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Preparation of aramid-based epoxy resin from low-grade aramid

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Aramid powder preparation

Aramid used in the reactions were low-grade aramid fibers that were generated in the industrial production, mainly including low molecular weight aramid, N-methyl pyrrolidone, sulfuric acid and salt. 150 g of aramid fibers were added to 300 mL water, stirred for 0.5 h, and then filtered. The filter cake continued to be washed with water until the filtrate was neutral. The filtrate then dried at 100°C to constant weight, grinded with a mortar, passed 40 mesh sieves, sealed, and stored.

Characterization

Refer to Standard "GB/T 1632-93 Polymer Dilute Solution Viscosity and Intrinsic Viscosity Determination" to determine the viscosity of aramid. 0.125 g of low-grade, middle-grade, and high-grade aramid powders were dried separately to constant weight and dissolved in 25 mL concentrated sulfuric acid. Ubbelohde viscometer was utilized to measure the weight of aramid powders at 30 ±0.5°C. In the meanwhile, the viscosities of low-grade, middle-grade, and high-grade aramids were measured at 4~6 concentrations under same conditions.

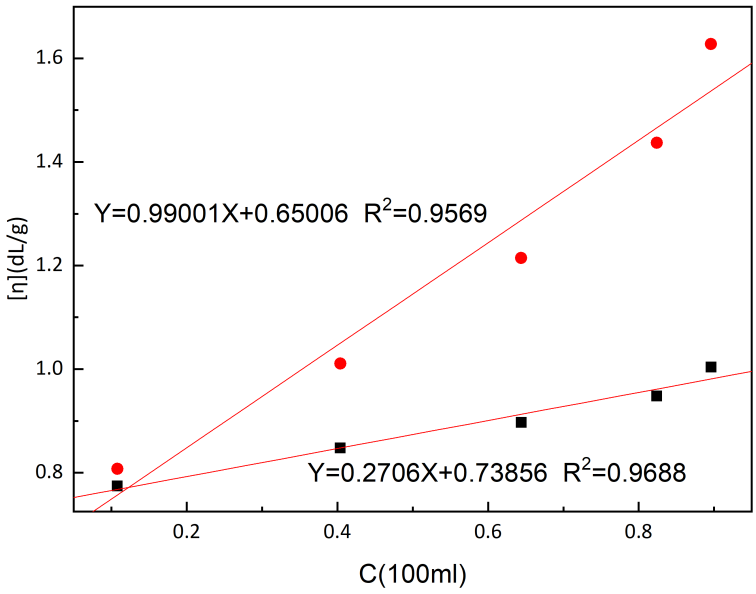


