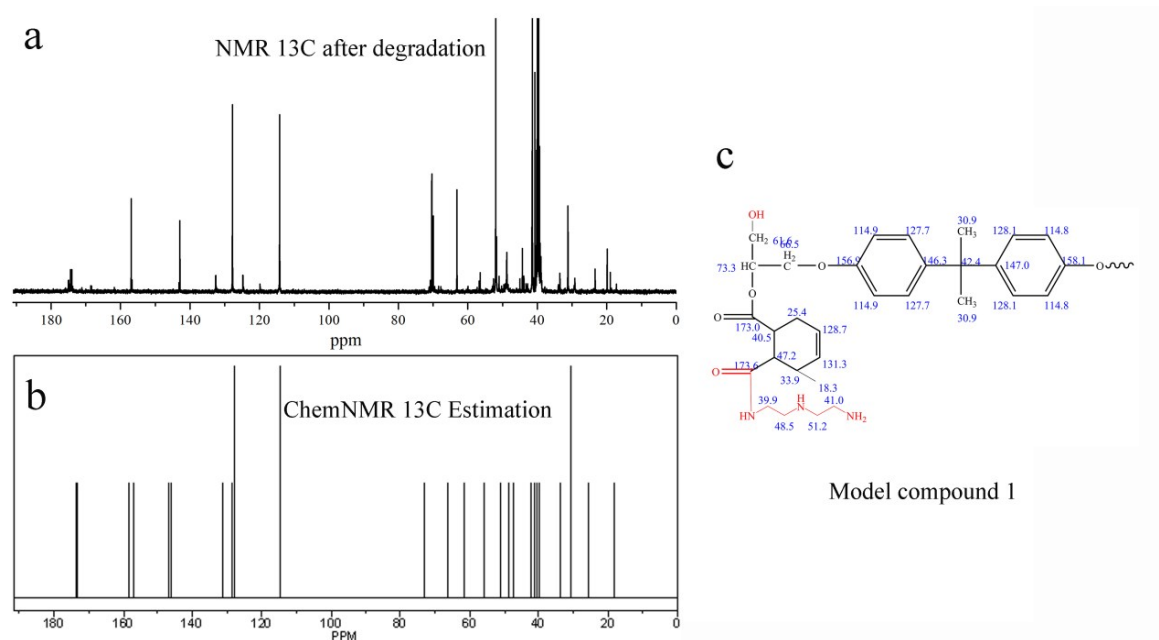
3. Characterization of DER

Figure S4 (a)^{13}C-NMR spectrum of actual DER , (b)simulated 13C-NMR spectrum of possible DER and(c) Possible structure of DER

Table S2 Elemental analysis of MER before and after degradation

Sample	Carbon (wt %)	Hydrogen (wt %)	Nitrogen (wt %)
Before degradation	70.26	6.94	0
After degradation	66.61	8.03	1.79

Reaction conditions: ratio of DCM-MER to DETA(w/w)=2/5, 130°C, 50 min.

4. Determination of active hydrogen

The total amine concentration C_{total}(mmol/L) in the degradation solution is determined by acid-base titration according to the equation (1). The degradation solution was diluted with ethanol to get a 0.367 g/L degradation solution-ethanol solution. 10.06 mmol/L HCl solution calibrated before was used to titrated the degradation solution-ethanol solution using bromocresol blue as an indicator.

$$C_{total} = \frac{C_{HCl}V_{HCl}}{V_{degradation}} \quad (1)$$

Where V_{HCl} and $V_{degradation}$ are the volume (L) of consumed HCl and volume (L) of degradation solution-ethanol solution respectively. C_{HCl} is the concentration of HCl (mmol/L).

The method for determining the amino group content is mainly based on the reaction of primary amine groups with aldehyde groups[1, 2]. The reaction produces water and thus the primary amine content can be obtained by measuring the water content. Mix 10 mL of 0.367 g/L degradation solution-ethanol solution and 3 mL of benzaldehyde at 60 °C water bath for 30 min, then naturally cool it down to room temperature and dilute with ethanol to 100 mL. For blank control, 10 mL of ethanol was used instead of the degradation solution-ethanol solution according to the above procedure. Finally, the water content was measured using a Karl Fischer moisture analyzer (ZDJ-2S,China) to obtain a primary amine concentration $C_{primary}$ according to the equation (2).

$$C_{primary} = \frac{(C_{H_2O} - C'_{H_2O})V}{V_{degradation}} = \frac{(C_{H_2O} - C'_{H_2O}) * 0.1 \, L}{0.01 \, L} \quad (2)$$

Where C_{H2O} and C'_{H2O} are the water content in mix solution and blank solution (mmol/L), $V_{degradation}$ and V are the original volume and diluted volume of degradation solution-ethanol solution respectively (L).

