

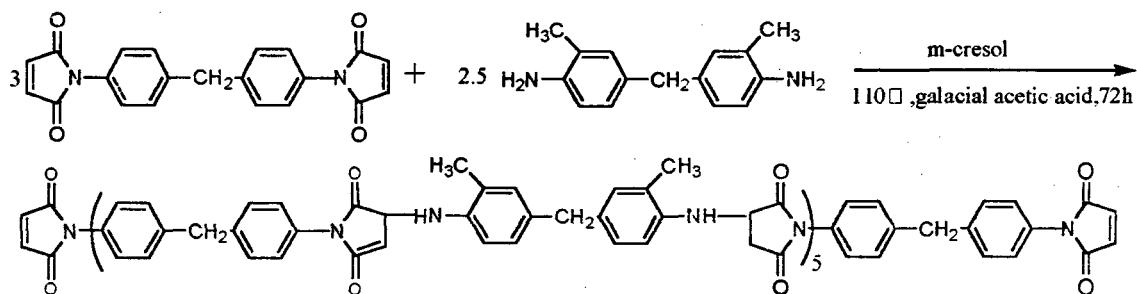
Investigation on radiation polymerization of cast system based on chain-extended bismaleimide DPBMI

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In this paper, we would like to report the preparation of IPN or Semi-IPN resin material by way of gamma ray radiation polymerization of cast system based on chain-extended bismaleimide DPBMI. Cast system we obtained in this work is a homogeneous, transparent binary solution with the solid content of 50 wt% composed of reactive solvent 1-vinyl-2-pyrrolidone and chain-extended bismaleimide DPBMI, which is prepared by reacting 4,4'-bis(maleimido)diphenylmethane with 3,3'-dimethyl-4,4'-diaminodiphenylmethane through Michael nucleophilic addition with the molar ratio of 3:2.5. The cast system was polymerized under ^{60}Co gamma ray radiation with the dose of 100 kGy and 200 kGy as designated as CS-1 and CS-2, respectively below, to obtain still transparent resin materials, which were subsequently postcured at 110°C(1h), 130°C(2h), 150°C(2h), 130°C(2h), 110°C(2h). The mechanical properties, water uptake property, gel content and Tg of the thus prepared resin material before and after postcuring were tested and evaluated. The synthesis of DPBMI is shown below.



Scheme 1: Synthesis of DPBMI

The experimental observation revealed that postcuring of CS-1 and CS-2 resulted in, to some extent, an increase of Tg and the mechanical properties, such as tensile strength at break(σ_b), tensile modulus(E_b) and impact strength. As seen in the Table 1, after postcuring, Tg and mechanical properties showed considerable improvement. As for CS-1, σ_b and E_b increased 5.3% and 20%, respectively after postcuring. Whereas σ_b , E_b of CS-2 increased 11.7% and 11.8%, respectively. It is especially noteworthy that, after postcuring, impact strength of both were enhanced greatly up to 153% and 75%, respectively. As expected, postcuring led to an increase of Tg of both resin material by 12°C and 6°C, respectively.

Table 1 Mechanical properties and Tg of resin material before and after thermal treatment

| Resin material | Radiation dose (kGy) | σ_b (MPa) | E_b (GPa) | Impact strength (kJ/m ²) | Tg (°C) |
|----------------|-------------------------|---------------------|----------------|---|------------|
| CS-1 | 100 | 76.2 | 2.610 | 1.56 | 170 |
| CS-1 postcured | 100 | 80.5 | 3.130 | 3.95 | 182 |
| CS-2 | 200 | 77.3 | 2.870 | 1.86 | 178 |
| CS-2 postcured | 200 | 86.4 | 3.380 | 3.26 | 184 |

Also, postcuring had an positive influence on water uptake property and gel content of CS-1 and CS-2. As seen in Table 2, after postcuring, water uptake of both resin material decreased from 22% to 15.7% and from 20.6% to 14.9%, respectively. As predicted, after postcuring, gel content of both resin material demonstrated an increase from 62.3% to 84.3% and from 64.5% to 77.5%, respectively.