Fig. S1 Intrinsic viscosities of high-grade aramid

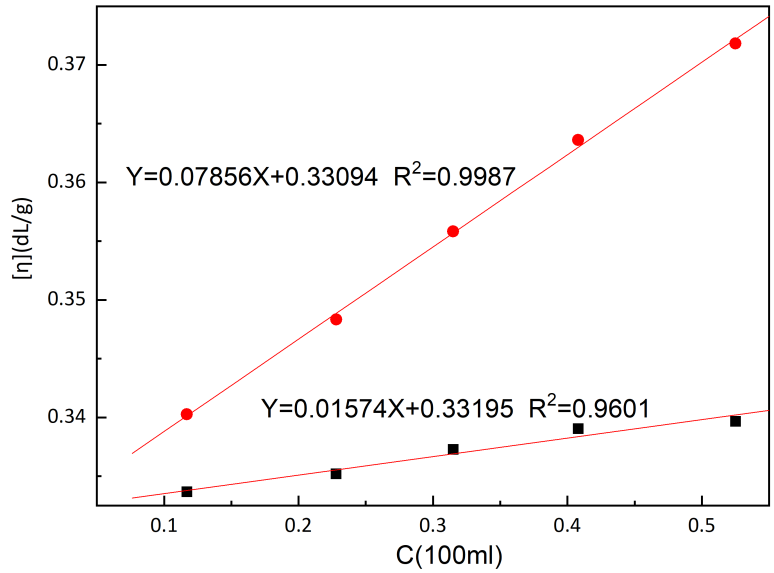


Fig. S2 Intrinsic viscosities of middle-grade aramid

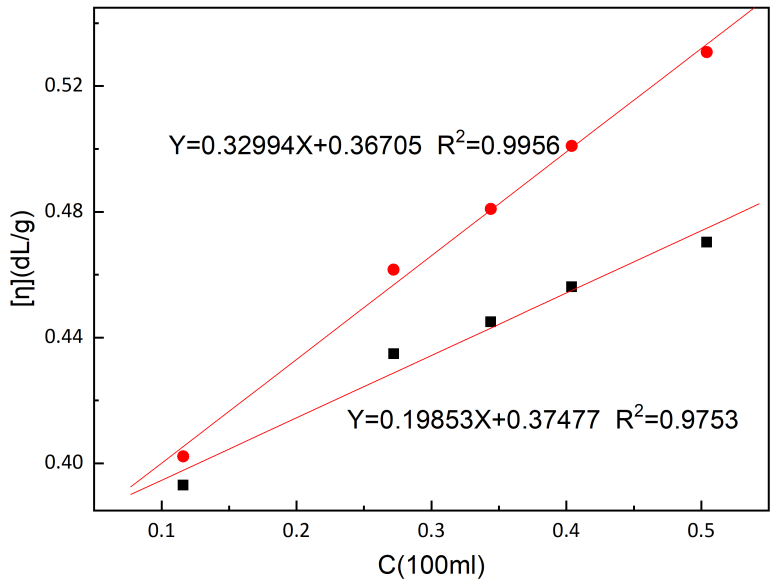
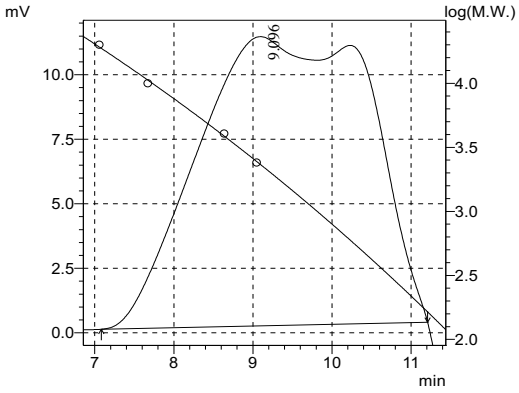


Fig. S3 Intrinsic viscosities of low-grade aramid

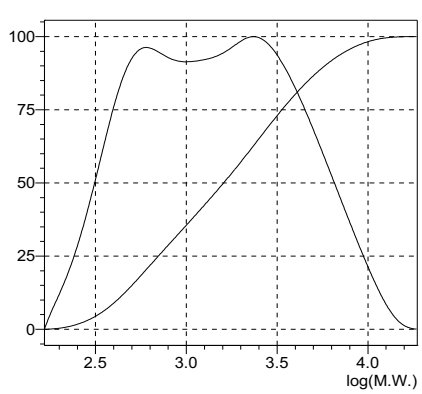
==== Shimadzu LabSolutions GPC Analysis Report ====

Acquired by	: System Administrator
Sample Name	: 8
Sample ID	: W8
Vail#	: 8
Injection Volume	: 60 uL
Data Filename	: 6.23_2021623_008.lcd
Method Filename	: 2020.9.5-fangfa.lcm
Batch Filename	: 6.23.lcb
Report Filename	: DEFAULT.lsr
Date Acquired	: 2021/6/23 13:55:49
Data Processed	: 2021/6/23 14:10:49

Chromatogram & Calibration Curve



Molecular Weight Distribution Curve



GPC Calculation Results

Peak#:1 (检测器A Ch1)
[峰信息]

	Time(min)	Volume(mL)	Molecular Weight	Height
Start	7.083	7.083	18743	137
Top	9.096	9.096	2320	11207
End	11.208	11.208	164	411

Area : 1703805
Area% : 100.0000

[平均分子量]

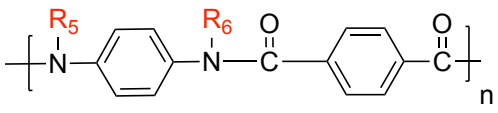
Number Average Molecular Weight(Mn)	1000
Weight Average Molecular Weight(Mw)	2465
Z Average Molecular Weight(Mz)	4845
Z+1 Average Molecular Weight(Mz1)	7034
Mw/Mn	2.46547
Mv/Mn	0.00000
Mz/Mw	1.96568

检测器A Ch1
[平均分子量(总计)]