Active hydrogen concentration C_H (mol/g) in the degradation solution is calculated according to the equation (3)

$$C_H\left(\frac{mol}{g}\right)$$
$$= \frac{C_{primary}V_{degradation} * 2 + (C_{total} - C_{primary})V_{degradation}}{C_{degradation}V_{degradation}} = \frac{C_{primary} + C_{tot}}{C_{degradation}}$$

(3)

Where $V_{degradation}$ are the original volume of degradation solution-ethanol solution (L).

The total amine concentration C_{total} was calculated as 2.89 mmol/L. The primary amine concentration $C_{primary}$ was calculated as 1.83 mmol/L. Thus, the concentration of active hydrogen was 0.0128 mol/g. That is, 1.28 mol of active hydrogen per 100 g of the degradation solution.
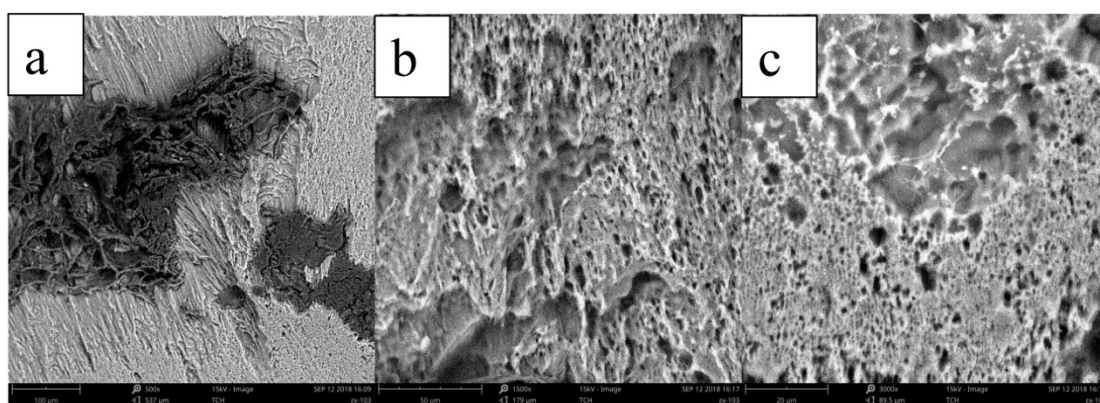
5. Characterization of ERr

Figure S5 SEM images of ERr. a: ×500, b: ×1500 c: ×3000. (130℃,50 min, ratio of DCM-MER to DETA(w/w)=2/5)
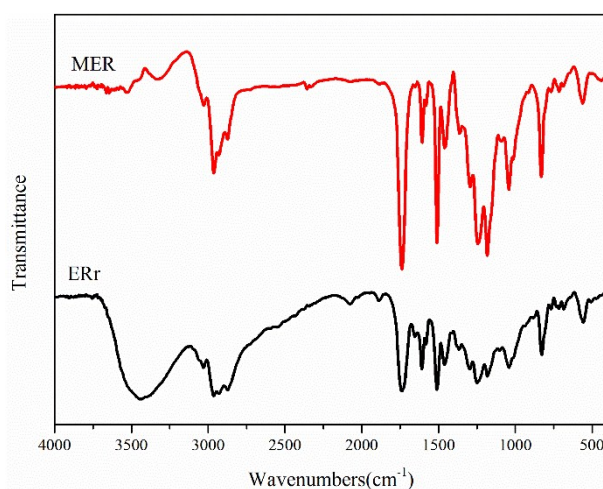
Figure S6 FTIR spectra of ERr obtained and acidified ERr. (130℃,50 min, ratio of DCM-MER to DETA(w/w)=2/5)
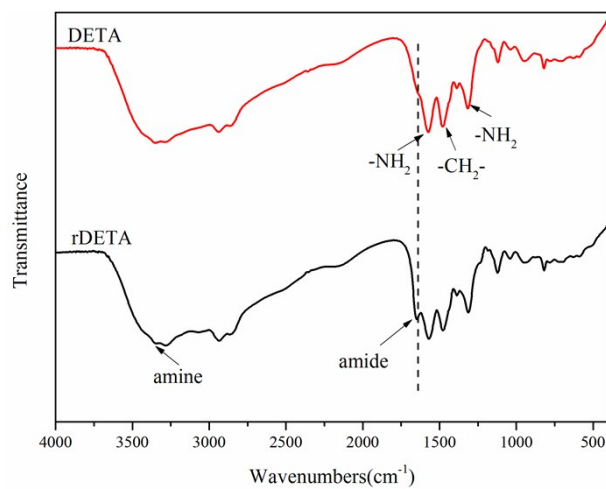
6. Characterization of rDETA

