

Supporting information

Preparation and Characterization of a Multifunctional Epoxy Structural Adhesive with Flexibility, Damping and Durability

S I. The amine value titration

1. Preparing perchloric acid - acetic acid standard solution (0.01mol/L)

Firstly, measured 8.7 ml of perchloric acid into 500 ml glacial acetic acid. Then 20 ml acetic anhydride slowly poured into the solution. Finally, added glacial acetic acid to the mixture solution to 1000 ml. Shook it to a homogeneous liquid and left overnight.

Put 0.15g (accurate to 0.0001) of Potassium acid phthalate to 50 ml of glacial acetic acid, shook to dissolve completely. Then added 3~4 drops of methyl violet indicator to the mixture. The end of the titration is when the color changes from purple to dark blue.

According to equation (1) to calculate concentration of the standard liquid:

$$C = \frac{m}{V * 0.2042} \quad (1)$$

Where m is weight of Potassium acid phthalate, V is volume of the standard liquid, and 0.2042 is on behalf of the mass of $C_8H_5KO_4$ in 1ml standard solution.

Table 1 The values of perchloric acid - acetic acid standard solution concentration titration

	1	2	3	Average value
$m_{C_8H_5KO_4}/g$	0.1523	0.1520	0.1505	0.1516
$V_{\text{the standard solution}}/ml$	7.11	7.03	7.00	7.05
$C_{\text{the standard solution}}/mol * L^{-1}$	0.1049	0.1059	0.1053	0.1054

2. Titration of the amine value of modified curing agent (MCA)

Measured 0.35g (accurate to 0.0001) of MCA to 50 ml of glacial acetic acid, shook to a homogeneous solution. Then 3~4 drops of methyl violet indicator was installed into the solution. The end of the titration is when the color changes from purple to dark blue.

According to equation (2) to calculate the amine value of MCA:

$$AN(\text{mgKOH/g}) = \frac{CV * 56.1}{m} \quad (2)$$

Where C is representative of concentration of perchloric acid - acetic acid standard solution, V is volume of the standard liquid, 56.1 represents the mass of KOH per mole and m is weight of MCA.

Table 2 The values of amine value titration

	1	2	3	Average value
m_{MCA}/g	0.3408	0.3549	0.3814	0.3590
$V_{\text{the standard solution}}/\text{ml}$	7.34	7.51	8.32	7.72
$AN/\text{mg KOH} \cdot \text{g}^{-1}$	127.35	125.12	128.99	127.15

S II. Research on Curing Process

In order to explore the cure temperature, used five different heating rate 5K/min · 10K/min · 15K/min · 20K/min and 25K/min and found their temperature at onset, peak, and end. By linear fitting, the curing temperature was finally obtained. And set the 80 °C as the curing temperature.

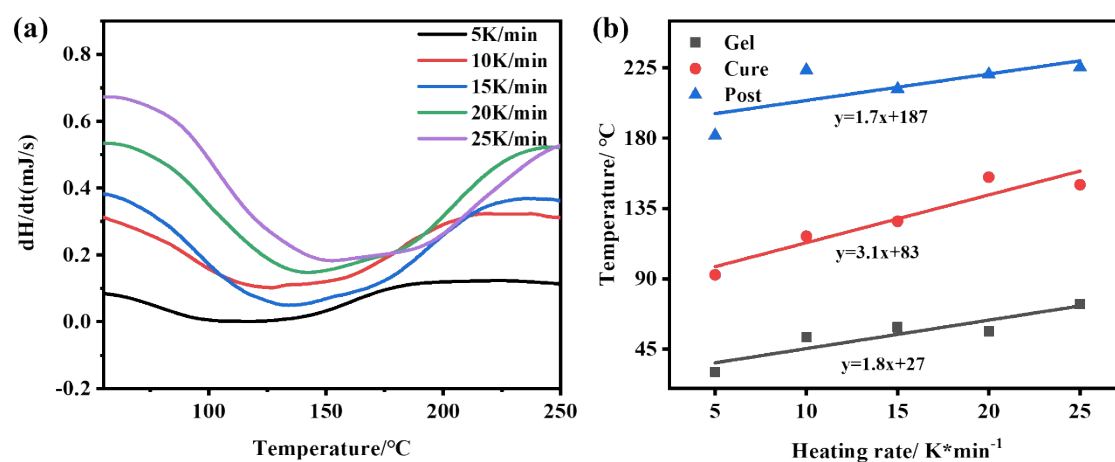


Figure S1. Exploring the curing process, (a) Exothermic behavior of curing at different heating rates; (b) Exploring the gel, pure and post temperature.