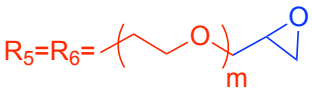
Number Average Molecular Weight(Mn)	1000
Weight Average Molecular Weight(Mw)	2465
Z Average Molecular Weight(Mz)	4845
Z+1 Average Molecular Weight(Mz1)	7034
Mw/Mn	2.46547
Mv/Mn	0.00000
Mz/Mw	1.96568

Fig. S4 Molecular weight of aramid-based epoxy resin

We estimated the structure of aramid-based epoxy resin by the determination results of hydroxyl value and the number average molecular weight. According to the experiments, the hydroxyl value of the ethoxylated modified aramid was 276.05 mg KOH/g and the number average molecular weight of aramid-based epoxy resin was 1000. We thus calculated the average functionality of the product is about 5 ($\frac{276.05\times1000}{56.1\times1000} \approx 4.92$) . We estimated the structures of our synthesized aramid-based epoxy resin in the following cases:



low-grade aramid-based epoxy resin



n= 2~5

m= 1~3

The possible percentage of aramid content is around 43%-70% after our calculation based on the experimental hydroxyl value and the number average molecular weight.

If n=2, m=1, k=3, Mw= 2*236+ 44*3 + 57*3+1 =776 61%

If n=2, m=2, k=3, Mw= 2*236+ 44*3*2 + 57*3+1 =908 52%

If n=3, m=1, k=5, Mw= 3*236+ 44*5 + 57*5+1 =1214 58%

If n=3, m=2, k=5, Mw= 3*236+ 44*5*2 + 57*5+1 =1434 49%

If n=3, m=3, k=5, Mw= 3*236+ 44*5*3 + 57*5+1 =1654 43%

If n=4, m=1, k=5, Mw= 4*236+ 44*5 + 57*5+3 =1452. 65%

If n=4, m=3, k=5, Mw= 4*236+ 44*5*3 + 57*5+3 =1672 56%

If n=5, m=1, k=5, Mw= 5*236+ 44*5 + 57*5+5 =1686 70%

If n=5, m=3, k=5, Mw= 5*236+ 44*5*3 + 57*5+5 =2130 55%

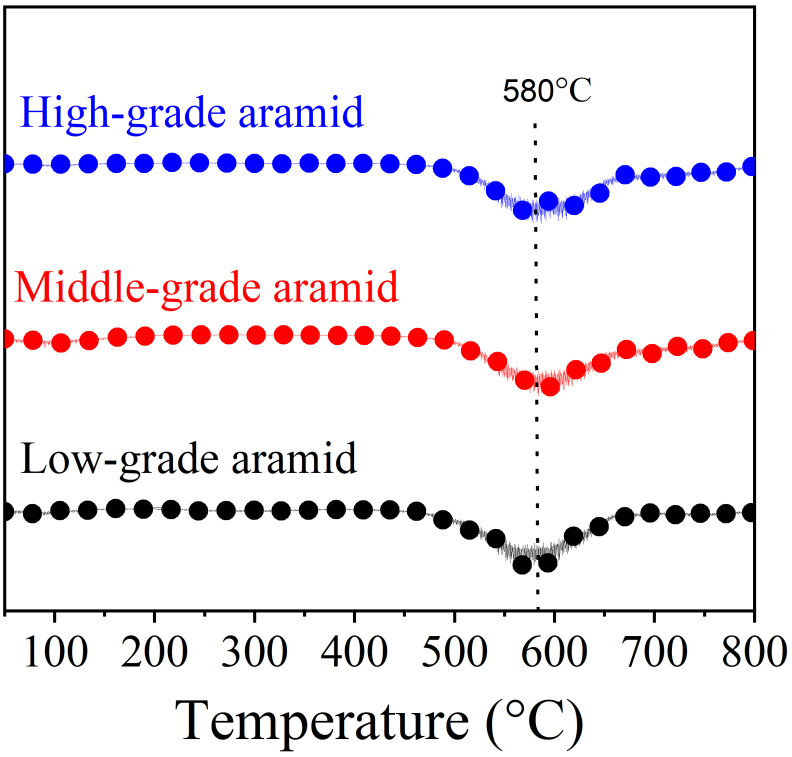


Fig. S5 Derivative of thermogravimetric curve of low-grade, middle-grade, and high-grade aramids