Figure S7 FTIR spectra of rDETA. (130 ºC,50 min, ratio of DCM-MER to DETA(w/w)=2/5)
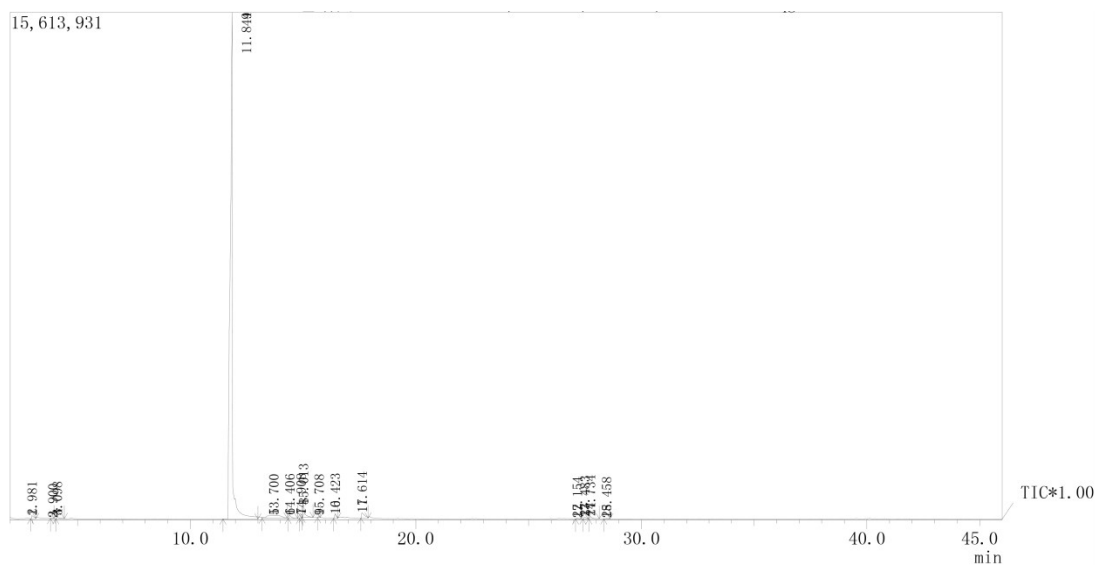

Figure S8 GC-MS analysis of rDETA. (130 ºC,50 min, ratio of DCM-MER to DETA(w/w)=2/5)
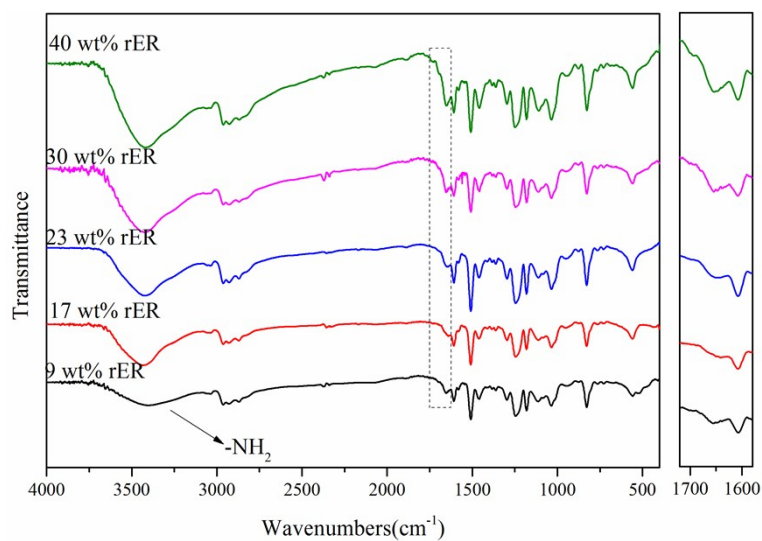
7. characterization of rER