S III. Mass Spectrum (MS)

Using MS to measure the molecular of MCA. According to MS of D-230, D-400 and MCA, the degree of polymerization of MCA is 2 or 3.

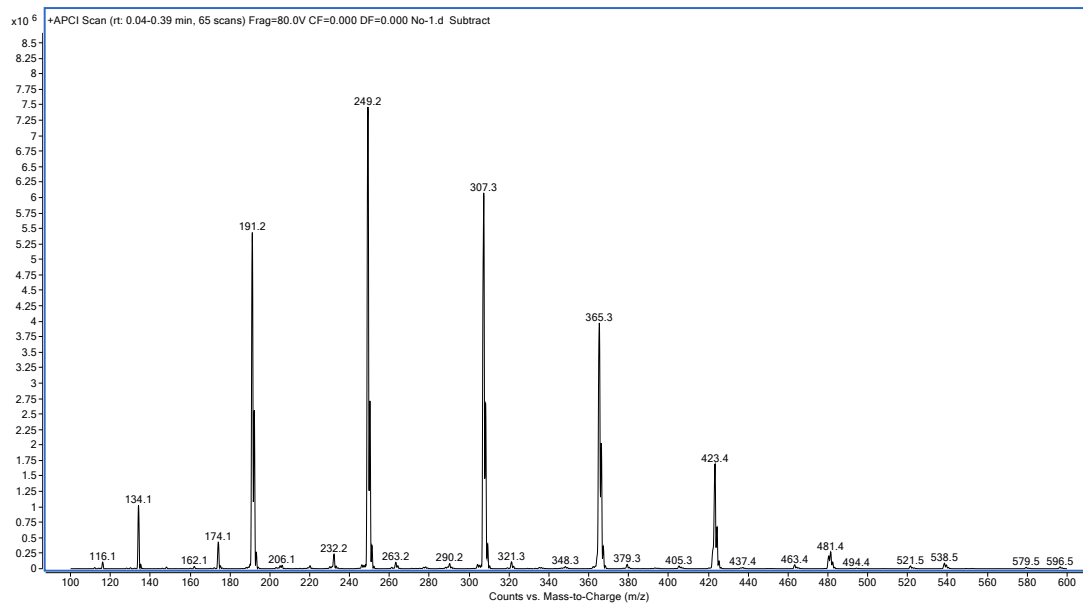


Figure S2. MS of D-230

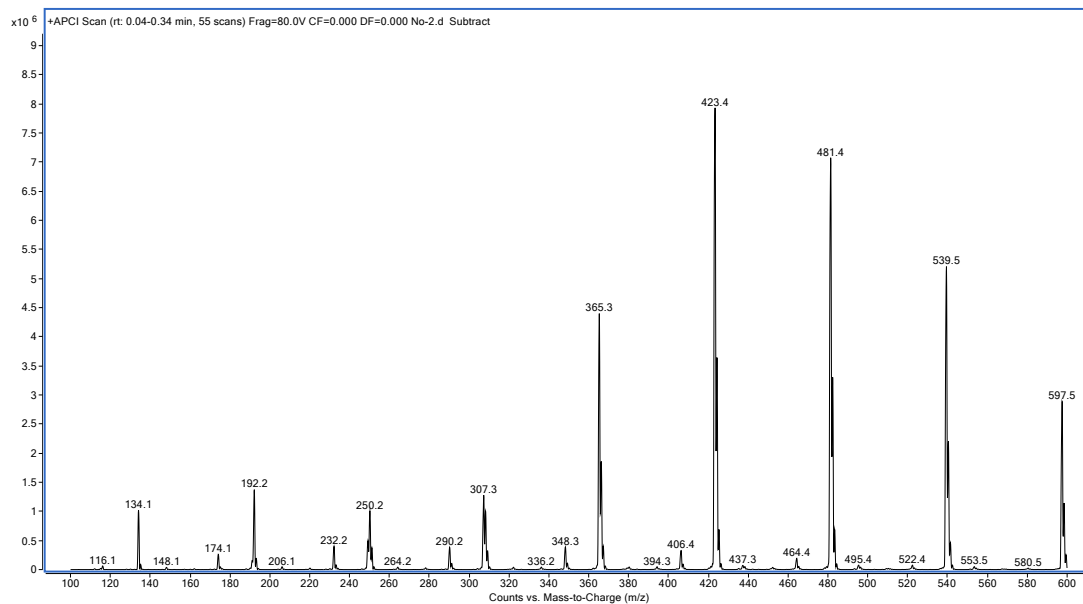


Figure S3. MS of D-400

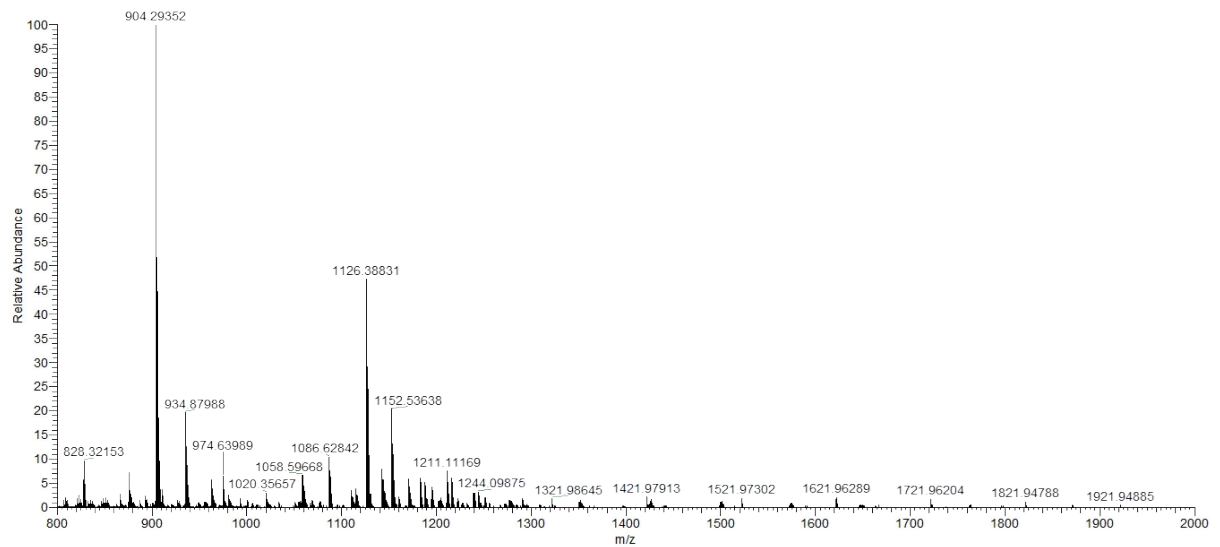


Figure S4. MS of MCA

S IV. Preparing specimens of Lap Shear Measurement

At first, polished the aluminum substrates (25mm*100mm*2mm) at 600 and 1200 mesh to remove the oxide layer on the surface, and put them into acetone. Then, ultrasonic cleaning was performed for 30 minutes to remove surface impurities. Next, the aluminum plates were dried and coated KH-500, dried again. Finally, DGEBA and modified curing agent were mixed fully with a ratio of 1:1.1~1.2. And epoxy adhesive was coated on the 12.5mm wide aluminum substrates. A thin adhesive layer was applied on both substrates and overlap was hold with two binder clips, as shown in Figure S2. The curing condition are 48h@80°C in a convection oven.

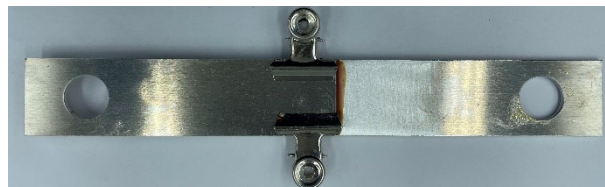


Figure S5 Lap shear specimen.

S V. The photos of experimental procedure.

To give a better visualization of the experimental procedure, some photos of the experimental process are attached.