Table 2 Water uptake and gel percentage of resin material before and after thermal treatment.

| Resin material | Radiation dose (kGy) | water uptake | Gel content |
|----------------|-------------------------|--------------|-------------|
| | | (%) | (%) |
| CS-1 | 100 | 22 | 62.3 |
| CS-1 postcured | 100 | 15.7 | 84.3 |
| CS-2 | 200 | 20.6 | 64.5 |
| CS-2 postcured | 200 | 14.9 | 77.5 |

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链延长的双马来酰亚胺低聚物的浇注体的辐照聚合的研究

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我们报道了对基于链延长的双马来酰亚胺低聚物 DPBMI 的浇注体体系通过 γ 射线辐照聚合的方法制备互穿网络或半互穿网络聚合物树脂材料。本实验中的浇注体体系是由活性溶剂 N-乙烯基吡咯烷酮和基于链延长的双马来酰亚胺低聚物 DPBMI 组成的固含量为 50wt% 的二元均相，透明体系。其中，链延长的双马来酰亚胺低聚物由 4,4'-一二马来酰亚胺基-二苯基甲烷和 3,3'-一二甲基-4,4'-一二氨基二苯基甲烷以摩尔比 3: 2.5 通过麦克尔亲核加成反应得到。该二元均相浇注体体系分别经过 ^{60}Co γ 射线源的 100KGy 和 200KGy 剂量的辐照聚合，得到透明的树脂材料，分别标记为 CS-1 和 CS-2。然后分别对 CS-1 和 CS-2 进行温度程序为 110°C (1h), 130°C (2h), 150°C (2h), 130 (2h), 110°C (1h) 的后处理，得到的树脂材料分别标记为 CS-1 postcured 和 CS-2 postcured。并且分别测试了上述树脂材料的力学性能、吸水性、凝胶含量和玻璃化温度。以下为 DPBMI 的合成路线。

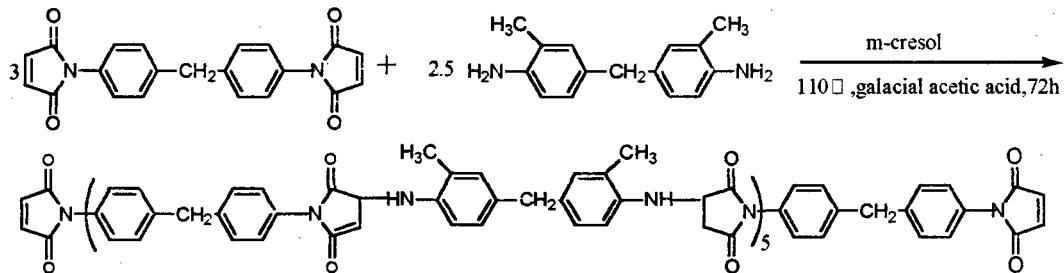


图 1：DPBMI 的合成

实验数据表明：对 CS-1 和 CS-2 进行后处理，在一定程度上提高了树脂材料的力学性能和玻璃化温度。从表 1 可以看出：后处理后，CS-1 的弯曲强度和弯曲模量分别提高了 5.3% 和 20%，CS-2 的弯曲强度和弯曲模量分别提高了 11.7% 和 11.8%。后处理对树脂材料的增韧效应尤其明显，经过后处理后，CS-1 和 CS-2 的冲击强度分别提高了 153% 和 75%。后处理使树脂材料 CS-1 和 CS-2 的玻璃化温度分别提高了 12°C 和 6°C。

表 1 后处理前后树脂材料的力学性能和玻璃化温度比较

| Resin material | Radiation dose (kGy) | σ_b (MPa) | E_b (GPa) | Impact strength (kJ/m ²) | Tg (°C) |
|----------------|-------------------------|---------------------|----------------|---|------------|
| CS-1 | 100 | 76.2 | 2.610 | 1.56 | 170 |
| CS-1 postcured | 100 | 80.5 | 3.130 | 3.95 | 182 |
| CS-2 | 200 | 77.3 | 2.870 | 1.86 | 178 |
| CS-2 postcured | 200 | 86.4 | 3.380 | 3.26 | 184 |

后处理对树脂材料的吸水性和凝胶含量也有正面的影响。从表 2 可以看出：后处理使树脂材料 CS-1 和 CS-2 的吸水性分别从 22% 和 20.6% 降至 15.7% 和 14.9%。凝胶含量也分别从 62.3% 和 64.5% 增至 84.3% 和 77.5%。

表 2. 后处理前后树脂材料的吸水性和凝胶含量

| Resin material | Radiation dose (kGy) | water uptake | | Gel content (%) |
|----------------|-------------------------|--------------|------|--------------------|
| | | (%) | (%) | |
| CS-1 | 100 | 22 | 62.3 | |
| CS-1 postcured | 100 | 15.7 | 84.3 | |
| CS-2 | 200 | 20.6 | 64.5 | |
| CS-2 postcured | 200 | 14.9 | 77.5 | |

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