Figure S9 FTIR spectra of the rER with different contents of degradation solution.
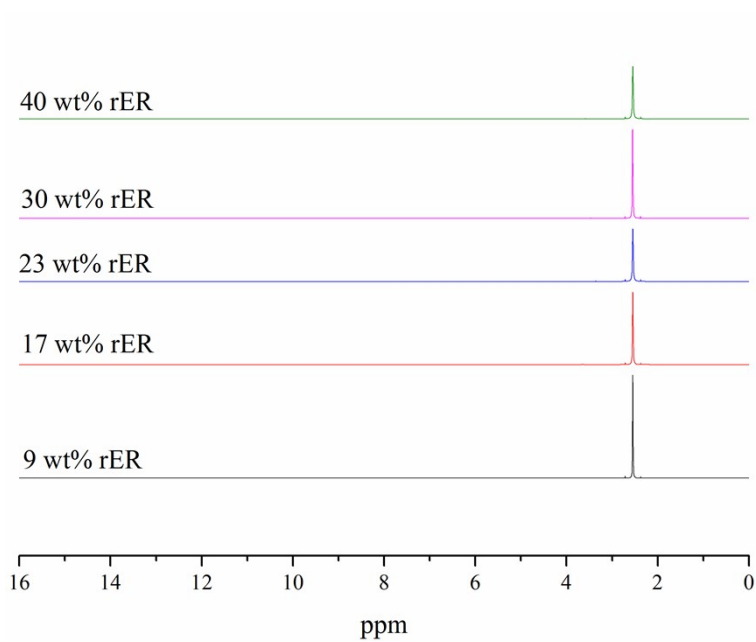

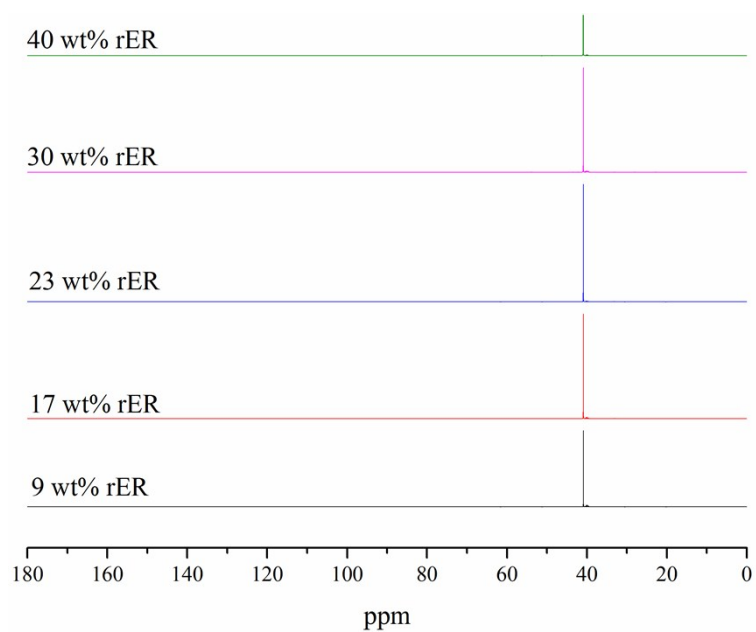Figure S10 ^{1}H-NMR of DMSO extract of rER resin for 10 days

Figure S11 ^{13}C-NMR of DMSO extract of rER for 10 days

1. J. Mitchell, W. Hawkins and D. M. Smith, *Journal of the American Chemical Society*, 2002, **66**.
2. J. E. Jackson, *Analytical Chemistry*, 1953, **25**, 1764-1765.