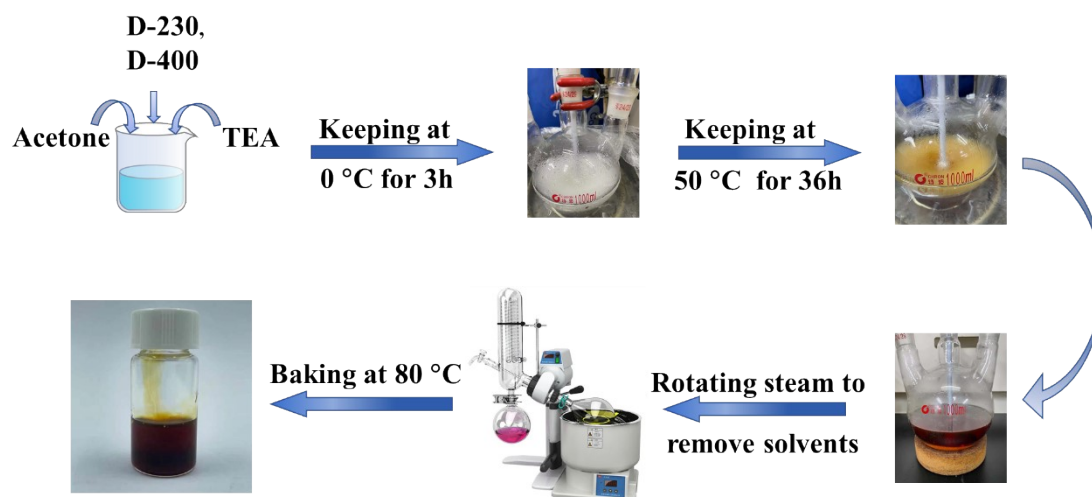


Figure S6. The photos of experimental procedure.

S VI. Using swelling method to measure the cross-linking density.

We have tested the mass changes of four epoxy resins using the swelling method, and the swelling of epoxy is expressed as a mass change in %. Details as follows:

$$R_m = \frac{m_c - m_0}{m_0} * 100$$

Where R_m is on behalf of the swelling of epoxy resin, and m_c and m_0 are denote the mass of epoxy at constant dissolution and at the beginning, respectively.

As shown in Table S1. and Figure S7, the change trend of the swelling ratio of epoxy resins is similar to the change trend of DMA. These two different measurements all tested the cross-linking density of MCA-cured epoxy is the minimum.

Table S1 The swelling ratio of epoxy resin

m_0	m_c	$R_m/\%$
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D-230	0.111	0.150	36.36
D-230:D-400=1:1	0.105	0.141	41.00
D-400	0.120	0.166	38.33
MCA	0.118	0.199	66.67

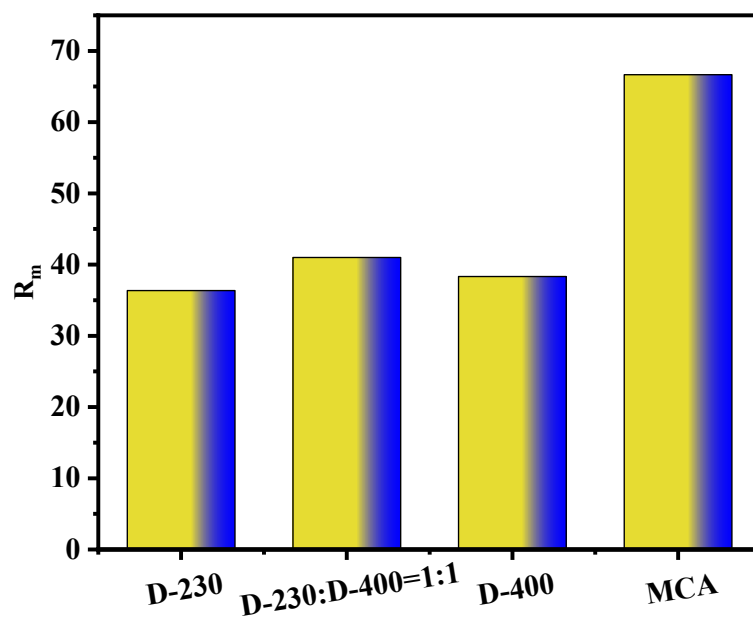


Figure S7. The swelling ratio of four different epoxy resin.